Enhancing Precision in Epilepsy Monitoring with Telescopic Hybrid  $\mathrm{M}^3$  Electrodes

by

Jonathan V. Garich

# A Dissertation Presented in Partial Fulfillment of the Requirements for the Degree Doctor of Philosophy

Approved April 2022 by the Graduate Supervisory Committee:

Jennifer M. Blain Christen, Chair James J. Abbas Stephen I. Helms Tillery Jitendran Muthuswamy Gregory B. Raupp

ARIZONA STATE UNIVERSITY

August 2022

#### ABSTRACT

For patients with focal drug-resistant epilepsy, surgical remediation can be a hopeful last resort treatment option, but only if enough clinical signs can point to an epileptogenic tissue region. Subdural grids offer ample cortical surface area coverage to evaluate multiple regions of interest, yet they lack the spatial resolution typical of penetrating electrodes. Subthreshold stimulation through subdural grids is a stable source for detecting eloquent cortex surrounding potential epileptic tissue. Researchers have each tried introducing microelectrodes to increase the spatial resolution but ran into connectivity challenges as the desired surface area increased. Meanwhile, clinical hybrid options have shown promise by combining multiple electrode sizes, maintaining surface area coverage with an increased spatial resolution where necessary. However, a benchtop method to quantify spatial resolution or test signal summation, without the complexity of an *in vivo* study, has not been found in the literature; a subdural grid in gel solution has functioned previously but without a published method. Thus, a novel hybrid electrode array with a telescopic configuration including three electrode geometries, called the M<sup>3</sup> array, is proposed to maintain cortical surface area coverage and provide spatial clarity in regions of interest using precision microfabrication techniques. Electrophysiological recording with this array should enhance the clinical signal portfolio without changing how clinicians interface with the broad surface data from macros; this would provide a source for simultaneous recording and stimulation from the same location due to the telescopic nature of the design. A novel benchtop test method should remove complexity from *in vivo* tests while allowing direct comparison of recording capabilities of different cortical surface electrodes. Implementing the proposed M<sup>3</sup> electrode array in intracranial monitoring improves the current technology without much compromise, enhancing patient outcomes, reducing risks, and encouraging swift clinical translation.

### DEDICATION

This dissertation is dedicated to my family, friends I have met along the way, and especially to my loving partner, Lena. Without the support of everyone around me along with outlets for stress relief, this degree would not have been possible.

Additionally, I would like to dedicate my pursuit of biomedical engineering to the

family members we have lost for various medical reasons, especially to my grandfathers, Edward Garich (Papaw) and George Barkley (Pappy), who we all miss very dearly. Finally, I would like the thank all essential workers who have helped the world throughout the COVID-19 pandemic and want to share this dedication with all those we have lost in these trying times.

#### ACKNOWLEDGMENTS

Thank you to my amazing partner, Dr. Lena Heffern, and her dog, Dr. Bones, for keeping me centered, balanced, and reminding me that the best things in life are even better when shared with someone special. Even though we've both been in the final stages of our doctoral degrees since we met, you've shown me compassion, courage, and dedication that I can only wish to emulate. I can't wait to see where this journey takes us and who we happen to meet along the way.

Thank you to my parents, Matt and Barb Garich, for the emotional, financial, and many other variations of support you have given me throughout my time in grad school as well as earlier in life. Thank you to my brother, Ryan Garich, for becoming the stable one in our family so that I could temporarily resume the role I had in undergrad again. Thank you to my Grandmother and my Nana for being so supportive through the years, and I apologize for not contacting you both more often! Thank you to my many cousins, aunts, uncles, second-cousins, and all of my extended family across the world for reminding me to take a breath every once in a while.

Thank you to Dr. Daniel Gulick for helping me organize, process, edit, and wrap-up this dissertation. Thank you to Dr. Jennifer M. Blain Christen for the amazing mentoring as well as the opportunity and resources to investigate the research both within this dissertation and throughout the years I have spent at Arizona State University. Thank you to Dr. Mark A. Benscoter and Dr. Seth A. Hara for guiding me and helping me grow during my fellowship with the Division of Engineering at Mayo Clinic. Thank you to Stephen T. Kuehn for keeping the multitude of projects on track and showing me project management from clinical and engineering views. Thank you to Dr. Gregory B. Raupp and Dr. Gregory A. Worrell for leading and directing the Mayo-ASU seed-funded project and for the insights shared throughout the years. Thank you to my committee members, Dr. James Abbas, Dr. Stephen Helms Tillery, and Dr. Jitendran Muthuswamy, for teaching me in coursework, guiding me towards helpful documentation and literature, and helping me understand what it takes to run a research lab and earn a PhD.

Thank you to the ASU NanoFab staff for the knowledge, guidance, intuition, and expertise in all things microfabrication. Thank you to all members of the BioElectrical Systems and Technology (BEST) lab for helping me with experiments, teaching me something new from your respective research, and ultimately for allowing me to vent a few too many times. From that BEST lab member list, I wanted to specifically thank Dr. Dan Gulick, Dr. Uwa Obahiagbon, Dr. Nick Fritz, Hany Arafa, Ian Akamine, Dr. Mark Bailly, Dixie Kullman, Dr. Shiyi Liu, Christopher Lue Sang, Ryan Flores, Josh Eger, and many more. Thank you to the many officers and members of the Snow Devils ski and snowboard club at ASU for making sure I remembered to enjoy my time in the mountains and to stop and smell the roses every now and then. Thank you to everyone in Sunrise Ski Patrol for teaching me to be safe instead of fast, for helping me teach others, and ultimately for allowing me to help others while we all participate in the winter sports we love dearly. Thank you to all of my friends I have made from Ohio, Michigan, Minnesota, Arizona, and those who have since traveled beyond; I promise I'll come visit everyone after I am no longer a broke grad student!

# TABLE OF CONTENTS

Pag	е
LIST OF TABLES	ii
LIST OF FIGURES in	х
CHAPTER	
1 INTRODUCTION	1
1.1 Motivation	1
1.2 History	3
1.3 Physiology	4
1.4 Clinical Use	5
1.5 Modern Perspective	7
1.6 Fabrication	8
1.6.1 Substrate and Insulation	9
1.6.2 Conductive Layer 10	0
1.6.3 Alternative Lithography 1	1
1.7 Characterization 12	2
$1.7.1  \text{Connectivity} \dots \dots$	3
1.7.2 Electrochemistry 1:	3
1.8 Significance and Limitations 14	4
1.9 Chapter Preview 1	5
2 THE M <sup>3</sup> ELECTRODE 10	6
2.1 Introduction 19	9
2.2 Materials & Methods 23	3
$2.2.1  \text{Fabrication}  \dots  2.2.1  \text{Fabrication}  \dots  2.2.1  \text{Fabrication}  \dots  2.2.1  \text{Fabrication}  \dots  2.2.1  \text{Fabrication}  \dots  \dots  \dots  \dots  \dots  \dots  \dots  \dots  \dots  $	3
2.2.2 Electrode Design $\dots 2^4$	4
2.2.3 Electrode Interface	6

	2.2.4 Benchtop Characterization	26
	2.2.5 In Vivo Evaluation	27
2.3	Results	29
	2.3.1 Electrochemical Impedance Spectroscopy	29
	2.3.2 Control Studies	30
	2.3.3 Seizure Studies	32
2.4	Discussion	38
3 CORT	ICAL STIMULATION	45
3.1	Introduction	45
3.2	Materials and Methods	49
	3.2.1 Design and Interface	49
	3.2.2 Benchtop Characterization	50
	3.2.3 In Vivo Evaluation	50
3.3	Results	52
	3.3.1 Cyclic Voltammetry	52
	3.3.2 In Vivo Motor Stimulation	53
3.4	Discussion	53
4 A BEN	NCHTOP SPATIAL TEST BED	58
4.1	Introduction	61
4.2	Materials and Methods	62
	4.2.1 Design	62
	4.2.2 Fabrication	63
	4.2.3 Enclosure System	64
	4.2.4 Test Bed Characterization	65

Р	a	ge
-	$\sim c$	$\neg$

4.2.5 Electrode Evaluation	66
4.3 Results	66
4.3.1 Cyclic Voltammetry	66
4.3.2 Electrochemical Impedance Spectroscopy	67
4.3.3 Electrode Evaluation	68
4.4 Discussion	71
5 OUTLOOK & SUMMARY	75
REFERENCES	78
APPENDIX	
A FABRICATION PROCEDURES	88
B ELECTRODE EVALUATION	215

# LIST OF TABLES

Table		Page
2.1 Elec	trode Geometric Surface Area	

Figure	Page
2.1 Electrode Comparison Graphic	25
2.2 EIS for M <sup>3</sup> and Subdural Strip	30
2.3 Control Study PSD Ranges	31
2.4 Control Study PSD Frequency Bands	32
2.5 Envelope Detection Method	33
2.6 Pre-Ictal Event 1 Filtered	35
2.7 Pre-Ictal Event 2 Subtracted	36
2.8 Pre-Ictal Event 2 Filtered	37
2.9 Ictal Event Subtracted	38
2.10 Ictal Event Filtered	39
2.11 Post-Ictal Event Subtracted	40
2.12 Post-Ictal Event Filtered	41
2.13 Non-Seizure Event Subtracted	42
3.1 Cited Electrode Comparison Graphic	49
3.2 CV of $M^3$ and Subdural Strip	52
3.3 Minimal Activation 1.	54
3.4 Minimal Activation 2.	55
3.5 Cortical Activation Electrode Combinations	56
3.6 Estimated Rat Motor Cortex Map	57
4.1 STB Electrode Design	63
4.2 STB Enclosure System	64
4.3 STB CV	67
4.4 EIS for STB and Subdural Grid	68
4.5 STB Source Amplitude	69

# LIST OF FIGURES

# Figure

# Page

4.6	STB Source Frequency	70
4.7	STB Source Distance Low Frequency	72
4.8	STB Source Distance High Frequency	73

#### Chapter 1

# INTRODUCTION

Electrodes are unique tools for clinical applications, providing a glimpse into the physiology that cannot be seen with non-invasive measures. However, many disease states and bodily functions lack a complete understanding of the biological environment involved, needing innovations and fresh perspectives to advance our knowledge. The work presented in this dissertation takes a novel approach to flexible electrode design for an epilepsy disease model: telescopic electrode configuration. Compared to a subdural electrocorticography (ECoG) grid as a gold standard, the electrodes were fabricated in an Arizona State University (ASU) cleanroom, characterized by multiple benchtop methods, and implanted into rodent models. Featuring three levels of spatial resolution - centimeter-scale macroelectrodes (referred to as "macros"), millimeter-scale mesoelectrodes ("mesos"), and submillimeter-scale microelectrodes ("micros") - the M<sup>3</sup> electrode may aid in improved clinical outcomes and decreased risk potential. Hopefully, these electrodes can help a portion of the epilepsy population suffering from lost independence and autonomy.

#### 1.1 Motivation

Epilepsy can be a debilitating disorder for 65 million patients worldwide - 3.4 million in the US alone - due to the loss of independence and heavy burden on family members or caretakers, leading most patients to seek therapeutic options hoping to reduce or eliminate those dependencies (Obsorne Shafer and Epilepsy.com, 2013). Medications are the first clinical option as they carry minimal risk and cost. However, approximately one-third of epilepsy patients cannot find effective pharmacological op-

tions, placing them in the drug-resistant epilepsy population. After exhausting all other non-invasive clinical options, surgical remedies have been proven to provide positive outcomes for patients in this category (Worrell et al., 2004, 2008; Stead et al. 2010; Warren et al., 2010; Worrell, 2011; Worrell et al., 2012; Kucewicz et al., 2014). However, there are numerous risks to these interventions (Önal et al., 2003; Fountas et al. 2004; Araki et al., 2006; Van Gompel et al., 2008) in addition to the reliance on an accurate definition of the seizure onset zone (SOZ) from multiple scopes of evaluations. Thus, it is crucial that cranial activity monitoring, especially intracranial modalities, is accurate in determining the spatial breadth of epileptogenic tissue within the SOZ. However, the current clinical standard for cortical surface monitoring - subdural grids and strips - was intended for estimation and summation to cover a sizeable cortical surface area and not for spatially-precise region mapping (Penfield and Jasper, 1954). Many attempts to update this evaluation strategy since it was introduced in the 1970s have focused on improving the spatial resolution (Khodagholy) et al., 2015; Wang et al., 2017) or reducing the number of connections (Viventi et al. 2011). However, few have sought the combination of these improvements (Van Gompel et al., 2008; Worrell et al., 2008, 2012) to enable swift translation to clinical practice. Manufacturing these proposed combination approaches has proven to be more difficult than anticipated and has found traction primarily with depth electrode implementations. Ultimately, the half-century-old subdural electrode technology has not yet been ousted from its clinical standing. Instead, it has been implemented alongside updated approaches to analysis and processing algorithms better to localize waveforms through dipole triangulation (Brinkmann et al., 2015).

#### 1.2 History

Medical devices, especially for neurological implantation, have had a long and sometimes arduous history. Sir Isaac Newton may have been the first to suggest that muscle movements are triggered by electrical activity from the brain and nervous system in the early 1700s (Newton, 1729). However, it should be noted that this was derived from the belief that spirits controlled the electricity. Luigi Galvani accidentally found that dissected frog legs could be reactivated with electricity while investigating static electricity in the late 1700s (Galvani, 1791). Alessandro Volta decided to connect his ear canals to a battery with two wires in the early 1800s, where he discovered the current produced a crackling sound he heard (Volta 1800). If it has not already been apparent, the beginnings of modern electrophysiology were not the most scientifically rigorous or safe test methods, mainly because there was no existing technology capable of what was being discovered at the time. As the technological advancements became increasingly significant, the experiments required more rigor. Notably, Alan Hodgkin and Andrew Huxley used a giant squid axon and fast electronic equipment in the 1950s to detect and mathematically predict the propagation of an action potential across the length of a neuron (Hodgkin and Huxley, 1952). The Food and Drug Administration (FDA) regulated everything after this discovery, including laboratory certifications (1944), performance standards for electronic and radiative devices (1968), and guidelines for the market preparation of medical devices (1976) (Geller, 1976). Devices with electrodes implanted in the human brain require rigorous testing and certification. Breakthroughs, such as an implantable pacemaker in 1958 (Elmqvist and Senning, 1959) and a cochlear implant in 1961 (House, 1987), opened the doors for other clinical uses of electrodes to help patients deal with various disorders. Electrocorticography (ECoG), developed by Wilder Penfield and Herbert Jasper in the 1950s, was established to locate epileptogenic zones in patients with severe epilepsy (Penfield and Jasper, 1954). Once seizure onset zones are identified, surgical removal of the epileptogenic tissue remains the most effective remediation. However, due to significant geometric gaps between the electrodes and anatomy below, ECoG does not detect localized events, while microelectrodes and other alternatives do.

# 1.3 Physiology

Electrophysiology is one of the few tools currently available to gain knowledge about the human body's inner workings. However, it requires a thorough understanding of the underlying principles of the anatomy and physiology beneath the electrodes. From a top-down perspective, the nervous system can be broken down into two parts: the central and peripheral nervous systems. Peripheral nerves act as gateways to the muscles and organs of the body. In contrast, central nerves are the relays and higher-thought processes relating to memory, emotion, and language, as well as orchestrating voluntary movement (Kandel et al. 2000). Spinal cord branches connect to the peripheral nerves, closing the loop on the nervous system, while the brainstem controls organ processes that maintain life, such as breathing, heart rate, and blood flow. The cerebellum controls coordinated movement and posturing, acting as the switchboard for sensory inputs and motor outputs. Primary processing power comes from the cortex, which is categorically organized into four lobes that each deal with various functions of the body: frontal, parietal, occipital, and temporal. Each lobe is further subdivided into sections to compartmentalize tasks and communication networks, typically in bumps ("gyri") separated by grooves ("sulci").

Frontal lobe divisions include primary, secondary, and tertiary motor networks, cognition, emotion, speech production, and many others - sometimes referred to as

the control center of our personality. Sensation processing and interpretation are located in the parietal lobe, including language processing and proprioception, or the awareness of the location of our body in space. Vision comes from the occipital lobe, which can be mapped to account for retinal input and the various color filtering, object recognition, and orientation calibration processing. Balance, hearing, and learning typically exist within the temporal lobe, which is also home to our short-term and long-term memory storage sources. These divisions within the lobes of the cortex help associate the trillions of connections between the billions of neurons, creating a network of possible pathways a signal can travel (Freeman *et al.*, 2000). Neurons are the base cell of the nervous system and act to transmit information using an electrical signal, called an action potential, to other neurons to enable or disable specific actions in the network. Proteins within a neuron act as channels for specific ions to flow into or out of the cell - namely sodium, potassium, calcium, and chloride ions (Hodgkin and Huxley [1952] - creating an ion flux that translates into an electrical field. However, this electrical field's polarity changes with the ions' circulation, creating a dipole of charge (Holmes and Khazipov, 2007). Layers of neurons in the neocortex, or the outer surface of the cortex, are organized into columns and typically network together in clusters (Kandel et al., 2000). Summation of dipole potentials from these cortical columns can be detected with an electrode on the surface of the cortex in the subdural space, which results in the electrophysiology previously discussed.

# 1.4 Clinical Use

Intracranial monitoring for patients without epilepsy reveals a range of wavelengths, primarily in the lower-frequency range, yet the action potential is typically in the range of 200-300 Hz (Freeman *et al.*, 2000). Sources of this variance may differ between each person. However, an electrode in the subdural space records local field

potentials, not action potentials, and detects the summation of synchronous activity in neighboring cortical columns (Blakely et al., 2008; Wang et al., 2017; Dubey and Ray 2019 Kanth and Ray 2020 of which the frequency loosely guides the location and action of the source network. For instance, delta waves (0.5-4 Hz) have a higher amplitude and reflect regional slowing typically seen in sleep states or just before a seizure event begins in some patients (Lundstrom et al. | 2018). Conversely, spikes or sharp waves indicate bursts of activity and can either be seen alone or in clusters. Electroencephalogram (EEG) recordings during seizures are typically largeamplitude spike clusters in specific regions that spread quickly throughout the brain, sometimes only briefly and others lasting for minutes. Spectrum analysis of these EEG waveforms helps clinicians determine therapeutic options, including intracranial monitoring targets and possible neuromodulation devices. However, these are always last resort options, with medications, diet, and imaging studies first. In addition, any surgical option carries an unnecessary risk to patients with milder epilepsy cases that can be treated with less-invasive alternatives, primarily due to the likelihood of inflammation and infection from subdural electrode implantations (Onal et al., 2003; Fountas et al., 2004; Araki et al., 2006; Van Gompel et al., 2008). However, the patients that do require surgical intervention to achieve seizure reduction have seen primarily positive results when the SOZ is confirmed with multiple sources (Wetjen et al. 2009), namely anatomical or physiological anomalies detected through imaging and electrophysiological monitoring studies. Consequently, the scale of the anatomy and physiology of humans is not entirely on the same scale as the electrode geometry, so innovative alternatives are still under investigation.

# 1.5 Modern Perspective

Modern takes on the 1950s ECoG technology are typically focused on many improvements, from the electrode geometry to the materials and post-processing algorithms. However, the current clinical standard remains the same. Addressing the inflammation and infection complications, researchers have found flexible polymers such as polyimide and parylene as suitable alternatives to the silicone used in subdural grids. However, the FDA has only recently approved polyimide in biomedical implant devices. On the other hand, parylene has a long history of use in implantables as a coating or encapsulation layer. Depositing biocompatible conductive layers such as platinum on parylene is not an easy feat due to the mismatch in thermal coefficients, resulting in cracking. Recent attempts at bio-inspired materials, such as liquid crystal polymer (Woods et al., 2018), may be able to better bridge the gap to the physiology but is years away from an FDA-approved clinical device. Innovations in implantation techniques through stereotactic EEG (SEEG) (Van Gompel et al.) 2010 Bourdillon et al. 2017 have shifted the focus of many researchers toward depth electrodes instead of surface electrodes to provide three-dimensional (3D) viewpoints to intracranial monitoring. Typically inserted as a catheter sleeve, depth electrodes commonly feature polyimide as the primary material due to its manufacturability and flexibility. However, inserting a catheter into the brain tissue still causes cellular damage that can be long-lasting if not permanent (Lee *et al.*, 2000) Campbell and Wu, 2018). Commonly, innovation attempts are focused on improving the spatial resolution of the electrode geometry to get closer to the scale of the physiology but quickly run out of available channels on the recording systems. Passive and active electronics, such as thin-film transistors (TFTs), familiar to CMOS and MEMS devices, can reduce the wiring complexity for recording systems but also require custom interfaces to convert the signals into a format required for clinical electrophysiology systems. While this is likely the future of electrode development moving forward, there are still improvements and certifications remaining for widespread implementation. Algorithms have eased interpretation and localization efforts for physician teams, typically using dipole triangulation from specific waveforms or timeframes (Brinkmann *et al.*) 2015). These capabilities can effectively increase spatial distinction from the current subdural grids, but physical separation in signals would reduce complexity cost and increase algorithm stability.

#### 1.6 Fabrication

Devices made for biological environments must follow strict guidelines to prevent harm to living organisms, set by regulatory bodies such as the FDA, ISO, ANSI, and AAMI. Manufacturing processes are regulated for repeatable data collection, biological safety, and traceability. Implantable devices, especially intracranial electrodes, require tight tolerances, controlled current and heat outputs, and longevity testing. The most prominent research has been focused on alternative material choices and closing the geometric gap between anatomy and the electrodes detecting physiological activity. Subdural grids and other implantable electrodes are manufactured with injection-molded silicone and fragile wires soldered to button discs for metal contacts, preventing geometry or material improvements without significant process changes. For decades, microfabrication has been used for making conductive metaloxide-semiconductor (CMOS) and microelectromechanical systems (MEMS) devices. It offers precision in both thickness control and minimum feature sizes due to the mechanisms of each method. Additionally, automation of these fabrication processes for batch manufacturing only requires a tested recipe and a production facility with the right equipment.

#### 1.6.1 Substrate and Insulation

Injection-molded silicone ensures an affordable manufacturing option but lacks specific capabilities like millimeter-scale precision and thickness. Alternative polymer options promise improved precision, on a micrometer-scale or smaller typically, bringing elevated flexibility and conforming abilities and simple microfabrication techniques for repeatable manufacturing. For example, parylene-C, a polymer similar to plastic wrap, is commonly used as a sealant when implanting devices made with biotoxic materials, such as silicon or copper, and has seen effective biocompatibility across multiple use cases (Del Valle et al., 2015)?). Controlled vaporized pyrolization completely coats everything in the chamber, with thickness control based on the amount of dimer used. Unfortunately, the thermal properties of Parylene-C are drastically different from typical metals, often resulting in cracking. The only known solution is to increase throw distance in the deposition systems, which disrupts calibrations on all other metal depositions (Rodger et al.) 2008 Ortigoza-Diaz et al. 2018). Fortunately, another polymer has shown promising biocompatibility (Richardson et al., 1993 Lenihan et al., 1996; Sun et al., 2009) with stronger thermal bonding than Parylene-C (Del Valle et al., 2015; De La Oliva et al., 2018). Primarily due to its prominent use in making flexible printed circuit boards (PCB), polyimide is a widely accepted conformal polymer compound. Available in sheets or spin-coated formulations, polyimide has also gained traction on the regulatory side of medical devices, recently obtaining 510(k) predicate (K192764 and K211367, NeuroOne Medical Technologies Corporation, Eden Prairie, MN, USA) approval from the FDA (Worrell *et al.* (2019). Specifically, the PI-2611 polymer-in-solution formula maintains high levels of precision in thickness and flexibility, with partial cure practices for bonding multiple layers together. This composition was chosen as the optimum substrate and insulation material for the electrodes discussed in later chapters.

### 1.6.2 Conductive Layer

Conduction through traces or wires is required for electrode connections, but the material choice depends on the disease model, anatomical target, and length of time in a biological environment. Typical biocompatible metals include gold, titanium, and platinum, with high conductivity levels and minimal biological reactivity. However, alternative options are under investigation, such as gold nanoparticle embedded polydimethylsiloxane (PDMS) (Minev *et al.*, 2015) or conductive carbon formulations like poly(3,4-ethylenedioxythiophene) polystyrene sulfonate (PEDOT:PSS) (Kayser and Lipomi, 2019). Each conductive material option has inevitable trade-offs that require consideration for each case study, resulting in many electrodes and devices using various materials for each disease model.

Gold, for instance, is famous for biological recording electrodes with a low impedance, low deposition temperature, and high natural surface area. However, the lower current density limits stimulation capabilities, and flakes are unfortunately familiar in a biological environment. Titanium is a heightened strength-to-weight material for structures or scaffolding in a physiological implant. However, oxidation prevents it from being a strong competitor in the recording or stimulation capacity (Casaletto *et al.*) [2001). On the other hand, platinum has low impedance, high stability in an aqueous solution, and high current density for optimum stimulation characteristics, making it the preferred conductive material for electrodes chronic implantation and cortical stimulation. A higher deposition temperature for platinum, however, does add limitations and extreme strain on the substrate material choices. Conductive polymers are a promising focus for continuing research and comparison. However, there has not been enough physiological testing exposure to prompt translation into the clinical setting, thus eliminating conductive polymers as an option for our electrodes.

#### 1.6.3 Alternative Lithography

Microfabrication techniques typically provide extreme levels of precision - down to Angstroms or nanometers - and rapid escalation into batch processing. However, microfabrication requires a dedicated cleanroom with engineered airflow, environmental controls, and periodic machine maintenance and upkeep. Additionally, tools and machines standard for microfabrication require significant funding and vast spaces due to the various engineering process controls, pumps, and connections to gas or fluid lines. As a result, some cleanroom laboratories offer paid services or student training since the cost can be a significant barrier to entry. Examples of these service offerings include the ASU NanoFab (Choi *et al.*) 2010; Theofanopoulos and Trichopoulos, 2019) and the Polymer Implantable Electrode (PIE) Foundry out of the University of Southern California (USC) (Weltman *et al.*) 2016; Hara *et al.*, 2016).

Alternative options to microfabrication, such as laser ablation, are available to researchers with tighter budgets seeking precision and rapid prototyping capabilities. A femtosecond laser system can ablate polymers, pattern metals, and expose contacts without requiring a cleanroom environment or significant laboratory space. Machines such as spinners, ovens, metal deposition systems, and metrology tools are still required for optimal fabrication, but these can all be customized to available space. Our team determined process parameters and requirements for femtosecond laser fabrication of a second version of the  $M^3$  electrodes presented in upcoming chapters, published in Frontiers of Biomedical Devices (Yeh *et al.*) 2020). The author of this dissertation was the second author of this publication with significant contributions to the process parameters, design, and editing. This alternative design required slightly larger traces and electrodes due to a 20 µm laser spot size in an ultraviolet (UV) wavelength. Summarizing these findings shows that controlled laser settings and a system capable of oxygen plasma cleaning or etching, along with some rudimentary photolithography, results in electrodes capable of electrochemical detection in standardized benchmark testing.

#### 1.7 Characterization

Manufacturing or fabrication is only part of the problem when designing electrodes, especially for use in a physiological environment; complexities and complications are only found when testing the electrodes prior to implantation, also known as characterizing the performance characteristics. Multiple benchtop-based test methods exist as benchmarks for different attributes of electrical or chemical performance, but electrodes specific to neurological physiology are typically tested using electrochemical impedance spectroscopy (EIS) and cyclic voltammetry (CV). Gamry (2021) has developed quality processes, methodologies, and metrics for testing and comparing electrical systems in these test methods for electrodes to batteries or fuel alternatives and even other resistive or capacitive circuitry, including approximations for equivalent circuit models to replicate these results in a discrete component manner. Bridging gaps between standard benchmarks and the complexities of *in vivo* studies are methods such as phantom electrophysiology, or simple signal detection and comparison in a saline environment to effectively simulate a physiological environment (Owda and Casson, 2020). However, considerations for connecting the electrodes to these systems require some forethought to reduce system impedances and better control and predict electrochemical behaviors.

# 1.7.1 Connectivity

Printed circuit boards (PCB) are the simplest method of connecting electrodes to systems, with many pre-existing options built for standard test systems making decisions easier. If the electrodes are custom-designed, a custom PCB option may also be required unless connection points are predetermined before manufacturing the electrodes. As discussed in the following chapters, thin-film electrodes with high flexibility may require specific connectors and thicknesses for repeatable connectivity. Considering shims or backing could aid in the thickness control needed. Benchmark testing may require additional custom PCB designs for each unique connector interface. For instance, electrochemical impedance spectroscopy (EIS) and phantom electrophysiology tests are conducted using two separate systems with individual connection requirements. However, additional connections in an electrode system increase impedance and could affect detection capabilities, especially with small microvolt ranges typical of ECoG arrays.

#### 1.7.2 Electrochemistry

Interestingly, there are only a handful of characterizations and benchtop tests to compare the performance of subdural electrodes before entering a biological system. Characterizations such as electrochemical impedance spectroscopy (EIS) and cyclic voltammetry (CV) reveal behaviors of the electrodes in basic terms such as equivalent circuit models and water windows, respectively (Gamry, 2021). Phantom electrophysiological signals in a solution can be a simple way to elevate the complexity of benchtop test methods but are not typically calculated systems and can depend on the quality of setup (Owda and Casson 2020). Ultimately, the goal is to test electrodes in the environment they will be placed in through *in vitro* or *in vivo* studies. However, physiology adds noise and complexity to the detected signals and can be challenging to filter out without disrupting essential datasets. Ideally, a benchtop test method that could quantify the relation between source distance, summation versus negation, and triangulation would enhance confidence in an electrode design compared to the clinical standard: subdural grids.

#### 1.8 Significance and Limitations

Spatial precision and cortical coverage sufficient for epilepsy studies are typically inversely proportional for subdural electrodes due to channel limitations on recording systems. So, a new implementation is needed to find a balance between both objectives to manage the connections required effectively. Electrodes have historically occupied a single space per electrode, with the diameter and spacing determining the spatial resolution. Larger electrodes should generalize a surrounding area, while smaller electrodes may distinguish between separate signals.

Interestingly, electrodes embedded within other electrodes have not been investigated yet, though this would allow for multiple spatial resolutions without increasing array size. Recording from each electrode geometry could be thought of as increasing the magnification of a telescope, which shall be called a telescopic configuration of electrodes. Larger electrodes could find regions of interest, while smaller electrodes could instantly gain more spatial precision. Each electrode's spatial reach in this telescopic configuration should be isolated from nearby electrodes enough that simultaneous stimulation and recording on the cortical surface is possible without artifacts typical of nearby pulse-width modulated current sources (Young *et al.*) [2018] [Shadmani *et al.*] [2018] [Zhou *et al.*] [2018] [Sellers *et al.*] [2019]. Additionally, a smaller electrode could potentially stimulate precise locations and induce a localized and isolated seizure waveform, called a microseizure event (Stead *et al.*] [2010]. If microseizures can be detected during an intraoperative procedure, surgical intervention can be the only additional assessment required. An intraoperative evaluation alone would reduce complication risks significantly, eliminating infection risk and inflammation responses (Önal *et al.*, 2003; Fountas *et al.*, 2004; Araki *et al.*, 2006; Van Gompel *et al.*, 2008). However, no benchtop test method exists that can evaluate microseizure detection, and *in vivo* induction of microseizures is limited in practice. Therefore, a test method capable of microseizure detection and distinction is required for complete comparison and characterization.

#### 1.9 Chapter Preview

Through some preliminary work and investigation, three novel contributions have been identified as outcomes to the proposed project, as mentioned above: (1) a novel telescopic  $M^3$  electrode array with similar recording capabilities as subdural grids, (2) cortical stimulation using the novel  $M^3$  electrode array, and (3) a novel benchtop test method to distinguish signal origination and quantify spatial resolution for subdural electrodes. Each novel contribution has been formulated into a separate chapter, with some chapters including upcoming journal or conference submissions. Each chapter will identify and test hypotheses relating to comparing the novel telescopic  $M^3$  array and subdural grids, the current clinical standard. Benchtop characterization and *in vivo* studies shall provide the data required to highlight differences or similarities between the electrodes. A multi-faceted test approach for the novel benchtop test method should provide quantifiable comparisons previously missing from the literature. Pilocarpine injections in the seizure animal models will be used to induce a seizure for recording purposes, following approved IACUC protocols (Turski *et al.*, 1983] [Clifford *et al.*] [1987] [Curia *et al.*] [2008]).

# Chapter 2

# THE M<sup>3</sup> ELECTRODE: FLEXIBLE TELESCOPIC CONFIGURATION ENHANCES SPATIAL RESOLUTION IN SUBDURAL RECORDING

Jonathan V Garich<sup>1,2,3</sup>, Ian R Akamine<sup>1</sup>, Seth A Hara<sup>2</sup>, Daniel W Gulick<sup>3</sup>, Mark A Benscoter<sup>2</sup>, Stephen T Kuehn<sup>2</sup>, Gregory A Worrell<sup>4</sup>, Gregory B Raupp<sup>5</sup>, Jennifer M Blain Christen<sup>1,3</sup>

<sup>1</sup>Biomedical & Health Systems Engineering, Arizona State University, Tempe, AZ, <sup>2</sup>Division of Engineering, Mayo Clinic, Rochester, MN,

<sup>3</sup>Electrical, Computer, & Energy Engineering, Arizona State University, Tempe, AZ, <sup>4</sup>Department of Neurology, Mayo Clinic, Rochester, MN,

<sup>5</sup>Engineering of Matter, Transport, & Energy, Arizona State University, Tempe, AZ

Paper to be submitted to JOURNAL OF NEURAL ENGINEERING with approval from Co-Authors Ian Akamine, Seth Hara, Daniel Gulick, Mark Benscoter, Stephen Kuehn, Gregory Worrell, Gregory Raupp, and Jennifer Blain Christen.

#### ABSTRACT

*Objective.* Surgical decisions for focal drug-resistant epilepsy patients are guided by seizure onset zone (SOZ) definition from intracranial monitoring, but a significant mismatch between electrodes and anatomic geometries could sacrifice key seizure metrics like higher-frequency oscillations (HFO) useful for region separation. We developed a subdural polyimide array with a telescopic configuration of three platinum electrode geometries, called the M<sup>3</sup> electrode, providing multiple levels of spatial resolution for epilepsy studies. Benchtop characterization and in vivo studies with a pilocarpine rodent model should compare the M<sup>3</sup> array to the subdural electrocorticography (ECoG) strips used clinically. Approach. Electrochemical behaviors should be distinctive based on geometry. Bilateral subdural implantation in a seizure-prone rat model should indicate the localization and HFO detection potential of the M<sup>3</sup> electrodes compared to a subdural strip. Main results. Impedance magnitudes followed geometric expectations, with the button-style ECoG electrodes one order of magnitude below all other electrodes at 1 kHz. Spectrum power under 100 Hz in an anesthetized control study was at least 35% higher in the subdural strip electrode than in the  $M^3$  electrodes. However, the  $M^3$  mesos and micros had a 15% higher power above 100 Hz than the subdural strip electrode. The three phases of a seizure were distinct in time-domain signals, with an envelope detection capturing significant events above a 30% threshold. Pre-ictal and inter-ictal events were localized by  $M^3$ mesos and micros to a 1 mm by 1 mm region. Ictal and post-ictal events generalized rapidly across all electrodes, though initial detection was still distinguished. The mesos and micros localized isolated events during a control study to a 2 mm by 2 mm region. Significance. Benchtop performance of the  $M^3$  electrodes is comparable to conventional ECoG electrodes, though stability at lower frequencies is best with the subdural strip electrodes. Significant grounding issues required additional post-processing steps. After subtracting the baseline artifact, localized detection was possible with the M<sup>3</sup> mesos and micros. The subdural strip electrode and M<sup>3</sup> macro only detect generalized events, though this may be due to the artificial seizure model. Naturally-occurring epilepsy models should be tested with the M<sup>3</sup> electrode array to confirm the spatial resolution distinctions.

# 2.1 Introduction

Intracranial electrodes provide distinct seizure detection for focal drug-resistant epilepsy patients, primarily when the seizure onset zone (SOZ) is located accurately. Region definition from these electrodes differentiates epileptic tissue from eloquent cortex and can increase chances of positive clinical outcomes (Wetjen et al., 2009). Surgical resection is the most effective treatment option in most cases (Hader *et al.*) 2013), making inaccuracies in SOZ definition detrimental to patient improvement. Tight resection margins could require additional interventions, but loose boundaries may impact functional areas and restrict independence. Spatial resolution in neurological activity ranges from micron-scale cortical columns to centimeter-scale synchronized networks, but available electrode options do not span this range clinically. Subdural electrocorticography (ECoG) grids offer a centimeter-scale spatial resolution to cover multiple spanning regions common in epilepsy cases (Penfield and Jasper, 1954). Microelectrodes provide a submillimeter-scale spatial resolution to track synchronized cortical columns and localized higher-frequency events (Kandel et al.) 2000; Holmes and Khazipov, 2007; Freeman et al., 2000; Schevon et al., 2008). Combinations of macro-scale and micro-scale electrodes revealed seizure-specific waveforms above 100 Hz, or higher-frequency oscillations (HFOs), with a significant spatial correlation to epileptic tissue detected primarily from the micro-scale electrodes (Worrell *et al.*) 2008, 2012; Brázdil et al. 2017). However, thermoplastic molds for these hybrid electrode combinations do not offer high precision or yield for the silicone-encased grids (Penfield and Jasper, 1954). In addition, implantation of subdural ECoG electrodes requires a large skull opening for placement, increasing probabilities of infection and inflammation complications as a result of the array's relative thickness and rigidity compared to the limited subdural space and gelatinous nature of the brain (Onal *et al.*, 2003; Fountas *et al.*, 2004; Araki *et al.*, 2006; Van Gompel *et al.*, 2008). An opportunity exists to develop a thin-film multi-scale electrode array with precision manufacturing principles to aid clinical research in accurate seizure region definition and HFO detection.

Material selection for intracranial electrodes depends on many factors, though manufacturing methods are often overlooked compared to performance and significance. Clinical epilepsy electrodes focus on simple manufacturing techniques, such as molded silicone for subdural ECoG grids or coated catheters for depth electrodes, where combinations of these electrode types commonly scatter across the hemisphere of an epilepsy patient (Wetjen et al., 2009). Prevalence of complication risks from surface electrode implantations (Onal et al.) 2003; Fountas et al., 2004; Araki et al. 2006 Van Gompel et al. 2008 encouraged significant materials research to better match Young's modulus of the cortex. Parylene showed promising conforming and biocompatibility properties, primarily as an encapsulation material, but thermal coefficient mismatch limited the use of certain conductive materials, such as platinum (De La Oliva et al. 2018; Ortigoza-Diaz et al. 2018). Following established fabrication processes from flexible printed circuit boards (PCB), polyimide added precision to manufacturing without reducing simplicity and proved capable of long-term implantation (Richardson et al., 1993; Sun et al., 2009; Romanelli et al., 2019). Additional materials such as liquid crystal polymer (LCP) have shown promising results, but manufacturing methods and longevity are still under investigation (Woods et al. 2018). Regulatory barriers are the largest encumbrance for these alternative materials to enter clinical usage (Lu et al., 2012), but recent market approval of a polyimide array may enable rapid commercialization (Worrell et al., 2019).

Microelectrodes improve spatial resolution naturally by reducing detection volume, thereby increasing possible electrode densities. Wang *et al.* (2017) recognized

the importance of smaller electrodes to distinguish the effects of blood vessels underneath implanted electrodes using two different arrays. The larger array featured 1.81 mm diameter electrodes and 3.5 mm spacing, while the smaller array utilized 0.87 mm diameter electrodes and 1.68 mm spacing. Cortical columns are significantly smaller than blood vessels, so Khodagholy et al. (2015) matched this geometry with 10 µm diameter electrodes and 40 µm pitch to prove that detection of independent action potentials was possible. Submillimeter scales are suggested for studying synchronized epilepsy networks (Freeman et al., 2000), so an electrode spacing of 1.25 mm allowed for epileptiform discharge detection by Schevon *et al.* (2008). Clinical settings must set a channel-count limitation for submillimeter-scale arrays to balance patient comfort and keep wire bundles manageable. Alternatively, an epilepsy patient may require thousands of submillimeter-scale electrodes and connections to monitor the scattered regions, but active electronics could eliminate the need for channel-count limitations. Viventi *et al.* (2011) showed that flexible thin-film transistors (TFTs) embedded in the array could reduce channel count to the number of columns instead of the total electrodes. Neuropixels, developed by Jun et al. (2017), implemented widely available transistors on a silicon headstage to digitize signals prior to preamplification, effectively switching between three groups of electrodes at will. Complexity costs of active electronics exponentially increase with electrode count, but evolution into active electrodes will soon be required for large-scale electrophysiological recording. Hybrid combinations of centimeter-scale and submillimeter-scale electrodes may be an immediate opportunity to access higher spatial resolution recording without compromising cortical surface coverage.

Hybrid electrode arrays, like those demonstrated by Worrell *et al.* (2008) 2012), showed that smaller electrodes could detect higher-frequency activity better than larger electrodes. Activity in each band of these higher-frequency oscillations (HFO) was proven to have a significant correlation to localized epileptogenic tissue in proximity to the detecting electrode (Brázdil *et al.*) [2017). Ultra fast ripple (UFR: 1-2 kHz) and very fast ripple (VFR: 0.5-1 kHz) bands represented the most substantial connection to nearby epileptic sources, while the fast ripple (FR: 250-500 Hz) band still maintained a significant correlation after biopsy confirmation. Ripple (R: 100-250 Hz) band activity sometimes indicated the presence of an epileptogenic zone, but higher frequency bands had better distinction from baseline. Some activity in the slow ripple (SR: 50-100 Hz) band may indicate neighboring seizure regions, though not necessarily a trustworthy source (Wang *et al.*) [2013). Post-processing for these HFO bands typically features a high-pass filter set to 80 Hz, eliminating most of the nominal signals recorded in standard clinical settings without designated radio frequency (RF) shielding (Stead *et al.*) [2010). Unfortunately, manufacturing these hybrid arrays is a complicated procedure with low precision and yield, so alternative solutions with simple manufacturing practices would drastically improve repeatability.

We present a microfabricated thin-film polyimide electrode array featuring multiple scales of electrodes organized in a telescopic configuration. Centimeter-scale macroelectrodes (referred to as "macros") maintain the cortical surface coverage desired for epilepsy cases and reduce complexity in clinical translation by keeping data expectations similar to that of subdural grids. Submillimeter-scale microelectrodes ("micros") add significant spatial resolution within the macro, assisting in determining the direction and orientation of SOZ location, size, and spread. Finally, millimeter-scale mesoelectrodes ("mesos") exist between the macros and micros, acting as an intermediary step between cortical coverage and spatial resolution in addition to a future source for cortical stimulation. Telescopic configuration allows the electrode scales to act like a microscope or telescope: increasing spatial resolution with each scale while reducing the field-of-view. We deem this the M<sup>3</sup> array for its three geometry levels and believe it can offer enhanced detection capabilities for epilepsy studies. Benchtop characterization and *in vivo* comparison to subdural electrodes establish the performance of each electrode. Spatially mapped results from an injection-based seizure model should aid in determining the origination, spread, and cutoffs of localized events.

### 2.2 Materials & Methods

# 2.2.1 Fabrication

Following common microfabrication principles, 10 µm layers of PI-2611 polyimide (HD Microsystems, Parlin, NJ, USA) were deposited onto a silicon wafer carrier as the substrate and insulation for our M<sup>3</sup> array. This polyimide composition was ideal due to its precision thickness control from spin-coating (Lenihan et al., 1996; Engel et al. 2003; Dobrzynska and Gijs, 2012) and proven biocompatibility (Richardson et al., 1993; Sun et al., 2009). Dehydration bakes before each layer deposition prevented moisture retention and ensured bonded seals between the polyimide layers. In addition, surface roughening after each dehydration bake added depth to the polyimide's naturally porous long-chain polymer structure, preventing platinum ears and delamination on ASTM D3359 tape tests following fabrication. Platinum was selected as the conductive material due to its low impedance and high charge storage capacity, especially since the specification requirements established a need for stimulation capabilities. A titanium adhesion layer engaged the deep wells of polyimide chains and allowed platinum to adhere completely. Deposition of a 5 nm titanium adhesion layer and a 100 nm platinum conductive layer occurred using an electron beam evaporation system (Kurt J. Lesker Company, Jefferson Hills, PA, USA). Lift-off photolithography was conducted using a 3 µm resolution chrome mask to pattern both metal layers. Contacts were exposed using a dry etching process with oxygen plasma in a PT-790 reactive ion etching system (PlasmaTherm, St. Petersburg, FL, USA)). Final stack thickness was measured at 20 µm, designated for optimum flexibility without tearing. All fabrication processing was performed in Arizona State University (ASU: Tempe, AZ) cleanrooms, with portions completed in the ASU NanoFab (Class 100), with the remaining conducted in the Soft Lithography Laboratory (Class 1,000).

# 2.2.2 Electrode Design

M<sup>3</sup> electrodes were intended to keep clinical translation simple while adding additional spatial resolution where desired. Macros were designated to match standard electrode dimensions from subdural ECoG grids and strips, with 4 mm diameter and 10 mm center-to-center spacing. Micros were designed to span a single cortical column, with 40 µm diameter and 350 µm center-to-center pitch, suggested by Worrell et al. (2012) to have complete spatial coverage without much overlap. Variations in diameters of intracranial electrodes commonly exist between 1-4 mm across surface and depth electrodes, so mesos were conceived to bridge the geometrical gap with 1 mm diameter and 1.5 mm center-to-center separation. Thus, the  $M^3$  array, seen in Figure 2.1, maintains cortical surface coverage of a subdural ECoG grid while carrying multiple spatial resolutions: centimeter-scale from macros, millimeter-scale from mesos, and submillimeter-scale from micros. The telescopic configuration of these electrode scales enables separate connections to each scale. For instance, initial monitoring can happen over an 8 cm x 8 cm grid of macros (64 channels) where zones can be mapped to symptoms or waveforms. After an initial mapping, a closer inspection can follow from selections of 2 mm x 2 mm clusters of mesos (4 channels) or 1 mm x 1 mm clusters of micros (7 channels). Switching between the three spatial resolution



Figure 2.1: Microscope image of a 32-channel M<sup>3</sup> electrode.

levels can be as simple as replacing connectors in the amplifier setup. However, it should be noted that connections for all electrodes still exist and would have to pass through the skull, though this opportunity cost is significantly lower than additional surgeries. Due to the limited available space on a rat cortex, a 32-channel  $M^3$  array and a single electrode from a 1 x 4 subdural ECoG strip (AD-Tech, Oak Creek, WI, USA) were selected for this study. A 32-channel  $M^3$  array contains one macro, four mesos, and 27 micros, covering a 2 mm x 2 mm region. Each subdural strip electrode has a 2.3 mm diameter contact opening on a 4 mm platinum button electrode. Notably, cutouts in the  $M^3$  macro and mesos were required for routing traces, partially limiting geometric surface area (GSA) as shown in Table [2.1].
# 2.2.3 Electrode Interface

Connection to the M<sup>3</sup> electrodes was made through a flexible printed circuit (FPC) zero insertion force (ZIF) connector with 39 pins (Molex Connector Company, Lisle, IL, USA). M<sup>3</sup> arrays were adhered to 127 µm thick polyether ether ketone (PEEK) with cyanoacrylate to maintain consistent contact with FPC ZIF pins. Electrode interface boards (EIB) were custom designed to connect the FPC ZIF to an RHD electrophysiology evaluation system (Intan Technologies, Los Angeles, CA, USA) using an Omnetics connector (Minneapolis, MN, USA). Additional EIB designs connected the M<sup>3</sup> array to post connections for benchtop characterization. All connections were confirmed with multimeter probes prior to testing and characterization.

# 2.2.4 Benchtop Characterization

Benchtop testing included potentiostatic electrochemical impedance spectroscopy (EIS) and phantom electrophysiology (phantom ephys). Conducted in a 1x phosphatebuffered saline (PBS) solution at room temperature, EIS was measured with a threecell electrochemical setup and recorded on a SquidStat Plus Potentiostat (Admiral Instruments, Tempe, AZ, USA). The reference electrode (RE) was a Ag/AgCl wire suspended in 3M NaCl solution (BASi Research Products, West Lafayette, IN, USA).

Array	Electrode	$GSA \ (mm^2)$
$M^3$	Macro	11.806
	Meso	1.118
	Micro	0.00126
Clinical	Grid/Strip	4.2

Table 2.1: Electrode Geometric Surface Area (GSA)

A 23 cm long coiled platinum wire (BASi Research Products) was used as a counter electrode (CE). The frequency was swept from 1 MHz down to 1 Hz with a 10 mV RMS AC source and a 0.5 V DC offset, determined from cyclic voltammetry. EIS was initialized and measured in the SquidStat User Interface (Admiral Instruments) and repeated three times per electrode for statistical averaging. Complex impedance data was converted, averaged, and plotted in MATLAB (MathWorks, Natick, MA, USA). Conducted in a 0.9% NaCl saline solution at room temperature, phantom ephys was measured with a three-cell electrochemical setup and recorded on a FreeLynx wireless electrophysiology system (Neuralynx, Bozeman, MT, USA). The RE was a stainless steel wire surrounding the base of an acrylic chamber, approximately 10 cm from the centered working electrode (WE). A 1 x 6 subdural ECoG strip (AD-Tech Medical) was secured to one of the acrylic walls to act as our CE, approximately 10 cm from the centered WE. Stimulation was induced with a Minirator MR2 audio generator (NTi Audio Inc, Tigard, OR, USA) across a platinum depth electrode (AD-Tech Medical), approximately 1 cm from the centered WE. Phantom signals were set to a 100 mV amplitude at 10 Hz. Baseline noise was recorded prior to phantom signal stimulation for each test. Electrophysiology recordings were initiated and recorded in the Cheetah software interface (Neuralynx) and repeated three times per electrode for statistical averaging. Phantom ephys data was converted, averaged, and plotted in MATLAB (MathWorks).

# 2.2.5 In Vivo Evaluation

Acute animal studies were conducted following ethical guidelines set by the US National Institutes of Health (NIH) and the specific protocol approved by the Institutional Animal Care and Use Committee (IACUC) of Arizona State University. Six Sprague-Dawley rats, three males and three females, were used as control and

seizure models for the study. Anesthesia was administered through a nose cone as a mixture of 2% isofluorane gas mixed with oxygen, continuously flowing at 2 liters per minute (Lpm). Consistent with protocol requirements, vitals and temperature were monitored with a MouseOx Plus pulse oximeter (Starr Life Sciences Corporation, Oakmont, PA, USA). Following a single scalp incision, a window craniotomy was opened in each parietal bone to expose both hemispheres. Windows were approximately 4 mm by 12 mm each, reaching approximately 4 mm anterior to bregma and approximately 4 mm posterior to lambda. The midline cranial bone remained intact to preserve blood flow in the superior sagittal sinus (SSS). After opening the parietal bones, the anesthetic was transitioned to a ketamine-xylazine (KX) cocktail delivered by an intraperitoneal (IP) injection. Symptomatic motor involvement was significantly reduced with isofluorane, but symptoms were preserved with a KX cocktail. After opening the dura mater, it was peeled towards the midline to prevent SSS bleeding. An M<sup>3</sup> array was slipped under the dura of one hemisphere, and a subdural strip was placed on the other. The electrophysiological recording was captured using an RHD evaluation system (Intan Technologies), including shielding from RF with a Faraday cage. A 200 µm diameter stainless steel wire, inserted under the scalp, was set as the hardware reference.

Typical anesthetic conditions were recorded for 5 minutes to represent baseline neurological waveforms (referred to as "control studies"). Pilocarpine convulsant was then delivered with an IP injection (Turski *et al.*, 1983; Clifford *et al.*, 1987; Curia *et al.*, 2008; Wetjen *et al.*, 2009) to initiate seizure symptoms ("seizure studies") with synchronized video for symptomatic confirmation. Seizures typically initiated approximately 5-15 minutes after injection, but earlier symptoms and electrographic activity were occasionally detected. Control and seizure study recordings were imported, converted, averaged, and plotted in MATLAB (MathWorks). Six M<sup>3</sup> micros with significant noise were identified through a cross-correlation and removed from calculations. The cross-correlation also revealed strikingly similar signals across all remaining channels, regardless of physical separation. Averaged baseline waveforms for each electrode size were subtracted from each channel, resulting in distinguished signals across every electrode. High-pass filters at 80 Hz, typically used to isolate HFO bands, also removed baseline waveforms for similar analysis to subtracted data. Significant events were segmented into 1-second windows for an accurate distinction of initial detection. Changes over 30% in the amplitude and power envelopes were set as thresholds for time-domain and frequency-domain analysis, respectively. The first electrode to breach the threshold in a given event was designated as the initial detection event (t=0), with all other detecting electrodes following. Times following initial detection were spatially mapped across all electrodes, with initial detection in white and electrodes without detection in black.

# 2.3 Results

# 2.3.1 Electrochemical Impedance Spectroscopy

Four subdural strip electrodes from a 1 by 4 strip and two 32-channels  $M^3$  arrays were measured with potentiostatic EIS. All electrodes were consistent throughout the three repetitions, but variance was higher from micros at frequencies above 10 kHz. Some electrodes were removed from consideration and averaging due to poor connections to the EIB. Complex impedance values depend on GSA in an inverse squared relationship, so larger electrodes should have much lower impedance than smaller electrodes. Impedance magnitude and phase for  $M^3$  and subdural strip electrodes, shown in Figure 2.2, follow these geometric expectations. Magnitudes at 1 kHz were around 300  $\Omega$ , 3 k $\Omega$ , 4 k $\Omega$ , and 300 k $\Omega$  for subdural strip electrodes, macros, mesos,



Figure 2.2: Average electrochemical impedance spectroscopy (EIS) magnitude (A) and phase (B) from the subdural strip electrodes (blue squares) and M<sup>3</sup> electrodes (red circles). Each frequency decade is highlighted from each electrode type for quick transcription.

and micros, respectively.  $M^3$  macros had the lowest corner frequency range at 10 Hz and 100 Hz, with subdural strips close behind with 20 Hz and 300 Hz. Mesos saw corner frequencies at 10 Hz and 400 Hz, and micros saw the highest corner frequency range at 30 Hz and 30 kHz.

# 2.3.2 Control Studies

Anesthetized waveforms established a baseline expectation for neurological behaviors from each animal model. Power spectrum density (PSD) values are shown in Figure 2.3 binned by Low (0-100 Hz) and HFO (100-1,000 Hz) frequency bands. Differences between these frequency bands should be expected since lower frequencies often dominate a signal spectrum. Across all electrodes, this is represented accordingly with higher PSD at lower frequencies and lower PSD at higher frequencies. Comparison between the electrodes, however, does not necessarily follow geometric and impedance expectations from EIS data. The subdural strip electrode had the highest average PSD in the lower frequency band at about 18 dB, with mesos being the closest at around 16 dB. Notably, macros had an average PSD in the lower frequency band of approximately 15 dB, while micros were at about 14 dB, despite a two-decade difference in diameter. Following expectations from impedance results, the subdural strip electrode should have the highest average PSD across the frequency spectrum. However, M<sup>3</sup> macros and mesos had the highest average PSD in the HFO frequency band, at approximately 7 dB each. The subdural strip electrode strayed from impedance expectations with an average PSD in the HFO frequency band at about 6 dB. M<sup>3</sup> micros had the lowest average PSD in the HFO frequency band at



Figure 2.3: Average power spectrum density (PSD) recorded from the subdural strip electrode (blue) and  $M^3$  array (red), shown binned by frequency range. Bands are designated as Low (0-100 Hz) and HFO (100-1,000 Hz). Errors bars show the variation in each electrode type.



Figure 2.4: Average power spectrum density (PSD) recorded from the subdural strip electrode (blue) and M<sup>3</sup> array (red), shown binned by frequency range. Bands are designated as Low (0-20 Hz), Mid (20-50 Hz), Slow Ripple (SR: 50-100 Hz), Ripple (R: 100-250 Hz), Fast Ripple (FR: 250-500 Hz), and Very Fast Ripple (500-1,000 Hz). Errors bars show the variation in each electrode type.

around 5 dB, despite specific design intentions to capture HFO activity. Interestingly, separation in individual bands, seen in Figure 2.4, further distinguishes the  $M^3$  electrodes from the subdural strip electrode, with micros and mesos the strongest above 250 Hz.

# 2.3.3 Seizure Studies

Out of six animal models studied, three models displayed distinctive symptomatic and electrographic seizures. Pilocarpine injection consistently reproduced seizure symptoms, including drooling, jaw clenching, whisker twitching, and motor involvement, but levels of anesthesia played a significant role in the prevalence of motor symptoms. All three phases of a typical seizure - pre-ictal, ictal, and post-ictal - were seen in various forms during the recorded seizure events, with some being more distinguished than others. After an averaged subtraction and a separate high-pass filter, a baseline envelope of time-domain data was set for each channel and each process type, with a threshold for detection seen in Figure 2.5. Examples of each seizure phase were found from visual inspection of time-domain and frequency-domain data and confirmation from the synchronized video recording. Plots for each significant event show the envelope detection from a 1-second time-domain segment and a spatial mapping of detection times following the initial detection. Times after initial detection are limited to a 50 ms window, unless otherwise specified, following centimeter-scale spread found from human ECoG studies (Gotman, 1983).



Figure 2.5: Subtracted time-domain detection using an envelope to evaluate times each channel crossed a threshold. Subtracted time signal is shown in black, with upper and lower envelope in dashed blue, upper and lower threshold in dotted red, and the time of threshold crossing vertically in dash-dotted cyan.

Pre-ictal and inter-ictal waveforms were the most common examples of activity outside the baseline established in the control studies. One example of a significant pre-ictal event happened about 13 minutes and 39 seconds after the pilocarpine injection, shown in Figure 2.6 Visually detected in spectrogram data under 20 Hz, this localized event was mapped using envelope detection from time-domain data with a 20% threshold. Initially seen on u19 and u21, the minimal threshold breach limited spread to a 1 mm x 1 mm region, seen on m17 and u23 shortly after the initial detection. Another example of a significant pre-ictal event occurred approximately 29 seconds after recording initiation or about 10 minutes and 29 seconds after the pilocarpine injection, as seen in Figure 2.7 and Figure 2.8. Detected visually in subtracted time data, high-pass filtered time data, and spectrogram data under 20 Hz, a 20% threshold change in the time-domain envelopes initiated detection across a localized region. Subtracted time data analysis saw initial detection on m17 with a quick spread to u19. On the other hand, high-pass filtered time data analysis showed initial detection on m17 and u19 with a spread to u21 and u23 within 10 ms. Both time-domain events depicted minimal threshold breach, limiting spread to a 1 mm x 1 mm region.

Waveforms in the ictal phase differed significantly from baseline signals in timedomain and frequency-domain data, following typical behaviors seen in clinical seizure activity. A significant example of an ictal transition event was detected around 18 minutes and 11 seconds after recording initiation or about 28 minutes and 11 seconds after the pilocarpine injection, depicted in Figure 2.9 and Figure 2.10. Visually detected on all channels across all time-domain and every frequency band, this generalized event was mapped using envelope detection from time-domain data with a 30% threshold. The threshold was increased due to false-flag events shifting the initial detection time. Subtracted time data analysis found initial detection on m32, u26,



Figure 2.6: Significant event during pre-ictal phase, about 13 minutes after a pilocarpine injection. (A) Filtered time-domain plots on all good channels with upper and lower envelope in dashed blue and time of threshold crossing vertically in dash-dotted cyan. (B) Spatial mapping of the times each electrode crossed threshold, limited to 50 ms after initial detection.

u28, and u29 with a quick spread to u5, u25, and u27, followed by m16, m17, u19, and u31. High-pass filtered time data analysis detected initial detection from m17 and u19 with a rapid spread to m32, u5, u21, u25, and u28, followed by u22, u23, u24, u27, and u29. Notably, all electrodes detect this ictal transition within 30 ms of the initial threshold breach.

During the post-ictal phase, the brain is recovering from an electrical storm and typically features rhythmic slowing and other lower frequency waveforms. For example, a post-ictal recovery burst was noticed 28 minutes and 55 seconds after recording initiation or 38 minutes and 55 seconds after the pilocarpine injection, shown in Figure [2.11] and Figure [2.12]. Highlighted by subtracted time data, filtered time data, and spectrogram data under 20 Hz, a 30% threshold in envelope time-domain data caught the generalized recovery wave. Subtracted time data analysis noted initial detection on multiple electrodes simultaneously, including m16, m17, u4, u6, u8, u10, u11, u12, u14, u19, u21, u23, u25, and u31. High-pass filtered time data analysis recognized initial detection from the subdural grid electrode, closely followed by m16, m17, u6, u8, u10, u11, u12, u14, u19, and u31. This significant event is the only distinction from the baseline first detected by the subdural grid electrode, though it was located on the opposite hemisphere than the M<sup>3</sup> array. Threshold breach was significant, and detection was seen on all electrodes from subtracted time data analysis, but some electrodes missed detection from high-pass filtered time data analysis.

Event detection outside the seizure studies was found during a control study due



Figure 2.7: Significant event during pre-ictal phase, about 10 minutes after a pilocarpine injection. (A) Subtracted time-domain plots on all good channels with upper and lower envelope in dashed blue and time of threshold crossing vertically in dash-dotted cyan. (B) Spatial mapping of the times each electrode crossed threshold, limited to 50 ms after initial detection.



Figure 2.8: Significant event during pre-ictal phase, about 10 minutes after a pilocarpine injection. (A) Filtered time-domain plots on all good channels with upper and lower envelope in dashed blue and time of threshold crossing vertically in dash-dotted cyan. (B) Spatial mapping of the times each electrode crossed threshold, limited to 50 ms after initial detection.

to a low anesthetic level before dosage adjustment. One example of such non-seizure event detection happened approximately 3 minutes and 18 seconds after electrode implantations and recording initiation, as seen in Figure 2.13. Visual detection of disturbances in subtracted time data and spectrogram data under 20 Hz enabled a 30% threshold to capture and track the spread of this non-seizure event using envelope detection from subtracted time data. A distinction was initially seen on u24, then quickly spread to u23 and u31, followed by detection on u10, u11, u21, and u25. Generalized event detection occurred outside of injection-induced seizures.

## 2.4 Discussion

As expected, impedance magnitude is inversely proportional to electrode diameter, with corner frequencies directly correlating with electrode geometry and material choices. Thicker button electrodes on the subdural strips provide more surface area for conduction than the M<sup>3</sup> macros, resulting in lower impedances as anticipated. M<sup>3</sup> macros and mesos are more comparable than geometry differences suggest, especially at higher frequencies. Micros show elevated variance compared to all other electrodes, but 10 µm traces could be a significant factor in system impedances. Based on the low-pass filter design of each electrode, HFO detection should be easier as frequency increases and electrode geometry decreases. Performance between the subdural strip



Figure 2.9: Significant event during ictal phase, about 28 minutes after a pilocarpine injection. (A) Subtracted time-domain plots on all good channels with upper and lower envelope in dashed blue and time of threshold crossing vertically in dash-dotted cyan. (B) Spatial mapping of the times each electrode crossed threshold, limited to 50 ms after initial detection.



Figure 2.10: Significant event during ictal phase, about 28 minutes after a pilocarpine injection. (A) Filtered time-domain plots on all good channels with upper and lower envelope in dashed blue and time of threshold crossing vertically in dash-dotted cyan.(B) Spatial mapping of the times each electrode crossed threshold, limited to 50 ms after initial detection.

electrodes and M<sup>3</sup> macros and mesos should be comparable throughout the frequency spectrum, though lower-frequency impedance is lowest on subdural strip electrodes.

Consistency and signal dominance from the subdural strip electrode during *in vivo* studies was expected in the 0-100 Hz range (Low) typically used clinically, confirmed by the control studies. However, the higher frequency performance was surprisingly more comparable to  $M^3$  electrodes in control studies. Geometric differences in the  $M^3$  electrodes did not equate to similar differences in spectrum power. Interestingly, the mesos consistently recorded higher spectrum power across both frequency ranges than their larger macro counterparts. Additionally, the micros detected comparable spectrum powers in both frequency bands, despite a two-decade difference in diame-

ters. Higher variance from the M<sup>3</sup> micros may suggest more localized detection across each electrode.

Pilocarpine injections consistently induce electrographic seizures, but anesthetic levels and isofluorane usage severely limited symptoms. Therefore, managing the anesthesia levels is the most crucial component of successful seizure induction with the pilocarpine model. However, this model did not present seizure waveforms in the higher-frequency ranges typical in patients or models with natural epilepsy. Most examples of event detection occurred under 100 Hz for this model. Therefore, the pilocarpine model may not be capable of microseizures. However, alternatives may show repeatable microseizure sourcing and spread, including penicillin injection in



Figure 2.11: Significant event during post-ictal phase, about 39 minutes after a pilocarpine injection. (A) Subtracted time-domain plots on all good channels with upper and lower envelope in dashed blue and time of threshold crossing vertically in dash-dotted cyan. (B) Spatial mapping of the times each electrode crossed threshold, limited to 50 ms after initial detection.



Figure 2.12: Significant event during post-ictal phase, about 39 minutes after a pilocarpine injection. (A) Filtered time-domain plots on all good channels with upper and lower envelope in dashed blue and time of threshold crossing vertically in dash-dotted cyan. (B) Spatial mapping of the times each electrode crossed threshold, limited to 50 ms after initial detection.

a porcine model (Stead *et al.*), 2010) or naturally-occurring epileptic canine models (Brinkmann *et al.*, 2015).

We believe grounding issues are apparent, exemplified by similar amplitude and timing across all electrodes with minimal time variation shown by the cross-correlation analysis. Furthermore, subtraction of the average signal, grouped by similar electrode sizes, substantially reduced the similarity between electrodes enabling analysis for timing effects. Filtering with a high-pass set to 80 Hz also reduced similarity between electrodes and produced similar, but not identical, delay measurements over space. High similarity between channels may arise from deficiencies in proper grounding and shielding of cables connecting the various systems: electrode array to Intan preamplifier, digitizer, PC, Faraday cage, etcetera. Further investigation into the grounding and shielding is necessary.

Cross-correlation is helpful in scientifically confirming the removal of noisy electrodes when the remaining electrodes are strikingly similar. If all signals are unique, cross-correlation can help identify time separation, but only if the significant event is the same waveform across multiple channels. Otherwise, an envelope detection method is necessary to distinguish time separation for unique waveforms or behaviors.

Frequency binning sizes are critical when focused on the time-based signal spread and orientation. For example, our original settings of a 1,024 sample window length



Figure 2.13: Significant event during a control study, about 3 minutes after recording initiation. (A) Subtracted time-domain plots on all good channels with upper and lower envelope in dashed blue and time of threshold crossing vertically in dash-dotted cyan. (B) Spatial mapping of the times each electrode crossed threshold, limited to 50 ms after initial detection.

and 256 samples hop distance at a 5 kHz sampling rate averaged spectrogram powers across a 51.2 ms window. Following the 50 ms cortical spread rates across a centimeter scale in human studies (Gotman, 1983), the original binning size would eliminate any timing distinction within that time window. Spatial mapping plots comparing detection times using the original settings confirmed this theory, becoming an all-ornone comparison.

The spatial spreading of activation waveforms across the brain should theoretically appear as a gradient of steadily increasing threshold-crossing delays as distance increases from the initial detection electrode. However, we did not observe this steady variation in delay compared to distance. Instead, certain events map to a small localized region while others generalize with detection on all electrodes. Pre-ictal and inter-ictal events have significant localization to the waveforms, generally found only on a few electrodes. Previous indications of HFO detection also happened in a preictal or inter-ictal state (Worrell et al., 2008) Stead et al., 2010; Worrell et al., 2012), so our localization findings have a physiological basis. Ictal transitions generalize quickly across all electrodes, though initial detection is narrowly apparent and spread is approximately spatially linear. Significant spikes across all channels are standard in clinical recordings during ictal initiation (Schevon et al.) 2008; Wetjen et al., 2009; Warren *et al.*, 2010. However, the initial detection of these ictal transition spikes is not typically analyzed clinically due to the large amplitudes. Post-ictal waveforms also show generalization, though the recovery time is different for each electrode and would be interesting to investigate further. Generalized rhythmic slowing is typical in clinical examples of post-ictal phases (Wetjen et al., 2009; Lundstrom et al., 2018), but spikes are not ordinarily present. Events during one of the control studies also showed generalization, though other non-seizure events were less generalized.

Clinical translation of the M<sup>3</sup> array will require testing in larger-animal models,

chronic studies, and scaled manufacturing. Platinum electrode arrays with polyimide substrates maintain performance for over six months of implantation (Romanelli *et al.*, 2019), though clinical implants only have approval for less than 30 days. Additionally, recent 510(k) predicates for polyimide electrodes (K211367 and K192764, NeuroOne, Eden Prairie, MN, USA) remove regulatory barriers for the potential commercialization of the M<sup>3</sup> electrodes.

Our M<sup>3</sup> electrode arrays proved that localized detection is possible with a telescopic configuration. Additionally, multiple scales of electrodes, especially 1 mm or less in diameter, pick up more distinctions than macros or subdural strip electrodes. Micros had the most useful spatial distinction, but high impedance and the number removed for poor connections or noise may mean 40 µm diameters is unnecessary. However, micros would still add spatial resolution previously missing from subdural grids or strips and deliver an improvement over the existing clinical standard. Additional bench testing is necessary to confirm spatial resolution differences between the micros and mesos, but no benchtop test methods currently have this level of precision. Finally, an investigation of stimulation parameters for the M<sup>3</sup> array may allow for localized cortical stimulation with simultaneous recording.

## Acknowledgments

This work was sponsored by a seed grant as part of the Mayo Clinic and Arizona State University Alliance for Health Care initiative. Grant money was funded by both institutions and was intended to create a tool for high density brain mapping, specific to epilepsy.

### Chapter 3

# CORTICAL STIMULATION WITH THE M<sup>3</sup> ELECTRODE: A STEP TOWARDS INTRAOPERATIVE EPILEPSY EVALUATION

### 3.1 Introduction

Subthreshold cortical stimulation has therapeutic benefits for patients with focal drug-resistant epilepsy, specifically by mapping seizure symptom locations and distinguishing functional regions from epileptic zones (Collinge et al. 2017; Lundstrom et al. (2019). Treatment options depend on region definition accuracy and proximity to the eloquent cortex, ranging from surgical resection for confident margins (Van Gompel et al., 2008; Wetjen et al., 2009) to neuromodulation implants when independent functions could be compromised (Uthman et al., 2004) Middlebrooks et al., 2018; Nair et al., 2020). Confidence intervals and accuracy evolve from the spatial resolution of electrode sources, but charge injection capacities required for stimulation restrict electrode geometries to a millimeter or grander scale (Cogan et al. 2016). Additionally, continuous recording during a stimulation pulse elicits an artifact signal, necessitating digital encoders and signal processing for elimination or suppression. Minimal scale reductions to stimulating electrodes could maintain effective charge injection while reducing the spatial reach of these artifacts. Investigations with macro-scale and micro-scale electrodes reported localized microseizures and higher-frequency oscillations (HFOs) exclusively detected on the microelectrodes (Stead *et al.*, 2010). Multiple sizes of electrodes, especially with scale options for stimulation and localized recording, may be necessary for epilepsy evaluations so that clinicians can have all the available analysis tools for decision making.

Safety should be the highest priority when considering cortical stimulation capabilities for an electrode array. Managing the heat transfer from power consumption and mitigating the possibility of current shunting directly to tissue are primary safety concerns due to the sensitivity of biological cells. Setting safe stimulation parameters requires benchmark tests such as electrochemical impedance spectroscopy (EIS), cyclic voltammetry (CV), and manual confirmation with a voltage transient. These benchmark tests, along with materials choices, factor into the water window for the electrode, which determines the voltages or currents where reduction or oxidation occurs. Additionally, the amount of tissue heating can be calculated from the power range of the water window. However, neurologic tissue should not be heated by more than 2°C (Dewhirst *et al.*) [2003), thus further limiting the stimulation parameters until a safe window is reached. Starting points for safe stimulation parameter decisions are often found in the literature where similar electrode setups are typically prevalent. Insert sources for stimulation parameters here.

Therapeutic stimulation benefits many patient populations, especially those with focal drug-resistant epilepsy. However, only a few chronic implant options are available; each is certified for a limited number of specific locations. Due to its significant therapeutic reach, deep brain stimulation (DBS) has the highest prevalence in epilepsy cases. However, implantation of DBS leads is restricted to the subthalamic nucleus (STN) and globus pallidus interna (GPi), with recent approval of the anterior nucleus of the thalamus (ANT) (Middlebrooks *et al.*) 2018). Patients with premonitions involving bradycardia or tachycardia (slow or fast heart rate, respectively) are encouraged to seek a less invasive implantation option with a vagus nerve stimulator (VNS) (Uthman *et al.*) 2004). Due to the broad, interconnected functions of the vagus nerve, connection to the afferent or efferent end will drastically change the downstream networks involved. Thus, the VNS system monitors more bodily information than any

other therapeutic implant, but the heart rate is the only one relevant to epilepsy. Recent innovations have included therapeutic stimulation with reactive sensing to close the feedback loop, called the responsive neurostimulation (RNS) device (Nair et al. 2020). Capable of learning the typical onset of the patient, RNS can adapt over time to deliver effective stimulation pulses where and when required. However, implantation locations are restricted to deep structures like the hippocampus or surface placement, though it is unclear if additional locations are approved. Finally, continuous subthreshold cortical stimulation (CSCS) using subdural grids and depth electrodes is both a short-term treatment option and a mapping tool. Patients with seizure onset zones (SOZ) near regions of the eloquent cortex require mapping of motor functions, speech regions, and symptomatic responses. This functional mapping reveals boundary regions for surgical interventions since disruptions in these areas significantly reduce the patient's independence or restrict specific functions. Notably, CSCS can only be used during a week-long epilepsy monitoring unit (EMU) visit due to an implantation limit under 30 days unless the grid is swapped with a long-term lead. Unfortunately, stimulation options do not work for all patients, and alternative treatments may still provide substantial improvement. For example, suppose a patient is incompatible with the apeutic options or has a symptomatic trigger where the implant would be placed. In that case, surgical resection has the highest percentage chance of seizure burden reduction and symptomatic improvement (Van Gompel

# *et al.*, 2008; Wetjen *et al.*, 2009).

Electrodes capable of stimulation have limited successful options for epilepsy clinically, but profound innovations were found through research objectives as simple as motor activation. For example, control of a previously paralyzed limb has been possible with stimulation, specifically from microelectrode arrays called the Utah array or Michigan array (Maynard *et al.*) [1997] Wise *et al.*] [2004). In addition, vision restoration can happen using stimulation electrodes on the retina (Christie *et al.*, 2016; Oswalt *et al.*, 2020). Multiple examples of stimulation show that electrodes can be capable of significant improvement in patient livelihood.

However, most of these examples were penetrating microelectrodes or required intricate surgery for implantation. Cortical surface stimulation could be capable of microseizure induction, as theorized by Stead *et al.* (2010), but stimulation through microelectrodes may still be a lofty goal. Microelectrodes were the only sources to detect microseizure activity, so including this submillimeter-scale is required for an electrode array seeking microseizure detection. Additionally, microseizure detection could be completed during an intraoperative procedure, recording for only a few hours instead of multiple days. Thus, infection risk and inflammation response could be practically eliminated as potential risks and costs to patients, clinical teams, and families will be reduced significantly. Previously described by Garich *et al.* (2022), the  $M^3$  array has shown promising capabilities for localized event detection with a potential to be useful for microseizure detection as well.

We evaluated current-source parameters and cortical stimulation localization results for the telescopic-configured  $M^3$  electrode array (Garich *et al.*) 2022). Compared to a subdural strip (AD-Tech Medical, Oak Creek, WI, USA), benchtop characterization of the array establishes the water windows of each electrode geometry and impedances at specific frequencies to determine safe stimulation parameters. Rodent models implanted with the  $M^3$  electrode array compare the spatial resolution of current-sourced cortical stimulation from bipolar combinations of the  $M^3$  array. Due to the lack of epileptic tissue in seizure-prone rodent models used by Garich *et al.* (2022), microseizure induction is impossible in this study.



Figure 3.1: Microscope image of a 32-channel M<sup>3</sup> electrode (Garich *et al.*) 2022).

# 3.2 Materials and Methods

## 3.2.1 Design and Interface

 $M^3$  electrodes were designed by Garich *et al.* (2022) and fabricated in Arizona State University (ASU) cleanrooms, with a detailed microscope view in Figure 3.1. Benchmark testing and characterization showed comparable performance to subdural strip electrodes. The 32-channel  $M^3$  array used in rat models has one 4 mm macro, four 1 mm mesos, and twenty-seven 40 µm micros. PI-2611 polyimide arrays are spincoated to 10 µm thick per layer, for a total stack thickness of 20 µm. Connection to a flexible printed circuit (FPC) zero-insertion force (ZIF) connector (Molex Connector Company, Lisle, IL, USA) occurs across a custom electrode interface board (EIB), with post connections for each electrode. Polyether ether ketone (PEEK) sheets were adhered to the M<sup>3</sup> arrays with cyanoacrylate and cut to size to ensure repeatable connectivity in the FPC ZIF connector.

# 3.2.2 Benchtop Characterization

Cyclic voltammetry (CV) was conducted in a 1x phosphate-buffered saline (PBS) solution at room temperature. The three-cell electrochemical setup was recorded inside a Faraday cage on a SquidStat Plus Potentiostat (Admiral Instruments, Tempe, AZ, USA). The reference electrode (RE) was a Ag/AgCl electrode in 3M NaCl solution (BASi Research Products, West Lafayette, IN, USA). The counter electrode (CE) was a 23 cm long coiled platinum wire (BASi Research Products). The applied potential across the working electrode (WE) was swept from +1 V to -1 V at a 50 mV/s rate, resulting in a current-voltage relationship. CV was set up and measured in the SquidStat User Interface (Admiral Instruments) and repeated three times per electrode for sufficient averaging. Cyclic voltammograms were converted, averaged, and plotted in MATLAB (MathWorks, Natick, MA, USA). Stimulation parameters were set based on the platinum water window (-0.6 to 0.8 V in reference to Ag/AgCl (Hudak *et al.*) [2010)), CV parameters, and previously published cortical stimulation literature (Wang *et al.*] [2017).

# 3.2.3 In Vivo Evaluation

Surgical procedures followed guidelines established by the US National Institutes of Health (NIH) and protocols approved for acute rodent studies by the ASU Institutional Animal Care and Use Committee (IACUC). Two Sprague-Dawley rats, one male and the other female, were anesthetized, and a window craniotomy was

opened on one hemisphere. Anesthesia was maintained using a mix of 2% isofluorane gas mixed with oxygen, continuously administered through a nose cone at 2 liters per minute (Lpm). Vitals and temperature were observed on a MouseOx Plus pulse oximeter (Starr Life Sciences Corporation, Oakmont, PA, USA). The craniotomy window began about 4 mm posterior to lambda and ended around 4 mm anterior to bregma, resulting in a 4 mm by 12 mm window. The superior sagittal sinus (SSS) was left intact along with the midline cranial bone to preserve blood flow. Isofluorane was diminished in favor of an intraperitoneal (IP) injection of a ketamine-xylazine (KX) cocktail to preserve motor responses. The dura mater was opened and peeled towards the midline, and the  $M^3$  array was placed on the cortex. A 200 µm diameter stainless steel wire was placed under the scalp as a reference. Bipolar stimulation was conducted across two M<sup>3</sup> electrodes through a Model 2100 isolated pulse stimulator (A-M Systems, Sequim, WA, USA). Biphasic pulses were set to a 200 µs pulse width, a 10 ms interstimulus interval, and a 100 ms train duration, with 1 second between trains. Amplitude was gradually increased until a stimulus was observed or 4 mA was reached, whichever occurred first, to obtain a minimal cortical activation (MCA) limit. One animal model was utilized for stimulation across three of the  $M^3$  mesos and one micro. The second animal model was part of a second stimulation attempt from M<sup>3</sup> macros. However, the available arrays did not have a complete connection to the macros and could not deliver a stimulus. Instead, this second animal model was transitioned to alternative investigations within the research group.



Figure 3.2: Stimulation parameters extracted for each electrode type with subdural strip electrodes (blue) and  $M^3$  electrodes (red) clearly indicated.

3.3 Results

# 3.3.1 Cyclic Voltammetry

Cyclic voltammetry results were consistent across  $M^3$  macros and mesos, showing hydrogen plating and oxide reduction peaks as expected for platinum. On the other hand, subdural strip electrodes only showed a minimal oxide reduction peak, possibly indicative of a narrow potential range. Impedance differences could partially explain the lateral voltage shift between  $M^3$  and subdural strip electrodes, with EIS results demonstrated by Garich *et al.* (2022). Curves for all electrodes follow typical water window patterns, with -0.6 to 0.8 V being standard for platinum electrodes in reference to a Ag/AgCl electrode.

# 3.3.2 In Vivo Motor Stimulation

Stimulus pulse widths of 100 µs were attempted initially but did not produce a motor effect successfully. Multiple bipolar electrode combinations were tested, but only three mesos and one micro resulted in motor responses. The forelimb paw was stimulated across m16 and m17, diagonally opposed across the array, at a minimum cortical activation (MCA) of 2.6 mA. A chest or throat response was noticed at an MCA of 0.9 mA across m17 and u15, diagonally opposed across the array. However, the same bipolar electrode combination produced a jaw response distinctively at 1.2 mA of MCA. Across m2 and m16, laterally aligned on the rostral side of the array, whisker involvement and wrist pronation occurred at an MCA of 3.2 mA.

The M<sup>3</sup> array was shifted 2 mm caudal to the initial placement to seek additional motor mapping. Hindlimb paw activation was found from an MCA of 2.4 mA across m16 and m17, diagonally opposed across the array. Stimulation across m2 and m16, laterally aligned on the rostral side of the array, produced forelimb paw and jaw involvement with an MCA of 3.6 mA. After testing each electrode combination with anodic-first and cathodic-first polarities, it seems anodic pulses were the primary stimulus drivers in this setup.

### 3.4 Discussion

Current-voltage relationships in CV tests confirm similarities between the  $M^3$  macros and mesos. Electrochemical stability exists between +0.3 to +1 V for both  $M^3$  macros and mesos. At the same time, the subdural strip electrodes are stable from -0.3 to +0.5 V. Current densities within the range of -0.01 to +0.02 mA/cm<sup>2</sup> are in the stability window for macros and mesos. Subdural strip electrodes are stable within current densities of -2.0 to +0.5 mA/cm<sup>2</sup>. Charge injection for  $M^3$  macros and

mesos was significantly less than for subdural strip electrodes, with 50 µm and 20 µm traces a significant limitation for macros and mesos, respectively. Voltage transients confirmed functionality from all three electrode types up to 4 mA of delivered current, but the macros and mesos quickly became unstable at currents above this.

Initial array placement seems to access muscle groups from forelimb to facial in the primary motor cortex of the rat model. In addition, the distinction between the lateral and diagonal orientations for bipolar stimulation seems to access different regions of motor activation. For instance, rostral lateral orientation recruited activation from the whiskers and forelimb wrist, while a diagonal orientation recruited involvement from the forelimb paw, jaw, and chest. Interestingly, stimulation across a single micro and a meso, diagonally opposed across the array, was possible and produced the most distinct muscle activation out of all electrode combinations. Though it is uncertain



Figure 3.3: Bar graph comparing minimal cortical activation currents for various muscle groups from the original array placement. (Insert) Drawing of array location relative to rat cortex with stimulating electrodes highlighted.



Figure 3.4: Bar graph comparing minimal cortical activation currents for various muscle groups from a second array placement. (Insert) Drawing of array location relative to rat cortex with stimulating electrodes highlighted.

if this activation involves the throat or the chest, capabilities of stimulating across a 40 µm diameter micro are novel findings for a surface electrode.

Shifting the  $M^3$  array placement 2 mm caudal from the initial placement confirms motor maps previously established (Neafsey *et al.*) [1986; Fonoff *et al.*], [2009). Forelimb activation exists rostral to hindlimb activation while joint activations are aligned laterally from the midline. Changes in activation were noticed when polarity was switched between the anode and cathode; the dipole orientation of the source may influence activation networks. Further investigation into the bipolar source orientation may provide higher spatial resolution to motor mapping.

The  $M^3$  electrode arrays created by Garich *et al.* (2022) proved that cortical stimulation of the primary motor cortex in a rat model is possible. Motor responses vary in activation level depending on location, with certain muscle groups capable of distinctive activation while others have subtle involvement. Spatial separation from the mesos provides an additional level of resolution previously unavailable from subdural grids and strips. Additionally, due to the reduced scale, the mesos can rotate to induce various orientations of dipole sources, but physiological relevance is not confirmed. Further investigations seeking comparisons between the subdural strip electrodes and M<sup>3</sup> macros and mesos are necessary to link to the current clinical comparative standard. Finally, simultaneously testing stimulation and recording capabilities, especially with a naturally-occurring seizure model with detectable microseizures, should complete the verification of the M<sup>3</sup> array's capabilities for clinical use or commercialization.



Figure 3.5: Scatter plot of minimal cortical activation current by electrode combination. Each muscle group is represented by a different color, with forepaw (red), throat (green), jaw (blue), whisker (yellow), forewrist (magenta), and hindpaw (cyan). Activations from the first array location have triangle markers and activations from the second array location have circle markers.



Figure 3.6: Drawing of rat motor cortex implant locations with estimated motor regions labeled in corresponding colors.

# Acknowledgments

This work was sponsored by a seed grant as part of the Mayo Clinic and Arizona State University Alliance for Health Care initiative. Grant money was funded by both institutions and was intended to create a tool for high density brain mapping, specific to epilepsy.

#### Chapter 4

# A BENCHTOP SPATIAL TEST BED FOR QUANTIFYING SPATIAL RESOLUTION OF SUBDURAL ECOG ARRAYS

Jonathan V Garich<sup>1,2,3</sup>, Ian R Akamine<sup>1</sup>, Seth A Hara<sup>2</sup>, Daniel W Gulick<sup>3</sup>, Mark A Benscoter<sup>2</sup>, Stephen T Kuehn<sup>2</sup>, Gregory A Worrell<sup>4</sup>, Gregory B Raupp<sup>5</sup>, Jennifer M Blain Christen<sup>1,3</sup>

<sup>1</sup>Biomedical & Health Systems Engineering, Arizona State University, Tempe, AZ, <sup>2</sup>Division of Engineering, Mayo Clinic, Rochester, MN,

<sup>3</sup>Electrical, Computer, & Energy Engineering, Arizona State University, Tempe, AZ, <sup>4</sup>Department of Neurology, Mayo Clinic, Rochester, MN,

<sup>5</sup>Engineering of Matter, Transport, & Energy, Arizona State University, Tempe, AZ

Paper to be submitted to a peer-reviewed indexed conference proceeding yet to be determined with approval from Co-Authors Ian Akamine, Seth Hara, Daniel Gulick, Mark Benscoter, Stephen Kuehn, Gregory Worrell, Gregory Raupp, and Jennifer Blain Christen.

#### ABSTRACT

Objective Intracranial electrodes, often called electrocorticography (ECoG) electrodes when recording from the cortical surface, come in multiple geometries and configurations, but the gaps between standardized benchmark tests and the complexities involved in *in vivo* studies leave interpretation up to the individual researchers. Techniques such as phantom electrophysiology exist to bridge this gap and yet there is no standard configuration for these test systems and source-to-target distances are never precise. Inconsistency such as this makes direct comparison between different electrode configurations difficult. Approach We present a standardized benchtop test method, called the Spatial Test Bed, using a series of 41 concentric electrodes with known distances between each source electrode and the target electrodes in all three axes. Electrode diameters of 350 µm and spacing ranging from 200 µm to 2 mm, controlled by 0.5 mm gaps between each concentric ring of six electrodes were used. These spatial test bed electrodes were compared to subdural grid electrodes in standard benchmark testing. Stimulation from the Spatial Test Bed through a saline environment sealed in an enclosure printed with stereolithography (SLA), a controlled version of phantom electrophysiology, was used to compare detection differences between subdural grids and a telescopic hybrid  $M^3$  electrode array recently developed. Main results Stimulation parameters and complex impedances for both the Spatial Test Bed and subdural grids were found to have no significant differences between them. This validated the Spatial Test Bed materials and fabrication process. Then, the test bed was used to compare signal detection between ECoG arrays. Single-source tests showed better detection capabilities from the higher density of M<sup>3</sup> electrodes as source-to-target distance increased, confirming design-based assumptions, and higher frequency sources were easier to distinguish with smaller M<sup>3</sup> electrodes. Multi-source tests highlighted these differences further as the source-to-source distance decreased and independent detection was achieved on the  $M^3$  array but not the subdural grid. Epileptogenic source tests aligned with the expectation that larger electrodes had too much summation to distinguish individual signals while smaller electrodes were capable of independent signal identification. *Significance* Signal detection from the  $M^3$  macro (4mm diameter) and subdural grid electrodes (2.3 mm diameter) matched closely throughout testing while significant spatial localization capabilities were found for the  $M^3$  mesos (1 mm diameter) and micros (40 µm diameter), implying that signals occurring at spatially distinct cortical columns or locally-networked neuronal regions could be detected separately. Spatial resolution could be quantified, extracted, and indexed into a database from this benchtop Spatial Test Bed for data-driven comparison of any electrode intended for implantation in the subdural space, allowing for a standardized test method bridging the gap between standard benchmark tests and *in vivo* complexities.

# 4.1 Introduction

Electrodes used for intracranial monitoring are offered in many variations, depending on the application, ranging from centimeter-scale to submillimeter-scale geometries. For example, electrocorticography (ECoG) grids span a 1 cm gap between electrodes to sample from multiple regions across the cortex (Penfield and Jasper [1954]). On the other hand, microelectrodes like the NeuroGrid (Khodagholy *et al.*) 2015) or Neuropixels (Jun et al. 2017) match anatomical geometries with 10 µm electrodes to achieve action potential levels of detection. However, deciding the spatial resolution for each application is not as simple as it should be. Epilepsy cases, for example, have synchronized networks across the cortex (Freeman et al., 2000; Kandel et al. 2000) and localized higher-frequency oscillations (HFO) (Worrell et al. 2008) 2012; Brázdil et al., 2017), where multiple spatial resolutions are involved. Benchmark characterization (Gamry, 2021) and simulations (Zhang et al., 2008; Freeman and Zhai 2009) can help estimate the spatial resolution(s) an application could require. However, other bench tests, such as phantoms, are often rudimentary and lack backing in literature. Therefore, a benchtop test method with the simplicity and stability of characterizations, an accurate environment like a phantom, and a capability of simulating *in vivo* intricacies would be capable of establishing benchmark tests to quantify ideal spatial resolutions for each application.

Standard benchmark tests, such as electrochemical impedance spectroscopy (EIS) and cyclic voltammetry (CV), are predictive estimates of electrode performance. However, these characterizations lack the complexity required to understand behaviors in physiological environments, especially for intricate systems like the central nervous system. Additionally, geometrical differences show more significant changes in electrode performance from benchmarks, while phantoms and *in vivo* studies typically
have more minor distinctions between electrodes of varying sizes (Garich *et al.*) [2022). Phantoms can replicate signals similar to physiological behaviors, but precision in test setup is typically lacking due to the fluidic test environment. However, simulating complex signals requires electronic hardware that combines multiple signals with various amplitudes, frequencies, and phases.

We present a standardized benchtop test method called the Spatial Test Bed. This testbed intends to establish predictable signal analysis and provide a testable platform for electrodes with various spatial resolutions. Concentric rings of 350 µm diameter electrodes, with five rings of electrodes, spaced 0.5 mm apart, encompass a 5 mm diameter for precise control over source-to-source and source-to-target distances. Printed with stereolithography (SLA), the enclosure system keeps the saline-filled electrode test chamber separate from the watertight electrical connections. Standard benchmark tests for complex impedance and stimulation parameters compare the Spatial Test Bed to subdural ECoG grids as a standard clinical electrode. In addition, precision phantom tests of changes in amplitude and frequency should confirm testbed performance compared to previous phantoms. Finally, source-to-target and sourceto-source variations should set the Spatial Test Bed apart from previous phantoms, with precise control over where signals are sourced.

#### 4.2 Materials and Methods

#### 4.2.1 Design

The Spatial Test Bed was designed with five concentric circles of electrodes spaced 0.5 mm apart, as depicted in Figure 4.1 Each ring consistents of six electrodes for a total of 41 electrodes, including the center. This testbed spans a 5 mm diameter to encompass a subdural grid electrode fully. Platinum electrodes have a 250 µm



Figure 4.1: Spatial Test Bed electrode chrome mask design with the center electrode design highlighted with more detail (insert).

diameter with rounded corners to prevent edge effects. Electrode pitch varies with each concentric ring, ranging from 350 µm to 1.25 mm center-to-center. Large 3 mm by 8.5 mm contact pads allow for repeatable connection without extreme precision.

#### 4.2.2 Fabrication

A 100 mm diameter silicon wafer served as the carrier substrate for the Spatial Test Bed electrodes. First, through plasma-enhanced chemical vapor deposition (PECVD), a 100 nm layer of SiO<sub>2</sub> was deposited at 350°C to insulate from the silicon. Next, electron-beam deposition of 100 nm platinum with a 5 nm titanium adhesion layer was patterned using lift-off photolithography. Finally, the conductive layer was sealed with another 100 nm layer of SiO<sub>2</sub> using a PECVD system, and electrode contacts were exposed using a CHF<sub>3</sub> plasma etch. All photoresist was removed through an ashing system and cleaned prior to each sequential step.



Figure 4.2: SLA-printed electrophysiology enclosure system with labeled trays for each component. O-rings keep the system watertight and bolts ensure a complete seal.

#### 4.2.3 Enclosure System

An enclosure was designed and built using stereolithography (SLA) to fit the Spatial Test Bed wafer, connections, a saline chamber, and trays for the electrode arrays, shown in an exploded view in Figure 4.2 O-rings separated electrical connections with the saline chamber to prevent shorting. Connection to the wafer relied on goldcoated compression pins with a 2 mm plunger length, soldered to 28 AWG wires. All wires exited the enclosure through a single channel and were labeled for the respective electrode connections. Multiple depths of saline chambers were designed to control the z-axis during electrode testing. Electrode trays for M<sup>3</sup> arrays and subdural grids centered the electrodes over the Spatial Test Bed and allowed precise control over movement away from the center. Finally, a labeled top cover was secured with bolts through all layers of the enclosure to ensure everything was watertight.

#### 4.2.4 Test Bed Characterization

Cyclic voltammetry (CV) tests were conducted with a SquidStat Plus Potentiostat (Admiral Instruments, Tempe, AZ, USA) in 1x phosphate-buffered saline (PBS) at room temperature. A three-cell electrochemical setup used a Ag/AgCl wire submerged in 3M NaCl solution (BASi Research Products, West Lafayette, IN, USA) as a reference electrode (RE). The counter electrode (CE) was a 23 cm length of coiled platinum wire (BASi Research Products). An applied potential on the working electrode (WE) was swept over a -1 to +1 V range at a 50 mV/s scanning rate. CV was initialized and recorded using a SquidStat User Interface (Admiral Instruments) and repeated three times for sufficient averaging. Voltammogram plots were converted, averaged, and plotted in MATLAB (MathWorks, Natick, MA, USA). Potentiostatic electrochemical impedance spectroscopy (EIS) summarized electrode performance compared to subdural ECoG grid electrodes. Recorded in a SquidStat Plus Potentiostat (Admiral Instruments), the same three-cell electrochemical cell from CV testing was submerged in room-temperature 1x PBS. The frequency was swept from 1 MHz down to 1 Hz with a 10 mV AC RMS input and no DC bias, based on stability from the CV data. EIS was set up and measured in the SquidStat User Interface (Admiral Instruments) and repeated three times per electrode for complete statistical analysis. Complex impedance data was converted, averaged, and plotted in MATLAB (MathWorks). The typical water window for platinum, typically -0.6 to +0.8 V with a Ag/AgCl reference, and CV and EIS testing results determined safe stimulation parameters. Voltage transients in PBS were tested prior to electrode placement to ensure adequate stimulation.

#### 4.2.5 Electrode Evaluation

Stimulation was conducted across a bipolar combination of Spatial Test Bed electrodes using an Arbitrary Waveform Generator (Keithley, Cleveland, OH, USA). Pulse widths were set to 250 µs, and separate parametric amplitude and frequency sweeps were conducted from 10 mV to 100 mV and 10 Hz to 2,000 Hz, respectively. Frequencies were divided into the bands typical in HFO detection for simple translation to previous studies with the  $M^3$  array (Garich *et al.*, 2022). Distances between bipolar electrode connections were also varied to determine spatial distinction as sources moved away from array centers. Noise recordings were conducted over 10 seconds without stimulation across the Spatial Test Bed electrodes. Signals were recorded at a 5 kHz sampling rate inside a Faraday cage using an RHD2000 evaluation system (Intan Technologies, Bozeman, MT, USA). Similar to the time-domain analysis Garich et al. (2022) conducted with the M<sup>3</sup> arrays, the averaged signal for each electrode type was subtracted from every channel to remove baseline interference, likely due to grounding issues. Envelope detection was used to calculate amplitudes across the source and noise signals, resulting in the signal-to-noise ratio (SNR). All data was imported, averaged, and plotted in MATLAB (MathWorks).

#### 4.3 Results

#### 4.3.1 Cyclic Voltammetry

Cyclic voltammetry results, shown in Figure 4.3 were consistent across subdural grid electrodes, showing hydrogen plating and oxide reduction peaks as expected for platinum electrodes. Spatial Test Bed electrodes, on the other hand, only showed a maximal hydrogen absorption peak and may indicate the presence of electromagnetic interference or incomplete connection within the electrochemical cell. The lateral



Figure 4.3: Cyclic voltammogram (CV) comparing water windows and currentvoltage relationships between Spatial Test Bed electrodes (A) and subdural grid electrodes (B).

voltage shift between the subdural grid and Spatial Test Bed electrodes could be caused by impedance differences, which will be seen in electrochemical impedance spectroscopy. Curves for all electrodes follow standard water window patterns, with -0.6 to 0.8 V being standard for platinum electrodes with a Ag/AgCl reference electrode.

#### 4.3.2 Electrochemical Impedance Spectroscopy

Electrochemical behavior was measured from eight subdural grid electrodes and eleven Spatial Test Bed electrodes. Measurements were consistent throughout testing and reflected appropriate responses throughout the electrodes tested. The complex impedances for subdural grid electrodes follow geometric expectations and stability, as shown in Figure 4.4. Surprisingly, the smaller and thinner Spatial Test Bed electrodes produced lower impedances throughout the frequency range and showed



Figure 4.4: Electrochemical impedance spectroscopy (EIS) comparing complex impedance magnitude (A) and phase (B) between Spatial Test Bed electrodes (green) and subdural grid electrodes (blue).

comparatively more stable phase performance. Magnitudes at 1 kHz were comparable at 300  $\Omega$  and 200  $\Omega$  for subdural grid electrodes and Spatial Test Bed electrodes, respectively. Corner frequencies of 1 kHz and 300 Hz for subdural grid electrodes and Spatial Test Bed electrodes, respectively, followed similar behaviors of thin-film platinum and button-style platinum shown previously (Garich *et al.*) 2022).

#### 4.3.3 Electrode Evaluation

#### Source Amplitude Dependence

Increasing source amplitude should correlate to an increasing signal from each electrode, but impedance may affect the amount of change in signal. As seen in Figure 4.5, the M<sup>3</sup> electrodes follow this amplitude expectation, with the macro detecting the highest amplitude with an SNR of 24 at 100 mV. Interestingly, the signal comparison



Figure 4.5: Sourced across bipolar electrodes with 4.5 mm separation (NW25-SE20), changes in signal-to-noise ratio (SNR) were tracked as source amplitude increased. Insert shows the source locations on the Spatial Test Bed.

between the micros, mesos, and macro are insignificant at lower amplitudes, regardless of differences in geometry. For instance, the macro had an SNR close to zero at 10 mV, while micros had an average SNR around 1, and mesos recorded an SNR of 4. Additionally, the subdural grid electrodes remained consistent despite increasing source amplitude, with an SNR of approximately 5 across the amplitudes. Notably, the lower limit on the waveform generator was 10 mV, so typical physiological amplitudes were impossible as a source.

#### Source Frequency Dependence

As frequency increases, signals should decrease in power due to the limited spatial spread of the higher-frequency waveforms. Typically, ECoG grids are low-pass filtered at 100 Hz to eliminate the higher-frequency noise standard in hospital patient monitoring rooms. As depicted in Figure 4.6, the performance of the subdural grid



Figure 4.6: Sourced across NW25 and SE20 electrodes, with 4.5 mm separation, changes in SNR were tracked as source frequency increased. Insert shows the source locations on the Spatial Test Bed.

electrodes under 200 Hz follows expectations with an SNR of about 6. However, the SNR of the subdural grid electrodes at higher frequencies is much lower at around 2 at 1 kHz. On the other hand, M<sup>3</sup> electrodes had significantly higher SNR at 1 kHz with 12, 14, and 16 for the micros, mesos, and macro, respectively. However, the M<sup>3</sup> electrodes did not record high SNR in the lower frequencies, with 5, 3, and 3 for mesos, micros, and the macro, respectively.

#### Source Distance Dependence

Distance is a crucial component of signal detection for electrodes, especially ECoG electrodes in the subdural space. As an electrode gets further from a source, the signal dissipates gradually before becoming undetectable. Figure 4.7 shows a test of this source movement at a frequency of 100 Hz to test lower-frequency behavior. Unfortunately, a 100 Hz signal's spatial reach is about equivalent to the electrode

arrays' size, so minimal SNR change was seen across the distance adjustments. Subdural grid electrodes had an SNR of around 1.5 until the source was 2.5 mm from the center, where SNR increased to 1.75. Notably, the subdural grid electrodes were not positioned directly over the Spatial Test Bed, so this increase at a further distance is expected. M<sup>3</sup> electrodes detected much lower SNR throughout the source distances, with mesos having the highest SNR of 1 at 1 mm from the center, while the macro peaked at an SNR of 0.5 at 2 mm from the center.

Alternatively, higher-frequency signals should depend on spatial proximity, as shown in Figure 4.8. The subdural grid electrodes were still reasonably consistent throughout the distances, with a maximum SNR of 3.5 at 1 mm from the center and a minimum SNR of 0.75 at 2 mm from the center. M<sup>3</sup> mesos follow a similar trend, with consistent SNR around 1.5 throughout the distances, though it tapers off at more than 2 mm from the center. However, the M<sup>3</sup> macro has a more significant taper throughout the distances, with much higher SNR at closer distances and much lower SNR at further distances. As expected, the micros have varied SNR across distances, but this tracks with the design intentions of the M<sup>3</sup> micros. Though it is not depicted here, comparing SNR mapped across the electrode arrays would be a good visualization of where the signal originates.

#### 4.4 Discussion

Benchtop characterization of the Spatial Test Bed does not follow expectations based on design. The subdural grid electrodes are stable across testing as expected. However, despite a smaller electrode diameter, the Spatial Test Bed electrodes exhibit a much lower impedance magnitude, though the phase is unstable. Additionally, the CV plots for the Spatial Test Bed electrodes did not carry relevant information. Notably, the enclosure seals were problematic during EIS and CV testing, poten-



Figure 4.7: Moving sources axially away from center, from 0.5 mm from center to 2.5 mm from center, changes in SNR were tracked from a 100 Hz source, with each source location plotted separately. (A) Every electrode is plotted in relation to source distance for each source location, with labels for significant SNR. (B) Spatial mapping of STB source and electrode detection for each source location.

tially inducing shorts across the connections. Unfortunately, not much information is trustworthy from the Spatial Test Bed during benchmark testing. Potential errors may exist in fabrication and enclosure design or sealing and would be corrected if submission timing was not an issue.

Grounding issues remain significant with the recording setup described by Garich et al. (2022). Subtracting the average signal from each electrode type enables successful post-processing, but the system requires further investigation. Nevertheless, amplitude tests follow expectations, especially from the  $M^3$  electrodes with a nearly linear slope. On the other hand, subdural grid electrodes maintain a similar signal while amplitude increases. There is a possibility that the subdural grid electrodes



Figure 4.8: Moving sources axially away from center, from 0.5 mm from center to 2.5 mm from center, changes in SNR were tracked from a 1,000 Hz source, with each source location plotted separately. (A) Every electrode is plotted in relation to source distance for each source location, with labels for significant SNR. (B) Spatial mapping of STB source and electrode detection for each source location.

have a low saturation limit, seen at around 20 mV, though this situation is unlikely. Frequency tests follow expectations throughout the electrodes in testing. The  $M^3$  electrodes can detect HFO signals better than the subdural grid electrodes, but the subdural grid electrodes dominate low-frequency performance. Surprising capabilities for HFO detection come from the macro, though performance should closely match that of subdural grid electrodes. Distance testing with a 100 Hz source is consistent across all electrodes, likely due to the maximum spatial spread of a lower-frequency signal. However, distance testing with a 1,000 Hz source follows expectations, likely due to the significant localization of a higher-frequency signal. Subdural grid electrodes lose signal as distance increases, though the orientation of the grid means another electrode starts picking up the signal at the furthest distance. The  $M^3$  macro also loses signal as distance increases, with no alternative orientation to pick it up again. Following expectations, the  $M^3$  mesos and micros track the signal as it moves across the array, though it is difficult to visualize without a mapping. Spatially mapping these signals as they move across the arrays would be helpful for further analysis.

This Spatial Test Bed system proves that signal tracking from a benchtop test method is possible. Of course, electrode fabrication and enclosure design will need another evaluation to fix the issues with waterproofing and grounding, but the test method works. Spatial mapping analysis of the detected signals will add a muchneeded perspective to the Spatial Test Bed electrodes. Further testing should include multiple bipolar sources and track changes as they move across an electrode array. Additionally, this movement detection can be confirmed using MATLAB to sum multiple sources mathematically and compare them to the physical detection of multiple sources. Unfortunately, the lack of trust in the Spatial Test Bed in its current state does not lend confidence in pushing this as a new benchtop standard test. However, addressing the fabrication and enclosure problems could be all that is necessary to utilize this novel phantom as a repeatable standard to test all surface electrodes.

#### Acknowledgments

This work was sponsored by a seed grant as part of the Mayo Clinic and Arizona State University Alliance for Health Care initiative. Grant money was funded by both institutions and was intended to create a tool for high density brain mapping, specific to epilepsy.

#### Chapter 5

#### **OUTLOOK & SUMMARY**

Chapter 2 discussed the development, characterization, and seizure model testing of the novel telescopic hybrid  $M^3$  electrode array, with three levels of electrode geometries, compared to subdural strip electrodes as a clinical equivalent. Findings from this study revealed spatial distinction from specific examples of seizure and non-seizure events. Localization was found on some waveforms, while others were generalized across the entire electrode array. Pilocarpine-injected seizure models developed similar symptoms to those seen clinically, including jaw clenching, drooling, and limb movement. Seizure symptoms were detected electrographically and confirmed with synchronized video. However, this injection-based model differs from naturallyoccurring epilepsy with minimal activity in higher-frequency oscillation (HFO) bands. Typically, HFO activity localizes near epileptogenic tissue, so the unnatural seizure activation could be the reason for this activity difference. Notably, the lack of HFO activity and no natural epileptogenic source also means this model is incapable of microseizure detection. Future studies shall include larger-animal studies utilizing a 128-channel version of the M<sup>3</sup> array and a subdural grid with at least four electrodes in cortical contact. Additionally, studies involving larger-animal models with naturally-occurring epilepsy will aid in tracking HFO regions and spread, possibly targeting postoperative biopsy confirmation of epileptic tissue. Chapter 2 will be submitted for publication in the Journal of Neural Engineering in the coming months once the Mayo-ASU team concludes editing and formatting figures.

Investigating stimulation parameters and characteristics between the  $M^3$  electrode array and subdural strip electrodes, Chapter 3 is not currently seeking publication but may evolve into publishable material with additional animal testing. Stimulation parameters matched expectations, given known values from the subdural strip electrodes, and stimulation of cortical tissue elicited a motor response. In addition, M<sup>3</sup> mesos were capable of selecting individual muscle groups to activate based on placement location. Unfortunately, macros were not adequately connected and were not tested for stimulation capabilities. However, injecting current across a micro and a meso revealed distinctive activation of anatomical structures not seen with other electrode combinations. Additionally, the orientation of the anode and cathode changed the activation group targeted by cortical stimulation. Further evaluation of the M<sup>3</sup> electrode is necessary for a naturally-occurring animal model to detect and track microseizures and other seizure-specific waveforms. Finally, a simultaneous stimulation and recording test would complete the specifications verification of the M<sup>3</sup> electrode array. If this information and background work seeks publication in the future, the author requests co-authorship for the investigations, characterization, and writing already completed.

This culmination of work showed the complexities of inventing a new medical product from the ground up. From determining stakeholder needs, use cases, design requirements, engineering specifications, and verification testing, this project gave me the experience needed to be a competent engineer in a medical devices company. Issues encountered along the way required perspective of the entire system for definitive solutions. Project management was a significant portion of the experience, providing experience in working with clients outside of my institution and organizing and managing the daily tasks required to meet the grant funding goals. During this process, I participated in a fellowship at Mayo Clinic, where I worked on a medical engineering team directly interfacing with clinicians in multiple departments of care and specialization. This fellowship experience prepared me significantly for my upcoming career path, continuing in the medical devices engineering field.

#### REFERENCES

- Araki, T., H. Otsubo, Y. Makino, I. Elliott, K. Iida, A. Ochi, S. K. Weiss, S. H. Chuang, J. T. Rutka and O. C. Snead, "Efficacy of dexamathasone on cerebral swelling and seizures during subdural grid eeg recording in children", Epilepsia 47, 176–180, URL http://doi.wiley.com/10.1111/j.1528-1167.2006.00384.x (2006).
- Blakely, T., K. J. Miller, R. P. Rao, M. D. Holmes and J. G. Ojemann, "Localization and classification of phonemes using high spatial resolution electrocorticography (ecog) grids", Proceedings of the 30th Annual International Conference of the IEEE Engineering in Medicine and Biology Society, EMBS'08 - "Personalized Healthcare through Technology" pp. 4964–4967 (2008).
- Bourdillon, P., P. Ryvlin, J. Isnard, A. Montavont, H. Catenoix, F. Mauguière, S. Rheims, K. Ostrowsky-Coste and M. Guénot, "Stereotactic electroencephalography is a safe procedure, including for insular implantations", World Neurosurgery 99, 353–361 (2017).
- Brinkmann, B. H., E. E. Patterson, C. Vite, V. M. Vasoli, D. Crepeau, M. Stead, J. J. Howbert, V. Cherkassky, J. B. Wagenaar, B. Litt and G. A. Worrell, "Forecasting seizures using intracranial eeg measures and svm in naturally occurring canine epilepsy", PLOS ONE 10, e0133900, URL https://journals.plos.org/plosone/ article?id=10.1371/journal.pone.0133900 (2015).
- Brázdil, M., M. Pail, J. Halámek, F. Plešinger, J. Cimbálník, R. Roman, P. Klimeš, P. Daniel, J. Chrastina, E. Brichtová, I. Rektor, G. A. Worrell and P. Jurák, "Very high-frequency oscillations: Novel biomarkers of the epileptogenic zone", Annals of Neurology 82, 299–310, URL http://doi.wiley.com/10.1002/ana.25006 (2017).
- Campbell, A. and C. Wu, "Chronically implanted intracranial electrodes: Tissue reaction and electrical changes", Micromachines 2018, Vol. 9, Page 430 9, 430, URL https://www.mdpi.com/2072-666X/9/9/430/htmhttps://www.mdpi.com/2072-666X/9/9/430 (2018).
- Casaletto, M. P., G. M. Ingo, S. Kaciulis, G. Mattogno, L. Pandolfi and G. Scavia, "Surface studies of in vitro biocompatibility of titanium oxide coatings", Applied Surface Science 172, 167–177 (2001).
- Choi, S., M. Goryll, L. Y. M. Sin, P. K. Wong and J. Chae, "Microfluidic-based biosensors toward point-of-care detection of nucleic acids and proteins", Microfluidics and Nanofluidics 2010 10:2 10, 231-247, URL https://link.springer.com/ article/10.1007/s10404-010-0638-8 (2010).
- Christie, B. P., K. R. Ashmont, P. A. House and B. Greger, "Approaches to a cortical vision prosthesis: implications of electrode size and placement", Journal of Neural Engineering 13, 025003, URL https://iopscience.iop.org/article/10.1088/1741-2560/13/2/025003https://iopscience.iop.org/article/10.1088/1741-2560/13/2/025003/meta (2016).

- Clifford, D. B., J. W. Olney, A. Maniotis, R. C. Collins and C. F. Zorumski, "The functional anatomy and pathology of lithium-pilocarpine and high-dose pilocarpine seizures", Neuroscience 23, 953–968 (1987).
- Cogan, S. F., D. J. Garrett and R. A. Green, "Electrochemical principles of safe charge injection", Neurobionics: The Biomedical Engineering of Neural Prostheses pp. 55-88, URL https://onlinelibrary.wiley.com/doi/full/ 10.1002/9781118816028.ch3https://onlinelibrary.wiley.com/doi/abs/ 10.1002/9781118816028.ch3https://onlinelibrary.wiley.com/doi/10.1002/ 9781118816028.ch3 (2016).
- Collinge, S., G. Prendergast, S. T. Mayers, D. Marshall, P. Siddell, E. Neilly, C. D. Ferrie, G. Vadlamani, J. Macmullen-Price, D. J. Warren, A. Zaman, P. Chumas, J. Goodden and M. C. Morrall, "Pre-surgical mapping of eloquent cortex for paediatric epilepsy surgery candidates: Evidence from a review of advanced functional neuroimaging", Seizure 52, 136–146 (2017).
- Curia, G., D. Longo, G. Biagini, R. S. Jones and M. Avoli, "The pilocarpine model of temporal lobe epilepsy", (2008).
- De La Oliva, N., M. Mueller, T. Stieglitz, X. Navarro and J. Del Valle, "On the use of parylene c polymer as substrate for peripheral nerve electrodes", Scientific Reports 2018 8:1 8, 1–12, URL https://www.nature.com/articles/s41598-018-24502-z (2018).
- Del Valle, J., N. De La Oliva, M. Müller, T. Stieglitz and X. Navarro, "Biocompatibility evaluation of parylene c and polyimide as substrates for peripheral nerve interfaces", International IEEE/EMBS Conference on Neural Engineering, NER 2015-July, 442–445 (2015).
- Dewhirst, M. W., B. L. Viglianti, M. Lora-Michiels, P. J. Hoopes and M. Hanson, "Thermal dose requirement for tissue effect: Experimental and clinical findings", Proceedings of SPIE-the International Society for Optical Engineering 4954, 37, URL /pmc/articles/PMC4188373//pmc/articles/PMC4188373/?report= abstracthttps://www.ncbi.nlm.nih.gov/pmc/articles/PMC4188373/ (2003).
- Dobrzynska, J. A. and M. A. Gijs, "Flexible polyimide-based force sensor", Sensors and Actuators, A: Physical 173, 127–135 (2012).
- Dubey, A. and S. Ray, "Cortical electrocorticogram (ecog) is a local signal", Journal of Neuroscience 39, 4299-4311, URL https://www.jneurosci.org/content/39/ 22/4299https://www.jneurosci.org/content/39/22/4299.abstract (2019).
- Elmqvist, R. and A. Senning, "An implantable pacemaker for the heart", Medical Electronics. Proceedings of the Second International Conference on Medical Electronics p. 24, URL https://scholar.google.com/scholar?hl= en&as\_sdt=0%2C3&q=elmqvist+senning+an+implantable+pacemaker+for+ the+heart&btnG=#d=gs\_cit&u=%2Fscholar%3Fq%3Dinfo%3Aa8VLU5CC7DcJ% 3Ascholar.google.com%2F%26output%3Dcite%26scirp%3D0%26hl%3Den (1959).

- Engel, J., J. Chen and C. Liu, "Development of polyimide flexible tactile sensor skin", Journal of Micromechanics and Microengineering 13, 359-366, URL https://iopscience.iop.org/article/10.1088/0960-1317/13/3/302https: //iopscience.iop.org/article/10.1088/0960-1317/13/3/302/meta (2003).
- Fonoff, E. T., J. F. Pereira, L. V. Camargo, C. S. Dale, R. L. Pagano, G. Ballester and M. J. Teixeira, "Functional mapping of the motor cortex of the rat using transdural electrical stimulation", Behavioural Brain Research 202, 138–141 (2009).
- Fountas, K. N., D. W. King, P. D. Jenkins and J. R. Smith, "Nonhabitual seizures in patients with implanted subdural electrodes", Stereotactic and Functional Neurosurgery 82, 165–168, URL https://www.karger.com/Article/FullText/81881 (2004).
- Freeman, W. J., L. J. Rogers, M. D. Holmes and D. L. Silbergeld, "Spatial spectral analysis of human electrocorticograms including the alpha and gamma bands", Journal of Neuroscience Methods 95, 111–121 (2000).
- Freeman, W. J. and J. Zhai, "Simulated power spectral density (psd) of background electrocorticogram (ecog)", Cognitive Neurodynamics 3, 97–103, URL https:// link.springer.com/article/10.1007/s11571-008-9064-y (2009).
- Galvani, L., "De viribus electricitatis in motu musculari commentarius", dvei URL https://ui.adsabs.harvard.edu/abs/1791dvei.book.....G/abstract (1791).
- Gamry, I., "Basics of eis: Electrochemical research-impedance", URL https: //www.gamry.com/application-notes/EIS/basics-of-electrochemicalimpedance-spectroscopy/ (2021).
- Garich, J., I. Akamine, S. Hara, D. Gulick, M. Benscoter, S. Kuehn, G. Worrell, G. Raupp and J. Blain Christen, "The m<sup>3</sup> electrode: flexible telescopic configuration enhances spatial resolution in subdural recording", Journal of Neural Engineering - In Preparation (2022).
- Geller, J. H., "The medical device amendments of 1976 major features and comparisons", Food, Drug, Cosmetic Law Journal **31**, URL https://heinonline.org/HOL/Page?handle=hein.journals/foodlj31&id= 450&div=93&collection=journals (1976).
- Gotman, J., "Measurement of small time differences between eeg channels: Method and application to epileptic seizure propagation", Electroencephalography and Clinical Neurophysiology 56, 501–514 (1983).
- Hader, W. J., J. Tellez-Zenteno, A. Metcalfe, L. Hernandez-Ronquillo, S. Wiebe, C. S. Kwon and N. Jette, "Complications of epilepsy surgery—a systematic review of focal surgical resections and invasive eeg monitoring", Epilepsia 54, 840-847, URL https://onlinelibrary.wiley.com/doi/full/ 10.1111/epi.12161https://onlinelibrary.wiley.com/doi/abs/10.1111/ epi.12161https://onlinelibrary.wiley.com/doi/10.1111/epi.12161 (2013).

- Hara, S. A., B. J. Kim, J. T. Kuo, C. D. Lee, E. Meng and V. Pikov, "Longterm stability of intracortical recordings using perforated and arrayed parylene sheath electrodes", Journal of Neural Engineering 13, 066020, URL https: //iopscience.iop.org/article/10.1088/1741-2560/13/6/066020https: //iopscience.iop.org/article/10.1088/1741-2560/13/6/066020/meta (2016).
- Hodgkin, A. L. and A. F. Huxley, "A quantitative description of membrane current and its application to conduction and excitation in nerve", The Journal of Physiology 117, 500, URL https://www.ncbi.nlm.nih.gov/pmc/articles/PMC1392413/ (1952).
- Holmes, G. L. and R. Khazipov, Basic neurophysiology and the cortical basis of EEG (Humana Press, 2007), URL https://link.springer.com/chapter/10.1007/978-1-59745-271-7\_2.
- House, W., "Cochlear implant: the beginning.", The Laryngoscope **97**, 996–997, URL https://europepmc.org/article/med/3302573 (1987).
- Hudak, E. M., J. T. Mortimer and H. B. Martin, "Platinum for neural stimulation: voltammetry considerations", Journal of Neural Engineering 7, 026005, URL https://iopscience.iop.org/article/10.1088/1741-2560/7/2/026005https: //iopscience.iop.org/article/10.1088/1741-2560/7/2/026005/meta (2010).
- Jun, J. J., N. A. Steinmetz, J. H. Siegle, D. J. Denman, M. Bauza, B. Barbarits, A. K. Lee, C. A. Anastassiou, A. Andrei, Çağatay Aydin, M. Barbic, T. J. Blanche, V. Bonin, J. Couto, B. Dutta, S. L. Gratiy, D. A. Gutnisky, M. Häusser, B. Karsh, P. Ledochowitsch, C. M. Lopez, C. Mitelut, S. Musa, M. Okun, M. Pachitariu, J. Putzeys, P. D. Rich, C. Rossant, W. L. Sun, K. Svoboda, M. Carandini, K. D. Harris, C. Koch, J. O'Keefe and T. D. Harris, "Fully integrated silicon probes for high-density recording of neural activity", Nature 551, 232–236, URL https: //www.nature.com/articles/nature24636 (2017).
- Kandel, E. R., J. H. Schwartz, T. M. Jessell, S. A. Siegelbaum and A. Hudspeth, Principles of Neural Science, Fifth Edition - Google Books (McGraw-Hill, 2000), 4th edn., URL https://books.google.com/ books/about/Principles\_of\_Neural\_Science\_Fifth\_Editi.html?id= s64z-LdAIsEChttps://books.google.co.in/books/about/ Principles\_of\_Neural\_Science\_Fifth\_Editi.html?id=s64z-LdAIsEC.
- Kanth, S.-T. and S. Ray, "Electrocorticogram (ecog) is highly informative in primate visual cortex", Journal of Neuroscience 40, 2430-2444, URL https://www.jneurosci.org/content/40/12/2430https://www.jneurosci.org/ content/40/12/2430.abstract (2020).
- Kayser, L. V. and D. J. Lipomi, "Stretchable conductive polymers and composites based on pedot and pedot:pss", Advanced Materials **31**, 1806133, URL https://onlinelibrary.wiley.com/doi/full/10.1002/adma.201806133https: //onlinelibrary.wiley.com/doi/abs/10.1002/adma.201806133https: //onlinelibrary.wiley.com/doi/10.1002/adma.201806133 (2019).

- Khodagholy, D., J. N. Gelinas, T. Thesen, W. Doyle, O. Devinsky, G. G. Malliaras and G. Buzsáki, "Neurogrid: Recording action potentials from the surface of the brain", Nature Neuroscience 18, 310–315, URL https://www.nature.com/ articles/nn.3905 (2015).
- Kucewicz, M. T., J. Cimbalnik, J. Y. Matsumoto, B. H. Brinkmann, M. R. Bower, V. Vasoli, V. Sulc, F. Meyer, W. R. Marsh, S. M. Stead and G. A. Worrell, "High frequency oscillations are associated with cognitive processing in human recognition memory", Brain 137, 2231–2244, URL https://academic.oup.com/brain/ article/137/8/2231/2848260 (2014).
- Lee, W. S., J. K. Lee, S. A. Lee, J. K. Kang and T. S. Ko, "Complications and results of subdural grid electrode implantation in epilepsy surgery", Surgical Neurology 54, 346–351 (2000).
- Lenihan, T., L. Schaper, Y. Shi, G. Morcan and J. Parkerson, "Embedded thin film resistors, capacitors and inductors in flexible polyimide films", Proceedings - Electronic Components and Technology Conference pp. 119–124 (1996).
- Lu, C. W., P. G. Patil and C. A. Chestek, Current Challenges to the Clinical Translation of Brain Machine Interface Technology, vol. 107 (Academic Press Inc., 2012).
- Lundstrom, B. N., J. V. Gompel, F. Khadjevand, G. Worrell and M. Stead, "Chronic subthreshold cortical stimulation and stimulation-related eeg biomarkers for focal epilepsy", Brain Communications 1, URL https://academic.oup.com/ braincomms/article/1/1/fcz010/5564187 (2019).
- Lundstrom, B. N., C. Meisel, J. V. Gompel, M. Stead and G. Worrell, "Comparing spiking and slow wave activity from invasive electroencephalography in patients with and without seizures", Clinical Neurophysiology 129, 909–919 (2018).
- Maynard, E. M., C. T. Nordhausen and R. A. Normann, "The utah intracortical electrode array: A recording structure for potential brain-computer interfaces", Electroencephalography and Clinical Neurophysiology **102**, 228–239 (1997).
- Middlebrooks, E. H., S. S. Grewal, M. Stead, B. N. Lundstrom, G. A. Worrell and J. J. V. Gompel, "Differences in functional connectivity profiles as a predictor of response to anterior thalamic nucleus deep brain stimulation for epilepsy: a hypothesis for the mechanism of action and a potential biomarker for outcomes", Neurosurgical Focus 45, E7, URL https://thejns.org/focus/view/journals/ neurosurg-focus/45/2/article-pE7.xml (2018).
- Minev, I. R., P. Musienko, A. Hirsch, Q. Barraud, N. Wenger, E. M. Moraud, J. Gandar, M. Capogrosso, T. Milekovic, L. Asboth, R. F. Torres, N. Vachicouras, Q. Liu, N. Pavlova, S. Duis, A. Larmagnac, J. Vörös, S. Micera, Z. Suo, G. Courtine and S. P. Lacour, "Electronic dura mater for long-term multimodal neural interfaces", Science 347, 159–163, URL https://www.science.org/doi/10.1126/science.1260318 (2015).

- Nair, D. R., K. D. Laxer, P. B. Weber, A. M. Murro, Y. D. Park, G. L. Barkley, B. J. Smith, R. P. Gwinn, M. J. Doherty, K. H. Noe, R. S. Zimmerman, G. K. Bergey, W. S. Anderson, C. Heck, C. Y. Liu, R. W. Lee, T. Sadler, R. B. Duckrow, L. J. Hirsch, R. E. Wharen, W. Tatum, S. Srinivasan, G. M. McKhann, M. A. Agostini, A. V. Alexopoulos, B. C. Jobst, D. W. Roberts, V. Salanova, T. C. Witt, S. S. Cash, A. J. Cole, G. A. Worrell, B. N. Lundstrom, J. C. Edwards, J. J. Halford, D. C. Spencer, L. Ernst, C. T. Skidmore, M. R. Sperling, I. Miller, E. B. Geller, M. J. Berg, A. J. Fessler, P. Rutecki, A. M. Goldman, E. M. Mizrahi, R. E. Gross, D. C. Shields, T. H. Schwartz, D. R. Labar, N. B. Fountain, W. J. Elias, P. W. Olejniczak, N. R. Villemarette-Pittman, S. Eisenschenk, S. N. Roper, J. G. Boggs, T. A. Courtney, F. T. Sun, C. G. Seale, K. L. Miller, T. L. Skarpaas and M. J. Morrell, "Nine-year prospective efficacy and safety of brain-responsive neurostimulation for focal epilepsy", Neurology 95, e1244–e1256, URL https://n.neurology.org/content/95/9/e1244.https://n.neurology.org/content/95/9/e1244.https://n.neurology.org/content/95/9/e1244.https://n.neurology.org/content/95/9/e1244.https://n.neurology.org/content/95/9/e1244.https://n.neurology.org/content/95/9/e1244.https://n.neurology.org/content/95/9/e1244.https://n.neurology.org/content/95/9/e1244.https://n.neurology.org/content/95/9/e1244.https://n.neurology.org/content/95/9/e1244.https://n.neurology.org/content/95/9/e1244.https://n.neurology.org/content/95/9/e1244.https://n.neurology.org/content/95/9/e1244.https://n.neurology.org/content/95/9/e1244.https://n.neurology.org/content/95/9/e1244.https://n.neurology.org/content/95/9/e1244.https://n.neurology.org/content/95/9/e1244.https://n.neurology.org/content/95/9/e1244.https://n.neurology.org/content/95/9/e1244.https://n.neurology.org/content/95/9/e1244.https://n.neurology.org/content/95/9/e1244.https://n.neurology.org/content/95/9/e1244.https://n.neurology.org/content/95/9/e1244.https://n.neurology.org/co
- Neafsey, E. J., E. L. Bold, G. Haas, K. M. Hurley-Gius, G. Quirk, C. F. Sievert and R. R. Terreberry, "The organization of the rat motor cortex: A microstimulation mapping study", Brain Research Reviews 11, 77–96 (1986).
- Newton, I., "Mathematical principles of natural philosophy", Great books of the western world; 55 pp. xviii, 552 (1729).
- Obsorne Shafer, P. and Epilepsy.com, "About epilepsy: The basics epilepsy foundation", URL https://www.epilepsy.com/learn/about-epilepsy-basics (2013).
- Ortigoza-Diaz, J., K. Scholten, C. Larson, A. Cobo, T. Hudson, J. Yoo, A. Baldwin, A. W. Hirschberg and E. Meng, "Techniques and considerations in the microfabrication of parylene c microelectromechanical systems", Micromachines 2018, Vol. 9, Page 422 9, 422, URL https://www.mdpi.com/2072-666X/9/9/422/htmhttps://www.mdpi.com/2072-666X/9/9/422 (2018).
- Oswalt, D., P. Datta, N. Talbot, Z. Mirzadeh and B. Greger, "A vision prosthesis based on electrical stimulation of the primary visual cortex using epicortical microelectrodes", bioRxiv p. 2020.12.15.422891, URL https://www.biorxiv.org/content/10.1101/2020.12.15.422891v1https: //www.biorxiv.org/content/10.1101/2020.12.15.422891v1.abstract (2020).
- Owda, A. and A. Casson, "Electrical properties, accuracy, and multi-day performance of gelatine phantoms for electrophysiology", bioRxiv p. 2020.05.30.125070, URL https://www.biorxiv.org/content/10.1101/2020.05.30.125070v1https://www.biorxiv.org/content/10.1101/2020.05.30.125070v1.abstract (2020).
- Penfield and Jasper, "Epilepsy and the functional anatomy of the human brain.", URL https://psycnet.apa.org/record/1955-01377-000?utm\_medium= email&utm\_source=transaction (1954).
- Richardson, R. R., J. A. Miller and W. M. Reichert, "Polyimides as biomaterials: preliminary biocompatibility testing", Biomaterials 14, 627–635 (1993).

- Rodger, D. C., A. J. Fong, W. Li, H. Ameri, A. K. Ahuja, C. Gutierrez, I. Lavrov, H. Zhong, P. R. Menon, E. Meng, J. W. Burdick, R. R. Roy, V. R. Edgerton, J. D. Weiland, M. S. Humayun and Y. C. Tai, "Flexible parylene-based multielectrode array technology for high-density neural stimulation and recording", Sensors and Actuators B: Chemical 132, 449–460 (2008).
- Romanelli, P., M. Piangerelli, D. Ratel, C. Gaude, T. Costecalde, C. Puttilli, M. Picciafuoco, A. Benabid and N. Torres, "A novel neural prosthesis providing long-term electrocorticography recording and cortical stimulation for epilepsy and brain-computer interface", Journal of Neurosurgery 130, 1166–1179, URL https://thejns.org/doi/abs/10.3171/2017.10.JNS17400 (2019).
- Schevon, C. A., S. K. Ng, J. Cappell, R. R. Goodman, G. McKhann, A. Waziri, A. Branner, A. Sosunov, C. E. Schroeder and R. G. Emerson, "Microphysiology of epileptiform activity in human neocortex", Journal of Clinical Neurophysiology 25, 321-330, URL /pmc/articles/PMC2967462//pmc/articles/ PMC2967462/?report=abstracthttps://www.ncbi.nlm.nih.gov/pmc/articles/ PMC2967462/ (2008).
- Sellers, K. K., W. L. Schuerman, H. E. Dawes, E. F. Chang and M. K. Leonard, "Comparison of common artifact rejection methods applied to direct cortical and peripheral stimulation in human ecog", International IEEE/EMBS Conference on Neural Engineering, NER 2019-March, 77–80 (2019).
- Shadmani, A., V. Viswam, Y. Chen, R. Bounik, J. Dragas, M. Radivojevic, S. Geissler, S. Sitnikov, J. Muller and A. Hierlemann, "Stimulation and artifactsuppression techniques for in-vitro high-density microelectrode array systems", IEEE Transactions on Biomedical Engineering (2018).
- Stead, M., M. Bower, B. H. Brinkmann, K. Lee, W. R. Marsh, F. B. Meyer, B. Litt, J. V. Gompel and G. A. Worrell, "Microseizures and the spatiotemporal scales of human partial epilepsy", Brain 133, 2789–2797, URL https://academic.oup.com/ brain/article-lookup/doi/10.1093/brain/awq190 (2010).
- Sun, Y., S. P. Lacour, R. A. Brooks, N. Rushton, J. Fawcett and R. E. Cameron, "Assessment of the biocompatibility of photosensitive polyimide for implantable medical device use", Journal of Biomedical Materials Research Part A 90A, 648– 655, URL http://doi.wiley.com/10.1002/jbm.a.32125 (2009).
- Theofanopoulos, P. C. and G. C. Trichopoulos, "Toward large-scale dynamically reconfigurable apertures using graphene", 2019 IEEE International Symposium on Antennas and Propagation and USNC-URSI Radio Science Meeting, APSURSI 2019 - Proceedings pp. 511–512 (2019).
- Turski, W. A., E. A. Cavalheiro, M. Schwarz, S. J. Czuczwar, Z. Kleinrok and L. Turski, "Limbic seizures produced by pilocarpine in rats: Behavioural, electroencephalographic and neuropathological study", Behavioural Brain Research 9, 315–335 (1983).

- Uthman, B. M., A. M. Reichl, J. C. Dean, S. Eisenschenk, R. Gilmore, S. Reid, S. N. Roper and B. J. Wilder, "Effectiveness of vagus nerve stimulation in epilepsy patients", Neurology 63, 1124–1126, URL https://n.neurology.org/content/ 63/6/1124https://n.neurology.org/content/63/6/1124.abstract (2004).
- Van Gompel, J. J., F. B. Meyer, W. R. Marsh, K. H. Lee and G. A. Worrell, "Stereotactic electroencephalography with temporal grid and mesial temporal depth electrode coverage: Does technique of depth electrode placement affect outcome?", Journal of Neurosurgery 113, 32–38, URL https://thejns.org/view/journals/ j-neurosurg/113/1/article-p32.xml (2010).
- Van Gompel, J. J., G. A. Worrell, M. L. Bell, T. A. Patrick, G. D. Cascino, C. Raffel, W. R. Marsh and F. B. Meyer, "Intracranial electroencephalography with subdural grid electrodes", Neurosurgery 63, 498–506, URL https://academic.oup.com/ neurosurgery/article/63/3/498/2558203 (2008).
- Viventi, J., D.-H. Kim, L. Vigeland, E. S. Frechette, J. A. Blanco, Y.-S. Kim, A. E. Avrin, V. R. Tiruvadi, S.-W. Hwang, A. C. Vanleer, D. F. Wulsin, K. Davis, C. E. Gelber, L. Palmer, J. V. D. Spiegel, J. Wu, J. Xiao, Y. Huang, D. Contreras, J. A. Rogers and B. Litt, "Flexible, foldable, actively multiplexed, high-density electrode array for mapping brain activity in vivo", Nature Neuroscience 14 (2011).
- Volta, A., "Xvii. on the electricity excited by the mere contact of conducting substances of different kinds. in a letter from mr. alexander volta, f. r. s. professor of natural philosophy in the university of pavia, to the rt. hon. sir joseph banks, bart. k.b. p. r. s", Philosophical Transactions of the Royal Society of London 90, 403–431, URL https://royalsocietypublishing.org/doi/abs/10.1098/rstl.1800.0018 (1800).
- Wang, S., I. Z. Wang, J. C. Bulacio, J. C. Mosher, J. Gonzalez-Martinez, A. V. Alexopoulos, I. M. Najm and N. K. So, "Ripple classification helps to localize the seizure-onset zone in neocortical epilepsy", Epilepsia 54, 370– 376, URL https://onlinelibrary.wiley.com/doi/full/10.1111/j.1528-1167.2012.03721.xhttps://onlinelibrary.wiley.com/doi/abs/10.1111/ j.1528-1167.2012.03721.xhttps://onlinelibrary.wiley.com/doi/10.1111/ j.1528-1167.2012.03721.xhttps://onlinelibrary.wiley.com/doi/10.1111/
- Wang, X., C. A. Gkogkidis, O. Iljina, L. D. Fiederer, C. Henle, I. Mader, J. Kaminsky, T. Stieglitz, M. Gierthmuehlen and T. Ball, "Mapping the fine structure of cortical activity with different micro-ecog electrode array geometries", Journal of Neural Engineering 14, 056004, URL https://doi.org/10.1088/1741-2552/aa785e (2017).
- Warren, C. P., S. Hu, M. Stead, B. H. Brinkmann, M. R. Bower and G. A. Worrell, "Synchrony in normal and focal epileptic brain: The seizure onset zone is functionally disconnected", Journal of Neurophysiology 104, 3530–3539, URL https://journals.physiology.org/doi/abs/10.1152/jn.00368.2010 (2010).
- Weltman, A., J. Yoo and E. Meng, "Flexible, penetrating brain probes enabled by advances in polymer microfabrication", Micromachines 2016, Vol. 7,

Page 180 7, 180, URL https://www.mdpi.com/2072-666X/7/10/180/htmhttps: //www.mdpi.com/2072-666X/7/10/180 (2016).

- Wetjen, N. M., W. R. Marsh, F. B. Meyer, G. D. Cascino, E. So, J. W. Britton, S. M. Stead and G. A. Worrell, "Intracranial electroencephalography seizure onset patterns and surgical outcomes in nonlesional extratemporal epilepsy: Clinical article", Journal of Neurosurgery 110, 1147–1152, URL https://thejns.org/view/ journals/j-neurosurg/110/6/article-p1147.xml (2009).
- Wise, K. D., D. J. Anderson, J. F. Hetke, D. R. Kipke and K. Najafi, "Wireless implantable microsystems: High-density electronic interfaces to the nervous system", Proceedings of the IEEE 92, 76–97 (2004).
- Woods, V., M. Trumpis, B. Bent, K. Palopoli-Trojani, C. H. Chiang, C. Wang, C. Yu, M. N. Insanally, R. C. Froemke and J. Viventi, "Long-term recording reliability of liquid crystal polymer µecog arrays", Journal of Neural Engineering 15, 066024, URL https://iopscience.iop.org/article/10.1088/1741-2552/aae39dhttps: //iopscience.iop.org/article/10.1088/1741-2552/aae39d/meta (2018).
- Worrell, G., K. Ludwig, J. Williams, J. Van Gompel, I. Kim and M. Benscoter, "Commercial-scale-production-of-thin-film-electrode-arrays-for-clinicalintracranial-eeg", URL https://cms.aesnet.org/abstractslisting/ commercial-scale-production-of-thin-film-electrode-arrays-forclinical-intracranial-eeg (2019).
- Worrell, G. A., "Sensing the body electric: biomarkers of epileptic brain.", Epilepsy currents **11**, 118-9, URL http://www.ncbi.nlm.nih.gov/pubmed/21852884http://www.pubmedcentral.nih.gov/articlerender.fcgi?artid=PMC3152153 (2011).
- Worrell, G. A., A. B. Gardner, S. M. Stead, S. Hu, S. Goerss, G. J. Cascino, F. B. Meyer, R. Marsh and B. Litt, "High-frequency oscillations in human temporal lobe: simultaneous microwire and clinical macroelectrode recordings", Brain 131, 928-937, URL https://academic.oup.com/brain/article-lookup/ doi/10.1093/brain/awn006 (2008).
- Worrell, G. A., K. Jerbi, K. Kobayashi, J. M. Lina, R. Zelmann and M. L. V. Quyen, "Recording and analysis techniques for high-frequency oscillations", (2012).
- Worrell, G. A., L. Parish, S. D. Cranstoun, R. Jonas, G. Baltuch and B. Litt, "High-frequency oscillations and seizure generation in neocortical epilepsy", Brain 127, 1496–1506, URL https://academic.oup.com/brain/article/127/7/1496/ 266204 (2004).
- Yeh, H.-L., J. V. Garich, I. R. Akamine, J. M. Blain Christen and S. A. Hara, "Laser micromachining of thin-film polyimide microelectrode arrays: Alternative processes to photolithography", Frontiers in Biomedical Devices, BIOMED - 2020 Design of Medical Devices Conference, DMD 2020 URL http://asmedigitalcollection.asme.org/BIOMED/proceedings-pdf/DMD2020/ 83549/V001T04A002/6552660/v001t04a002-dmd2020-9057.pdf (2020).

- Young, D., F. Willett, W. D. Memberg, B. Murphy, B. Walter, J. Sweet, J. Miller, L. R. Hochberg, R. F. Kirsch and A. B. Ajiboye, "Signal processing methods for reducing artifacts in microelectrode brain recordings caused by functional electrical stimulation", Journal of Neural Engineering 15, 026014, URL https://iopscience.iop.org/article/10.1088/1741-2552/aa9ee8https: //iopscience.iop.org/article/10.1088/1741-2552/aa9ee8/meta (2018).
- Zhang, Y., W. van Drongelen, M. Kohrman and B. He, "Three-dimensional brain current source reconstruction from intra-cranial ecog recordings", NeuroImage 42, 683–695 (2008).
- Zhou, A., B. C. Johnson and R. Muller, "Toward true closed-loop neuromodulation: artifact-free recording during stimulation", Current Opinion in Neurobiology 50, 119–127 (2018).
- Onal, C., H. Otsubo, T. Araki, S. Chitoku, A. Ochi, S. Weiss, W. Logan, I. Elliott, O. Snead and J. Rutka, "Complications of invasive subdural grid monitoring in children with epilepsy", Journal of Neurosurgery 98, 1017–1026, URL https:// thejns.org/view/journals/j-neurosurg/98/5/article-p1017.xml (2003).

### APPENDIX A FABRIATION PROCEDURES

# **Fabrication Procedures**

Laboratory Manual for the fabrication of intracranial electroencephalography arrays for Brain Mapping & Epilepsy research





#### Table of Contents

1	Wafer Preparations	6	
	1.1 Equipment Needed		
	1.2 Materials Needed	6	
	1.3 Preparations Process	6	
	1.3.1 Wafer Cleaning	6	
	1.3.2 Adhesion Promoter Deposition	8	
2	Substrate Deposition	12	
	2.1 Equipment Needed	12	
	2.2 Materials Needed	12	
	2.3 Deposition Process	12	
	2.3.1 Polyimide Spinning	12	
	2.3.2 Inspection	15	
3	Photoresist Deposition for Metal Patterning	18	
	3.1 Equipment Needed	18	
	3.2 Materials Needed	18	
	3.3 Deposition Process	18	
	3.3.1 Photoresist Spinning	18	
	3.3.2 Photoresist Exposure	24	
	3.3.3 Photoresist Developing	28	
	3.3.4 Inspection	31	
4	Substrate Surface Preparations	33	
	4.1 Equipment Needed	33	
	4.2 Materials Needed	33	
	4.3 Preparations Process	33	
	4.3.1 Inspection	33	
	4.3.2 Surface Roughening	35	
5	Conductive Metal Deposition	38	
	5.1 Equipment Needed		
	5.2 Materials Needed		
	5.3 Deposition Process	38	
	5.3.1 E-Beam Evaporation	38	

	5.3.2	Inspection	46
6	Lift-Off for	Conductive Metal Patterning	48
	6.1 Equi	pment Needed	48
	6.2 Mate	erials Needed	48
	6.3 Lift-C	Off Process	48
	6.3.1	Photoresist Stripper & Cleaning Baths	48
	6.3.2	Inspection	58
7	Conductive	e Metal Testing	60
	7.1 Equi	pment Needed	60
	7.2 Mate	erials Needed	60
	7.3 Testi	ing Process	60
	7.3.1	Four-Point Probe Testing (Stationary)	60
	7.3.2	Probe Station Testing (Micromanipulators)	62
8	Pre-Insulat	tion Surface Preparation	67
	8.1 Equi	pment Needed	67
	8.2 Mate	erials Needed	67
	8.3 Prep	parations Process	67
	8.3.1	Inspection	67
	8.3.2	Surface Roughening	69
9	Insulation Deposition		72
	9.1 Equi	pment Needed	72
	9.2 Mate	erials Needed	72
	9.3 Depo	osition Process	72
	Preface	9	72
	9.3.1	Coater Loading	73
	9.3.2	Coater Unloading	76
	9.3.3	Inspection	78
10	Photoresis	st Deposition for Electrode Contacts	81
	10.1 Equip	oment Needed	81
	10.2 Mater	81	
	10.3 Depo	81	
	10.3.1	Photoresist Spinning	81
	10.3.2	Photoresist Exposure	87
	10.3.3	Photoresist Developing	91

	10.3.4	Inspection	94
11	Insulation	Etching for Electrode Contacts	97
	11.1 Equip	oment Needed	97
	11.2 Mater	rials Needed	97
	11.3 Etchir	ng Process	97
	11.3.1	Reactive Ion Etching Machine	97
	11.3.2	Inspection	97
12	Photoresis	st Removal after Electrode Contacts Etching	98
	12.1 Equip	oment Needed	98
	12.2 Mater	rials Needed	98
	12.3 Remo	oval Process	98
	12.3.1	Photoresist Stripper & Cleaning Baths	98
	12.3.2	Inspection	98
13	Electrode	Contact Testing	99
	13.1 Equip	oment Needed	99
	13.2 Mater	rials Needed	99
	13.3 Testir	ng Process	99
	13.3.1	4-Point Probe Testing (Stationary)	99
	13.3.2	Probe Station Testing (Micromanipulator)	99
	13.3.3	Inspection	99
14	Photoresis	st Deposition for Device Removal	100
	14.1 Equip	oment Needed	100
	14.2 Mater	rials Needed	100
	14.3 Depo	sition Process	100
	14.3.1	Photoresist Spinning	100
	14.3.2	Photoresist Exposure	106
	14.3.3	Photoresist Developing	110
	14.3.4	Inspection	113
15	Substrate	Etching for Device Removal	116
	15.1 Equip	oment Needed	116
	15.2 Materials Needed		116
	15.3 Etchii	ng Process	116
	15.3.1	Reactive Ion Etching Machine	116
	15.3.2	Inspection	116

16	Photoresist Removal after Device Removal Etching		117
	16.1 Equip	117	
	16.2 Mater	117	
	16.3 Remo	117	
	16.3.1	Photoresist Stripper & Cleaning Baths	117
	16.3.2	Inspection	117
17	Device Removal		118
	17.1 Equipment Needed		118
	17.2 Mater	118	
	17.3 Remo	oval Process	118

# **1** Wafer Preparations

# 1.1 Equipment Needed

- Spinner
- Pyrex dish (>4" diameter) (Qty: 4)
- Block/Stand (for elevation and angulation of the Pyrex dish)
- Fume Hood (for Spinner & Pyrex Dishes)
- Small (~5-15 mL) container for Adhesion Promotion mixture (we use a 15 mL centrifuge tube)
- Vortexer or Stirrer
- Pipettes for small volumes (we use a 1-10 µL Pipetman and a 3 mL pipette)
- Refrigerator for storage of VM-651 Adhesion Promoter
- Timer

# 1.2 Materials Needed

- Wafer Tweezers or Wand (4")
- Wafer (3" or 4") Bare Silicon
- Cleanroom Notebook
- VM-651 Polyimide Adhesion Promoter (~1-2 µL per batch)
- DI Water for adhesion promotion (~1-2 mL per batch)
- Acetone (~10 mL per wafer)
- Isopropyl Alcohol (~10 mL per wafer)
- DI Water for cleaning (~30 mL per wafer)
- Nitrogen air gun for drying
- Waste Containers for each Material (~4)
- Cleanroom Wipes
- Marker for wafer labeling
- White Tape for Adhesion Promoter container labeling

## **1.3 Preparations Process**

#### 1.3.1 Wafer Cleaning

- 1. Grab 3 Pyrex dishes (>4" diameter) and set them on one Cleanroom Wipe each.
- Fill the "start" of the dishes line with Acetone (~100-200 mL or until the wafer will be completely submerged) and label the Cleanroom Wipe underneath with "1 - Acetone", the date, and your initials.
- 3. Fill the "middle" of the dishes line with Isopropyl Alcohol (~100-200 mL or until the wafer will be completely submerged) and label the Cleanroom Wipe underneath with "2 IPA", the date, and your initials.
- Fill the "end" of the dishes line with DI Water (~100-200 mL or until the wafer will be completely submerged) and label the Cleanroom Wipe underneath with "3 - DI Water", the date, and your initials.
- 5. Ensure the Nitrogen air gun is nearby and handy.
- Add a Cleanroom Wipe after the DI Water bath and label the Cleanroom Wipe with "4 N2 Dry", the date, and your initials. The Nitrogen air gun should be easily accessible to this empty Cleanroom Wipe.
- 7. Grab the Wafer Tweezers or Wand and place it next to the "start" dish with Acetone inside.
  - a. Note: It is a lot cleaner processing to have one Wafer Tweezer or Wand per chemical bath in order to prevent as much cross contamination as possible. If this is not available, one pair of tweezers is acceptable, but will require cleaning separately before and after the processing.
- 8. Grab the Wafer Box containing bare Silicon wafers that are ready for processing and place it next to the "start" dish with Acetone inside. If there is tape on the box of wafers, this would be the time to remove the tape.
  - a. If available, grab an empty Wafer Box that the "Clean" wafers can be placed into once dried. If not available, ensure that the "Clean" wafers are separated and designated from the "Dirty" wafers inside the single wafer box.
- 9. The following steps (Steps 10-18) are for one single wafer. Please repeat these steps for each wafer that is going to be cleaned.
- 10. Using the Wafer Tweezers or Wand, grab the next wafer in the Wafer Box that is ready to be cleaned.
- 11. Place the wafer into the Acetone bath and start the Timer for 30 seconds.
  - a. Agitation is recommended, so use the tweezers to slowly lift and lower the wafer such that the wafer does not surface from the Acetone submersion, but the solution should be moving during the agitation.
- 12. Remove the wafer from the Acetone bath once the Timer expires and place the wafer into the IPA bath and start the Timer for 30 seconds.
  - a. Agitation is recommended, so use the tweezers to slowly lift and lower the wafer such that the wafer does not surface from the IPA submersion, but the solution should be moving during the agitation.

- 13. Remove the wafer from the IPA bath once the Time expires and place the wafer into the DI Water bath and start the Timer for 30 seconds.
  - a. Agitation is recommended, so use the tweezers to slowly lift and lower the wafer such that the wafer does not surface from the DI Water submersion, but the solution should be moving during agitation.
- 14. Remove the wafer from the DI Water bath once the Timer expires and place the wafer onto the "N2 Dry" Cleanroom Wipe.
- 15. Using the Nitrogen air gun, dry the wafer from the center. Rotate the wrist of the hand holding the Nitrogen air gun to push the liquid solutions off the edges of the wafer. Be careful not to do this too aggressively as this could result in the wafer getting lifted by the force of the Nitrogen air gun.
- 16. Ensure all moisture has been taken off the front of the wafer and lift the wafer using the wafer tweezers or wand. Dry the back of the wafer while the wafer is in the tweezers or wand.
  - a. Note: If needed, you can place one edge of the wafer on the Cleanroom Wipe and hold it at a ~45°-90° angle (with the back facing up) such that the wafer's backside can be cleaned easier.
- 17. Turn the wafer back to the front side and dry the edges of the wafer by using a "back-and-forth" motion going from in front of the wafer to behind the wafer to in front of the wafer, and continuing the trend. The wrist of the hand holding the tweezers can be rotated so that the entire wafer's edges can be cleaned.
  - a. Note: I can demonstrate this in picture format if that is necessary for understanding.
- 18. Place the "Clean" wafer into the Wafer Box.
  - a. Note: This is either the "Clean" side of the single Wafer Box being used or it is the "Clean" Wafer Box that used to be empty.
- 19. Repeat Steps 10-18 until all wafers that will be processed have been cleaned completely. (Reiterating Step 9).
- 20. Once the batch is completed, empty the respective baths into their respective Waste Containers. The top(s) of the Wafer Box(es) being used can then be placed onto the respective bottom of the Wafer Box(es) being used.
  - a. Note: If multiple batches will be completed in a single day, cover the baths between batches so the baths will not evaporate. Recommended that the baths be replaced every 24 hours at most. If the bath starts to become dirty (likely after a few batches), a fresh bath may be needed for complete processing.
- 21. Clean the respective bath dishes with ~100 mL of DI Water, swirl around, and empty into the respective Waste Container.
  - a. Note: First rinse should always be in the waste container that was just used to empty the contents of the bath into. Second & Third rinses can be disposed down the drain.
- 22. Repeat Step 21 twice more (3x total), but this can be emptied down the drain instead of into the respective Waste Container.
- 23. Using the Nitrogen air gun and a Cleanroom Wipe, dry off the Pyrex dishes that were used for cleaning.
- 24. Set these dishes to the side so that they can air dry before being used again.

#### **1.3.2** Adhesion Promoter Deposition

- 1. Polyimide does not necessarily require an adhesion promoter, so the following steps are only necessary if you would like the edges of the polyimide to be "glued" to the wafer carrier. In other words, this section can be skipped if doing simple testing.
- 2. If you decide to continue and use an adhesion promoter to secure the outside of the wafer, start by removing the VM-651 Adhesion Promoter from the refrigerator.
  - a. Allow at least 20 minutes for the bottle condensation to slow or stop before removing the tape from the cap and using the contents within.
- 3. While you are waiting for the VM-651 Adhesion Promoter to return to room temperature, prepare the DI Water part of the mixture. We use a 15 mL centrifuge tube for the mixture, so I will refer to the small container for the Adhesion Promoter mixture as "the centrifuge tube".
  - a. Note: This container is just an example of what can be used. If this is not available, the mixture can be prepared directly in the Pyrex dish (>4" diameter) that will be used later in the process, but this makes the mixing/stirring a bit more difficult.
- 4. Partially fill a small (3 mL) pipette with DI Water up to the 1 mL mark. Empty this pipette into the centrifuge tube and close the cap to prevent dust, dirt, and debris from entering the mixture.
- 5. Remove the tape from the cap of the VM-651 Adhesion Promoter bottle and remove the cap.
- Using a small volume pipette (we use a 1-10 μL Pipetman), withdraw 1 μL of the VM-651 solution from the bottle.
- 7. Open the cap of the centrifuge tube, deposit the small volume of VM-651 Adhesion Promoter, and close the cap of the centrifuge tube.
- The centrifuge tube should now contain 1 mL of DI Water and 1 μL of VM-651. Place the cap back onto the VM-651 bottle, replace the tape over the cap, and return the bottle to the refrigerator.
- 9. Stir the mixture using either a Vortexer, a stirrer, or manual stirring. Recommended stirring time is at least 60 seconds, or longer if the mixture is still separated.
- 10. Grab a piece of white tape and write the Date, Time, & Initials of the preparer. The VM-651 and DI Water mixture is only valid for 24 hours, so noting this is important.
- 11. Place the labeled white tape on the Adhesion Promoter mixture container. Next steps will be inside the cleanroom.
- 12. Move the Adhesion Promoter container to the location of the Spinner and prepare the Pyrex dish (>4" diameter) for the solution.
- 13. Place a Cleanroom Wipe underneath the Pyrex dish (>4" diameter) that will be used for the Adhesion Promoter mixture and label it with "Adhesion Promoter", the date, and your initials.
- 14. Pour the Adhesion Promoter mixture into the Pyrex dish (>4" diameter)
- 15. Set the block/stand underneath one side of the Pyrex dish. This should lift the Pyrex dish to at least a 45° angle and move the Adhesion Promoter mixture to one corner/side of the dish.
  - a. Note: We use a smaller Pyrex dish for the block/stand. Almost anything can be used if needed.
- 16. Grab 2 Wafer Tweezers or Wands to be used for processing. I will use #1 and #2 to designate which tweezer to use.
- 17. Set a Cleanroom Wipe next to the Pyrex dish containing the Adhesion Promoter and label it with "Ready for Spinning", the date, and your initials.
- Set up the spinner machine so that it is ready to use for processing when each wafer is done with the Adhesion Promoter dips. Recommended spinning speeds are from 2,000-4,000 rpm. I typically use 3,000 rpm.
- a. Our Laurell Spinners require the system to be turned on, the N2 air valve to be opened, and the vacuum valve to be opened. There is also a wafer placement wand that holds the wafer in the center of the vacuum chuck, based on the diameter of the wafer.
- b. ASU NanoFab's Brewer Science CEE Coaters require the application to be started, account log in, "Spin Process" to be selected, "Spin Recipe" to be loaded, and the vacuum chuck to be installed.
- c. Ensure the correct size vacuum chuck is available and attached to the spinner machine.
- d. The spin speed will then have to be adjusted. I use a process recipe that uses a 500 rpm for 5 seconds "spread" step (ramp rate of 500 rpm/s), followed by a 3,000 rpm for 30 seconds "spin/dry" step (ramp rate of 1,000 rpm/s), followed by a 0 rpm for 5 seconds "ramp down" step (ramp rate of 1,000 rpm/s).
- 19. The following steps (Steps 20-41) are for one single wafer. Please repeat these steps for each wafer that is going to have Adhesion Promoter added to it.
  - a. Note: The following steps may be a bit confusing on the first read. I am working on adding some picture steps such that this makes it easier to understand.
- 20. Using the Wafer Tweezers or Wand (#1), grab the next Silicon wafer to be processed (bare and cleaned).
- 21. Rotate your wrist to the left such that it is at a  $\sim$ 45° angle to the right of your arm.
- 22. Dip the wafer into the Adhesion Promoter mixture without touching the bottom of the dish (if possible) and start the Timer for 20 seconds.
- 23. Once the Timer expires, remove the wafer from the solution and rotate your wrist back to center such that it is at a  $\sim 0^{\circ}$  angle from your arm.
- 24. Dip the wafer into the Adhesion Promoter mixture without touching the bottom of the dish (if possible) and start the Timer for 20 seconds.
- 25. Once the Timer expires, remove the wafer from the solution and rotate your wrist to the right using your wrist such that it is at a  $\sim$ 45° to the left of your arm.
- 26. Dip the wafer into the Adhesion Promoter mixture without touching the bottom of the dish (if possible) and start the Timer for 20 seconds.
- 27. Once the Timer expires, remove the wafer from the solution and grab the Left edge of the wafer (~90° to the left of the current #1 tweezers location) with the other Wafer Tweezers or Wand (#2).
- 28. Repeat Steps 21-27 using Wafer Tweezers or Wand #2 (left side, middle, and right side) and then grab the Left edge of the wafer (~90° to the left of the current #2 tweezers location) with the first Wafer Tweezers or Wand (#1).
- 29. Repeat Steps 21-27 using Wafer Tweezers or Wand #1 (left side, middle, and right side) and then grab the Left edge of the wafer (~90° to the left of the current #1 tweezers location) with the other Wafer Tweezers or Wand (#2).
- 30. Repeat Steps 21-27 using Wafer Tweezers or Wand #2 (left side, middle, and right side) and then place the wafer onto the Cleanroom Wipe with the "Ready for Spinning" label.
- 31. Grab the wafer that is now "Ready for Spinning" using Wafer Tweezers or Wand #2 and place it on the Wafer Placement Wand.
  - a. Note: This is just what is required of the Laurell Spinners. The ASU NanoFab's spinners have a "centering" tool to aid in this process.
- 32. Press the "Vacuum" button on the Spinner's interface to initiate the vacuum pull before the wafer is placed onto the chuck.

- a. Note: This would be the "Hold" button on the ASU NanoFab spinners, but requires the wafer to be on the vacuum chuck prior to being pressed.
- 33. Open the lid of the Spinner.
- 34. Place the wafer on the vacuum chuck from the Wafer Placement Wand and pull the Wand away once the wafer is under vacuum on the chuck.
  - a. Note: The ASU NanoFab's spinners require a Centering step be completed before starting the process. Do this now so that the process is consistent every time. (And to ensure the wafer is centered on the chuck)
- 35. Close the lid of the Spinner.
- 36. Press "Play/Start" on the Spinner's interface to start the process.
- 37. When the Spinner's interface reads "DONE", Press the "Vacuum" button on the Spinner's interface to release vacuum from the chuck.
  - a. Note: It is not necessary to release the vacuum from the chuck for the ASU NanoFab's spinners, it is done automatically.
- 38. Open the lid of the Spinner.
- 39. Using the Wafer Tweezers or Wand #2, grab the wafer from the vacuum chuck.
- 40. Close the lid of the Spinner.
- 41. Place the wafer into the Wafer Box.
  - a. Note: This Wafer Box can either be the same Wafer Box you have been using with designations of what has been done to each wafer OR a new empty Wafer Box can be used here as well to separate the wafers from the processing they have already seen.
  - b. Other Note: This is where a marker comes in handy, so that the wafers can be marked on the back with what processing they have seen (C for "Cleaned"; AP for "Adhesion Promoter"; PI for "Polyimide Deposited"; etc., etc.)
- 42. Repeat Steps 20-41 for each wafer needing Adhesion Promoter processing.
- 43. Once the batch is complete, empty the Adhesion Promoter mixture from the Pyrex dish (>4" diameter) to the respective Waste Container.
- 44. Rinse the now empty Pyrex dish with ~100 mL of DI Water and empty the contents into the respective Waste Container.
- 45. Repeat Step 44 twice, but the contents can be emptied down the drain instead of into the Waste Container.
- 46. Using the Nitrogen air gun and a Cleanroom Wipe, dry off the Pyrex dish.
- 47. Place the Pyrex dish in a location where it can be air dried before it is used again.
- Place the lid back onto the Wafer Box(es) with wafers that are now ready for the next step of processing.

# 2 Substrate Deposition

# 2.1 Equipment Needed

- Spinner
- Hot Plate or Oven (90°C & 150°C)
- Hot Plate or Oven (350°C @ 4°C/minute)
- Small Pyrex bottle, ~100 mL (for Polyimide pouring)
- Fume Hood
- Microscope (for inspection)
- Camera (for microscopic imaging)
- Timer
- Thickness Measurement tool (Filmetrics F20 Reflectometer or 3D Profilometer)
- Freezer for PI-2611 Polyimide Storage

## 2.2 Materials Needed

- Wafer Tweezers or Wand (4")
- Wafer (3" or 4") Silicon (w/ or w/o adhesion promotion)
- Cleanroom Notebook
- PI-2611 Polyimide Solution (~2 mL per wafer)
- Cleanroom Wipes
- Marker for wafer labeling

## 2.3 Deposition Process

#### 2.3.1 Polyimide Spinning

- 1. The PI-2611 Polyimide Solution should always be kept in the freezer until it needs to be used, so having a smaller bottle of Polyimide Solution (~100-200 mL) will help the longevity of the Polyimide so that most of the bottle is left in the freezer until the smaller bottle needs to be refilled.
- 2. Remove the smaller bottle of PI-2611 Polyimide Solution from the freezer. Leave this out in a Room Temperature environment for at least 60 minutes to allow it to thaw out.
  - a. Note: If the smaller bottle of PI-2611 Polyimide Solution is almost out, you will need to remove the larger bottle of PI-2611 Polyimide Solution from the freezer to allow it to thaw out as well so that it can then be used to fill the smaller bottle.
- 3. Once the PI-2611 Polyimide Solution is thawed, processing can then begin.
- 4. Set up the Spinner machine to ensure it is ready for processing.
  - a. Our Laurell Spinners require that the system be turned on, the N2 air valve opened, and the vacuum valve opened. The recipe then needs to be selected. I used Recipe 5, which has 3 steps.
  - b. ASU NanoFab's Brewer Science CEE Coaters require that the application be started, login information entered, "Spin Process" selected, recipe loaded, and correct size vacuum chuck installed.
  - c. Step 1 ("Spread") is a 5 second step at 500 rpm with a ramp rate of 500 rpm/s. Step 2 ("Spin") is a 30 second step at the specified spin speed (5,000 rpm for ~5 microns of polyimide and 2,500 rpm for ~10 microns of polyimide) with a ramp rate of 1,000 rpm/s. Step 3 ("Ramp Down") is a 5 second step at 0 rpm with a ramp rate of 1,000 rpm/s.
- 5. Set one of the Hot Plate/Oven to 90°C and set the other Hot Plate/Oven to 29°C.
  - a. Note: My method involves using one Hot Plate for the soft bake process (both temperatures) and another Hot Plate for the curing process. If there is only one Hot Plate and one Oven, the Hot Plate should be used for soft baking and the Oven should be used for curing.
- 6. Place the smaller bottle of PI-2611 Polyimide Solution next to the Spinner to have it ready for processing.

- 7. Grab a Cleanroom Wipe and place it next to the bottle of PI-2611 Polyimide Solution. I recommend folding it in half so that it is easier to use. This will be used to clean off the Wafer Tweezers or Wand after moving the wafers from the Spinner to the Hot Plate/Oven.
- 8. Place the Wafer Box containing the wafers that will be processed next to the Spinner. Open the box so that it can be ready for the following steps.
- 9. The following Steps (10-33) are for a single wafer. Please repeat those steps for each wafer that needs to be processed.
- 10. Using the Wafer Tweezers or Wand, grab the next wafer to be processed.
  - a. Note: I grab from the major flat of the wafers.
- 11. Place the wafer onto the loading or centering tool to put it into the Spinner.
  - a. Our Laurell Spinners have a Wafer Loading Wand that can be used for this.
  - b. The ASU NanoFab Brewer Science CEE Coaters have a centering hand tool to use for this.
- 12. Open the lid to the Spinner.
- 13. Press the "Vacuum" button on the interface of the Spinner to engage the vacuum suction on the chuck.
  - a. Note: This is the "Hold" button on the ASU NanoFab's spinners, but requires that the wafer be placed on the vacuum chuck before you press the button
- 14. Load the wafer into the center of the vacuum chuck.
  - a. Note: The ASU NanoFab's spinners require a Centering step be completed before starting the process. Do this now so that the process is consistent every time. (And to ensure the wafer is centered on the chuck)
- 15. Close the lid to the Spinner.
  - a. Note: This is to minimize dirt and debris that can get on the wafer prior to polyimide deposition. This is especially needed if there is polyimide in the spinner chamber that could potentially drip onto the wafer.
- 16. Open the lid to the PI-2611 Polyimide Solution bottle and set it to the side.
- 17. With the Pi-2611 Polyimide Solution in one hand, open the lid to the Spinner with the other hand.
- 18. Pour the PI-2611 Polyimide Solution onto the center of the wafer. Only about a quarter size glob
  - is needed to spin across the entire wafer, so this typically only takes one "drop" from the bottle.
    a. Note: I would consider this more of a "gloop" considering the texture and consistency of the liquid
- 19. Close the lid to the Spinner as soon as the glob is on the wafer and the bottle is clear from the Spinner.
  - a. Note: Due to the thick nature of the PI-2611 Polyimide Solution, this means that there will be material that can potentially drip down from the sides and lid of the Spinner. The opening and closing of the lid to the Spinner must be done in a quick (but cautious!) manner to avoid drops landing on a new wafer or on the hand of the operator.
- 20. Start a Timer for 30 seconds to allow for potential bubbles from the PI-2611 Polyimide Solution to come to the surface and disappear.
- 21. Once the Timer expires, press the "Start/Play" button on the interface of the Spinner.
- 22. Once the recipe is complete, the Spinner interface will read "DONE".
- 23. Press the "Vacuum" button on the interface of the Spinner to disengage the vacuum suction on the chuck.
  - a. Note: This is not necessary for the ASU NanoFab's spinners, it is done automatically.

- 24. Open the lid to the Spinner.
- 25. Grab the wafer with the Wafer Tweezers or Wand.
  - a. Note: I will try to grab from the major flat, but if it is too far towards the lid's hinge, I will just use as little edge of the tweezers as possible on a different edge of the wafer.
- 26. Close the lid to the Spinner once the wafer is out.
- 27. Place the wafer onto the Hot Plate that is currently set to 90°C and start a Timer for 120 seconds (2 minutes).
- 28. Once the Timer reaches 15 seconds remaining (105 seconds into the time), set the new temperature, on the Hot Plate the wafer is currently on, to 150°C.
  - a. Note: The Hot Plates take a few seconds to register that a temperature change has been made, so this is why I will set this change before the time expires.
- 29. Once the Timer expires, stop the Timer. It does not need to be restarted until the temperature reaches 150°C.
- 30. Once the temperature of the Hot Plate reaches 150°C, start the Timer for 120 seconds (2 minutes). See Note above for clarification on this temperature choice.
- 31. Once the Timer reaches 15 seconds remaining (105 seconds into the time), set the new temperature, on the Hot Plate the wafer is currently on, to 90°C.
  - a. Note: This step is to get ready for the next wafer. If no more wafers need to be processed, this Hot Plate can be turned off AFTER the wafer has been removed from the Hot Plate.
- 32. Once the Timer expires, remove the wafer from the Hot Plate.
- 33. Depending on the use of an Oven or a Hot Plate for curing, the next steps will differ.
  - a. For a Hot Plate, this can be set to 29°C so that it is ready for the curing process and the wafers can be placed directly onto the Hot Plate to wait for the rest of the batch.
  - b. For an Oven, the entire batch will likely have to go in all at once, so the wafer will have to sit and wait on a "Ready" Cleanroom Wipe or in a "Ready" Wafer Boat.
- 34. Repeat the above Steps (Steps 10-33) for each wafer in the batch that needs to be processed.
  - a. Note: Due to the size of the Hot Plate we use for the curing process, we are limited to a maximum of 4 wafers per curing batch (4" or 3"). We can cram 6 of the 3" wafers onto the Hot Plate, but it is very tight. An Oven would allow for larger batches.
- 35. Program #7 (Pr 07) is the curing program we use on the Torrey Pines Scientific hot plate in the ERC 444 cleanroom. The curing process is fairly simple, but requires a programmable function to the Hot Plate or Oven for the correct temperature, ramp rate, and duration to be set. I use the following program because it is consistent and it is pretty easy to program.
  - a. Step 1 "Set Base Temp" Temperature: 30°C Ramp Rate: 425°C/hour Duration: 30 seconds (just to make sure it is at temperature)
  - b. Step 2 "Ramp Up & Hold" Temperature: 350°C Ramp Rate: 240 °C/hour (4 °C/minute) – Duration: 30 minutes (to hold the temperature at 350 °C for the specified duration from the datasheet)
  - c. Step 3 "Ramp Down" Temperature: 30 °C Ramp Rate: 240-320 °C/hour (4 °C/minute+) Duration: 10 minutes (just to hold it at starting temperature for a bit to give some time for me to get back to the system and remove the wafers)
    - i. Note: This cooldown process does not need to be as consistent as the ramp up process, so the rate at which you cooldown is a matter of time preference and

the quality of the hot plate/oven you are using. I have used a rate of 320 °C/hour and a rate of 425 °C/hour without any faults in the Polyimide substrate layer

- d. \*Optional Step\* Step 4 "Shut Down" Temperature: 17 °C Ramp Rate: 425 °C/hour Duration: 30 minutes (useful for a Friday afternoon run so that the system will be left at the temperature it would shut down at over the weekend)
- 36. Once the curing process is done, remove the completed batch of wafers and place them into the corresponding Wafer Box.

#### 2.3.2 Inspection

- 1. There are three methods of inspection we use to measure the quality of polyimide we get after spinning, baking, and curing.
  - a. The first is a visual inspection using our eyes.
  - b. The second is a visual inspection using a microscope.
  - c. The third is a metric inspection using a measurement tool to get the thickness of the layer.
- 2. As a first inspection, just looking at the wafer will show us some large defects if they are present. This would include bubbles, tears, voids, and colored swirls. The bubbles typically indicate dirt and debris beneath the layer or an adhesion problem while tears and voids indicate that the layer wasn't complete. Bubbles can continue into further processing (unless they are excessively large) while voids and tears means the entire wafer will have to be reworked to prevent additional defects (unless the tear or void is outside of the region that will be used for metallization). Colored swirls on the other hand, are natural of the polyimide material based on the frequency and wavelength of white and filtered light. However, if these swirls are too prevalent and too close together, then the measurement techniques will have a very hard time differentiating between the thicknesses in those areas. This is a very objective determination, but also why the process has been set in the manner that it is listed above, to remove as many inconsistencies as possible.
- 3. The second inspection involves using a microscope to inspect the open areas with no defects to ensure the entire material is as uniform as possible. This also gives us the chance to investigate the defects we found from the first inspection and see if they are actual defects or just blemishes.
- 4. The third inspection is where we measure the thickness of the polyimide layer we just spun onto the wafer. At ASU, we use a Reflectometer in the form of a Filmetrics F20 system, so these processing steps will follow the technique we require for that process.
  - a. Note: We can program a new material into the Filmetrics Software if needed. A tutorial is being written about how to go about doing that. PI-2611 (soft-baked & cured), Parylene, AZ nLOF 2020, & AZ 15nXT are all present on the F20, soon to be added to the F40.
- 5. Ensure the PC at the F20 station is operational and move the mouse around if the monitor is blank.
- 6. Run a baseline test by grabbing the blank Silicon wafer with your Wafer Tweezers or Wand and placing it underneath the beam of the system.
- 7. Click "Baseline" on the F20 software.
- 8. The software will now ask you to tilt the object at 10°-80° to cancel out the background light it might see. Grab the Container the blank Silicon wafer was sitting on the place it next to the beam spot. Then grab the blank Silicon wafer and set it onto the container such that the flat part is

sitting on the base while the curved part is elevated and resting partially on the container. Click "OK" in the software to grab the measurement.

- 9. The software is now ready for processing, so start by loading the correct recipe.
  - a. Note: Load "PI-2611 (Cured)" from "Others" for cured films. Load "PI-2611 (Soft-Baked)" from "Others" for soft-baked films.
- 10. Once the recipe is selected, you will now have to select the correct nominal thickness. Click "Edit Recipe" to change the nominal thickness as well as the measurement type (Angstroms, nanometers, or micrometers).
- 11. The following Steps (12-19) should be repeated for every wafer that needs to be tested for thickness.
- 12. Place the wafer that needs to be inspected onto the base such that the center of the wafer is directly underneath the beam of the system. This can be checked by placing your Wafer Tweezers or Wand underneath the beam and barely above your wafer to give yourself a better idea of where the beam is landing.
  - a. Note: Never touch your Tweezers or Wand directly onto the wafer surface!
- 13. Click the "Measure" button on the software to grab the measurement. Write this measurement into a table in your Cleanroom Notebook as the "Center" location.
- 14. Move the wafer (using your Wafer Tweezers or Wand's edge) to the right such that the left middle of the wafer is now underneath the beam.
  - a. Note: For reference, I consider the major flat to be the bottom of the wafer and the opposite side to be the top of the wafer. This makes everything easier to think about in my mind. Orientation is a personal choice and does not matter, as long as it is consistent.
- 15. Click the "Measure" button on the software OR press the "Enter" key on the keyboard. Write this measurement into a table in your Cleanroom Notebook as the "Left Middle" location.
- 16. Move the wafer to the right such that the left side is now underneath the beam.
  - a. Note: You do not want to capture the far edges of the wafer as this is where the edge bead has formed. Make sure this measurement is not on the edge bead section.
- 17. Click the "Measure" button on the software OR press the "Enter" key on the keyboard. Write this measurement into a table in your Cleanroom Notebook as the "Left" location.
- Continue the previous steps to get the "Right Middle", "Right", "Top Middle", "Top", "Bottom Middle", and "Bottom" measurements. Write each measurement into the table in your Cleanroom Notebook.
  - a. Note: These specific locations are important because they ensure consistency and they also cover enough sections of the wafer that any major defects can be determined simply by thickness variance. Gathering 9 points of data also helps in statistical verification as well as getting a surface profile of the wafer every time a process is completed.
- 19. Using your Wafer Tweezers or Wand, scoot the wafer off the base platform and load it back into the Wafer Box it came from.
- 20. Repeat Steps 12-19 for every wafer in the batch that is needing to be tested for thickness.
- 21. Once all wafers are completed, take the data from the Cleanroom Notebook you wrote it on and transfer it to an Excel sheet (or Google Sheet) such that it can be analyzed for averages, standard deviations, and other statistical calculations. Saving all data into a common location can help with product tracing throughout the device's fabrication as well as ensuring that the processing steps remain consistent.

a. Note: Examples of the tables used in the Cleanroom Notebook as well as the Excel tables can be provided. This can all be recorded directly into an Excel Sheet or Google Sheet instead of onto a cleanroom notebook. I have to write the measurements onto a cleanroom notebook just because that is the nature of the lab in which the measurement tool is located in and I have not implemented a Google Sheet for this yet.

# **3** Photoresist Deposition for Metal Patterning

## 3.1 Equipment Needed

#### • Spinner

- Mask Aligner (i-line ~365 nm UV) w/ Z-axis calibration (~66 mJ/cm<sup>2</sup>)
- Fume Hood (for developer)
- Pyrex dish (>4" diameter) (Qty: 2)
- Hot Plate or Oven (110°C & 105-115°C)
- Nitrogen air gun (for wafer drying)
- Microscope (for inspection)
- Camera (for microscopic imaging)
- Timer
- Thickness Measurement Tool (Filmetrics F20 Reflectometer or 3D Profilometer)
- Refrigerator for storage of AZ nLOF 2020 Photoresist

### 3.2 Materials Needed

- Wafer Tweezers or Wand (4")
- Wafer (3" or 4") Silicon w/ Polyimide
- Cleanroom Notebook
- Cleanroom Wipes
- Marker for wafer labeling
- AZ nLOF 2020 Photoresist (~2 mL per wafer)
- 5" Soda Lime Glass mask plate (Clear for Mylar films OR complete Mask)
- Kapton Tape OR Blue Cleanroom Tape, Medium-Tack (for Mylar masks)
- AZ 300MIF Developer Solution (~100-200 mL per batch)
- DI Water (~100-200 mL per batch)
- Waste bottles for all chemicals (~2)

### 3.3 Deposition Process

#### 3.3.1 Photoresist Spinning

- 1. The process for spinning photoresist is going to depend on the type of spinner system you are using, so these steps will serve as a guideline for what we do here at ASU. Since we typically use the ASU NanoFab for these steps of the process, I will be following the procedure I would use in that cleanroom.
  - a. Note: The mask aligner we have in our cleanroom does not currently have control of the Z-axis nor the Y-axis due to some broken micro-manipulators.

- 2. The ASU NanoFab has 2 Brewer Science CEE Coater systems for spinning use. One of them has a hot plate included and the other does not. I will be walking through the operation of the coater that includes the hot plate (#1) so that the controls are known. If you are using the other coater system (#2), there is a hot plate to be used next to the coater system and just has to be preset to the specified temperature.
- 3. Touch anywhere on the interface screen of the Brewer Science CEE Coater #1 to wake it up from its "Sleep" mode.
- 4. If the software is not running (Windows background), double-click on the CEE Software shortcut in the upper right-hand side of the background.
- 5. Touch the "Log In" button in the upper right-hand side of the software interface.
- 6. Use the password "recipe" for normal use (spinning and hot plate)
  - a. Note: There is another password that can be used for higher functionality, including the ability to run the spinner with the lid up. This is useful for cleaning the coater once you have completed the spinning process. This password is "secret".
- 7. Once the password has been entered and accepted, click on the "Thermal" button to enter the controls of the hot plate.
  - a. Note: The only two button options here are "Spin" or "Thermal"
- 8. Touch the "Load Recipe" button near the top of the interface and select the recipe you would like to use.
  - a. When using the AZ nLOF 2020 resist, we require the use of a 110°C temperature for 60 seconds, so the "110C 60s" recipe works the best for us here.
- 9. Regardless of the current temperature of the hot plate, press the "Start Process" button to begin a cycle of the hot plate.
  - a. Note: This is just to get the hot plate temperature up to where we need it to go.
- 10. A pop-up window will appear if the temperature is outside of the setpoint's range asking if you would like to continue even though the temperature is out of range. Select "Yes" and allow the cycle to run.
- 11. While the temperature of the included hot plate is getting to its setpoint, you can begin to set the spinning system up. Lift the lid to the spinner chamber to allow for the tools to be installed.
  - a. Note: The process for the temperature to get up to setpoint will alarm after 1 minute since it completed the process it was running. Make sure to hit "OK" on the interface once this starts alarming. This may be after you have installed everything or during your installation process, so just be aware you will need to complete this before the annoying alarm destroys your sanity.
- 12. Grab a bowl from the top of the Solvent Hood (to the right of the spinner systems table) and place it inside the spinning chamber.
  - a. Note: There are 2 types of bowls to choose, 1 with a lip and 1 without a lip. If you are only doing 1-2 wafers, the bowl with a lip is best for easy cleaning after spinning. If you are processing more than 2 wafers (normal batch is 4 wafers), use the bowl without a lip so that everything can go down the drain once it reaches the bowl. The bowl without a lip likely requires the use of the "secret" password for the cleaning process.
  - b. Note: Make sure the entire bowl is flush with the chamber and is not sitting on the Stainless Steel rim surrounding the spinning chamber.
- 13. Grab the chuck toolbox and open it up (located underneath the spinning systems).

- 14. Select the corresponding wafer chuck and centering tool based on the size of wafers you are using.
  - a. Note: We typically use a 4" wafer, so the 4" centering tool and the 2.5" wafer chuck (requires screw) are what we typically use.
- 15. Install the wafer chuck with the post aligned with the slot in the wafer chuck. Screw this in using the allen wrench (red handle in the chuck toolbox)
  - a. Note: This is hard to describe in words, but you need to make sure the openings in the wafer chuck line up with the things sticking out of the spinner post. Not that this additional description actually helps, but it will make sense when you see the system, hopefully.
- 16. Place the centering tool next to the system for later use.
- 17. Select the "Run" tab on the bottom left-hand side of the interface.
  - a. Note: This step cannot be completed until the Thermal process has completed.
- 18. Select the "Spin" button from the center of the screen.
- 19. Press the "Load Recipe" button on the top left-hand side of the interface and load the recipe needed from the list.
  - a. When using the AZ nLOF 2020 photoresist, we typically use a 3,000 rpm spin speed and a 30 second duration. The "3000 30s KN" recipe includes a 500 rpm (5s) spreading step followed by the 3,000 rpm spin step (30s) and a 500 rpm (5s) cooldown step. This is the preferred recipe. You can save this off as another recipe name if you would like to have one you can always find. Be aware that this is a public system for anyone in the cleanroom to use, so whatever name you pick for a new recipe name would have to be unique and descriptive.
- 20. You can press the "Edit Recipe" button to check that the recipe is correct if you would like.
  - a. Note: Press the "Back" button on the lower right-hand side of the interface to return to the previous screen.
- 21. The system has now been set up and we can begin to process the wafers. Grab 2 cleanroom wipes and place them next to the spinning system.
  - a. Note: 1 of these cleanroom wipes will be used for cleaning the spinning system once you have completed your processing. The other cleanroom wipe will be used for the Mask Aligner system and for cooldown after the PEB (Post-Exposure Bake after the exposure in the Mask Aligner).
- 22. Grab the bottle of photoresist you are using and place it next to the spinning system, on the upper right edge of the cleanroom wipes.
  - a. Note: We store AZ nLOF 2020 in the refrigerator in ERC 320, so you should have placed this bottle inside of the toolbox you are taking into the cleanroom with you. The ASU NanoFab supplies resists such as AZ 3312, AZ 4330, and a couple others. They also supply HMDS if that is required for your processing. Anything else will have to be purchased when we run out and will be stored in our lab space.
- 23. Move the centering tool to the upper left edge of the cleanroom wipes next to the spinning system.
- 24. Grab the Wafer Box containing the wafers you will be processing and place it on the left side of the cleanroom wipes next to the spinning system.
- 25. Ensure the Wafer Tweezers are present next to the spinning system as well.
- 26. Open the bottle of photoresist and set the cap to the side.
- 27. Grab a "Large Pipette" from the bins between the spinning systems.

- 28. Squeeze the bulb of the pipette until you cannot squeeze it further.
- 29. Place the squeezed pipette into the bottle of photoresist and release the bulb once the tip has been fully submerged into the photoresist.
- 30. Open the lid to the spinning bowl.
- 31. Dispense the entirety of the pipette into the bowl in a circular fashion.
  - a. Note: We need to create a Solvent-rich environment for the photoresist to spin consistently every time, so this should be distributed throughout the entire bowl as evenly as possible.
  - b. Note: This is the first coating of two to ensure a complete environment.
- 32. Empty the entire pipette into the bowl, keep the bulb depressed, place it back into the bottle of photoresist, and release the bulb once the tip has been fully submerged into the photoresist.
- 33. Dispense the entirety of the pipette into the bowl in a circular fashion.
  - a. Note: We need to create a Solvent-rich environment for the photoresist to spin consistently every time, so this should be distributed throughout the entire bowl as evenly as possible.
  - b. Note: This is the second coating of two to ensure a complete environment.
- 34. Empty the entire pipette into the bowl, keep the bulb depressed, place it back into the bottle of photoresist, and release the bulb once the tip has been fully submerged into the photoresist.
- 35. Close the lid to the spinning chamber.
- 36. Steps 37-73 below should be repeated for every wafer in the batch you are trying to process.
- 37. Open the lid to the spinning chamber.
- 38. Grab the next wafer to be processed in your dominant hand.
- 39. Grab the centering tool in the other hand.
- 40. Place the centering tool around the wafer chuck such that its ridge is in contact with the bottom and side of the wafer chuck.
  - a. Note: Do not release the centering tool from its location until the vacuum has been established on the wafer.
- 41. Place the wafer onto the wafer chuck and push it against the centering tool with the edge of your tweezers.
  - a. Note: Maintain the same contact position with the centering tool during this time.
- 42. Using the tweezers hand, Press the "Hold" button on the interface.
- 43. This should illuminate the "Substrate Present" box in blue (upper right-hand side of the interface). Once that is illuminated in blue, you can remove the centering tool from its contact position on the wafer chuck.
- 44. Place the centering tool back on its place on the cleanroom wipes (upper left-hand side).
- 45. Press the "Start Centering" button on the bottom left-hand side of the interface.
- 46. Check to be sure the wafer is completely centered on the wafer chuck. If this is not the case, Press the "Release" button, remove the wafer from the wafer chuck, and repeat Steps 38-46 until the wafer is centered on the wafer chuck.
- 47. Grab the bottle of photoresist in one hand.
- 48. Grab the pipette from the bottle of photoresist in your dominant hand and pull it from the bottle such that the tip is above the photoresist level, but still within the opening of the bottle.
  - a. Note: The photoresist will be dripping from the pipette, so ensure these drops make it back into the bottle of photoresist.

- 49. Once the dripping has slowed to at least a couple seconds between drops, move the pipette from the bottle to the bowl in a swift (but cautious!) movement.
- 50. Once the dripping has slowed to at least a few seconds between drops, move the pipette to the center of the wafer and begin the dispensing process.
  - a. Note: The pipette should be held at approximately a 45° angle from vertical and the tip should be as close to the wafer as possible without touching the wafer or the photoresist.
- 51. To dispense the photoresist evenly and consistently, squeeze the bulb of the pipette with enough pressure that you have a steady and constant flow of photoresist coming out of the tip of the pipette.
- 52. Continue to dispense the photoresist until you have at least a half dollar-sized amount on the wafer or until you have a minimal amount of photoresist remaining in the pipette (and/or start seeing bubbles coming out of the pipette).
- 53. If any bubbles exist in the photoresist that was dispensed, now is the only time you have to get rid of them. Completely depress the bulb of the pipette (emptying any contents into the spinning bowl) and move the pipette to the surface of the photoresist, next to/on top of the bubble. Release the bulb slowly to get the bubble to go up into the pipette tip. Once the bubble has been sucked up into the pipette tip, stop releasing the bulb. Move the pipette from the wafer surface to the spinning bowl and squeeze the bubb completely to expel the bubble from the pipette. Repeat this step until all bubbles have been eliminated from the photoresist puddle.
- 54. Completely depress the bulb to the pipette (emptying any contents inside into the spinning bowl), keep it depressed, move it back to the bottle of photoresist, place the tip into the photoresist liquid, and release the bulb once the tip has gone entirely below the surface of the photoresist.
  - a. Note: DO NOT do this step if there are no more wafers to process. Instead, place the emptied pipette onto the cleanroom wipe for disposal later.
- 55. Place the bottle of photoresist back on its place on the cleanroom wipes (upper right-hand side).
- 56. Close the lid to the spinning chamber.
- 57. Ensure the "Lid Closed" and "Substrate Present" boxes on the interface are both illuminated in blue.
- 58. Press the "Start Process" button on the bottom left-hand side of the interface to begin the spinning process.
- 59. Wait for the process to complete. Once the process is complete, the system will alarm. Press the "OK" button when this alarm goes off.
- 60. After the alarm has been acknowledged, press the "Run" tab on the bottom left-hand side of the interface.
- 61. Select the "Thermal" option.
- 62. Ensure that the temperature is illuminated with a green background, denoting that the temperature is within its setpoint range.
- 63. Open the lid to the spinning chamber.
- 64. Grab the wafer with your Wafer Tweezers in your dominant hand and remove it from the spinning chamber.
- 65. Using your other hand, Press the "Start Process" button on the bottom left-hand side of the interface.
  - a. Note: The hot plate in this system has a 5 second step before it adds vacuum so that the wafer can be placed on the hot plate and adjusted to the center position before it adds a vacuum under the wafer and starts the 60 second timer.

- 66. Open the lid to the Hot Plate chamber with your free hand and place the wafer onto the hot plate such that the 2 standing posts are keeping it centered.
  - a. Note: This lid will not stay up, so you must keep it held with one of your hands while you are placing the wafer onto the hot plate.
- 67. Ensure the wafer achieves a vacuum (gets sucked down onto the hot plate) and close the lid to the Hot Plate chamber.
  - a. Note: If there is a previous wafer cooling down on the cleanroom wipe, remove it from the wipe and place it back into the Wafer Box.
- 68. Wait for the timer to get close to expiring (approximately 1-3 seconds remaining) and open the lid to the Hot Plate chamber.
- 69. The wafer should lift up off the hot plate for 5 seconds after the 60 second timer expires, so utilize this time to grab the wafer from the hot plate.
- 70. Place the wafer onto the cleanroom wipe below.
- 71. Close the lid to the Hot Plate chamber.
- 72. The system should alarm once the entire recipe has completed, so hit the "OK" button once this goes off to acknowledge the alarm.
- 73. Keep the wafer on the cleanroom wipe for at least 60 seconds for it to cool down before placing it back into the Wafer Box.
- 74. Repeat Steps 37-73 for all wafers in the batch.
- 75. Once all of the wafers have finished spinning and baking, you then begin the process of returning the system back to how you found it. While the last wafer is cooling down on the cleanroom wipe, you can prepare the system for cleaning. Do this by grabbing the Acetone and Isopropyl Alcohol (IPA) bottles from the General Waste Bin station (or the Solvent Hood).
- 76. Move the pipette from the cleanroom wipe to the Flammable/Solid Waste bin.
  - a. Note: if this still remains full inside the bottle of photoresist, empty its contents into the spinner's bowl completely and then place directly into the Flammable/Solid Waste bin.
- 77. Return the cap to the photoresist bottle and place it back where it came from.
  - a. Note: This should be the toolbox you came in with if it is AZ nLOF 2020, but it could be the dry box in the cleanroom if you were using one of the ASU NanoFab's provided photoresists.
- 78. Ensure the "secret" login information was used so that you have access to the Diagnostics page of the software.
  - a. Note: You can manually clean the spinning bowl and the system, but that just requires some cleanroom wipes and Acetone/IPA combination. I will outline the steps for cleaning when a whole batch was completed.
- 79. Click on the "Diag" tab at the bottom center of the interface.
- 80. Select the "Spin Diagnostics" option.
- 81. Press the spin speed and change it from 250 rpm to 1,000 rpm.
- 82. Press the ramp rate and change it from 1,000 rpm/s to 250 rpm/s.
- 83. Open the lid to the spinning chamber.
- 84. Grab a blank Silicon wafer (not part of a batch) with your dominant hand.
- 85. Grab the centering tool with your other hand.
- 86. Place the centering tool into its contact point with the wafer chuck.
- 87. Place the wafer onto the wafer chuck and push it against the centering tool with the edge of your wafer tweezers.

- 88. Without moving the centering tool from its location, use your tweezer hand to press the "Yes" button under the "Vacuum" section of the interface (center-bottom left-hand side).
- 89. Once you see a checkbox ("Wafer Present?") appear above the button you just pressed, you can remove the centering tool from the spinning chamber.
- 90. Ensure there are no wafers still cooling down on the cleanroom wipe before proceeding to the next step.
- 91. Spray Acetone onto the wafer currently on the chuck until the entire wafer has a coating of Acetone on it.
- 92. Press the "Start" button under the "Spin" settings of the interface to begin spinning.
- 93. Spray Acetone onto the center of the spinning wafer for 5 seconds.
- 94. Continue spraying Acetone onto the center of the spinning wafer and also spray IPA onto the center of the wafer for 5 seconds.
- 95. Stop spraying the Acetone onto the spinning wafer and continue spraying IPA onto the center of the spinning wafer for 5 seconds.
- 96. Stop spraying the IPA onto the spinning wafer and wait until all of the rainbow colored streaks disappear from the wafer.
- 97. Wait another 15 seconds after the rainbow colored streaks disappear.
- 98. Press the "Stop" button until the "Spin" setting of the interface to stop spinning.
- 99. Press the "No" button under the "Vacuum" section of the interface to release the vacuum on the wafer.
- 100. Grab the wafer off the wafer chuck and place it back into the Wafer Box.
- 101. Grab the allen wrench from the chuck toolbox.
- 102. Unscrew the wafer chuck and remove it from the system, placing it back in the chuck toolbox.a. Note: Make sure you keep track of the screw and place it in a spot inside the toolbox that
- is easy to find.103. Spray Acetone onto one of the cleanroom wipes (preferably the top one since it will need to be disposed of already).
- 104. Wipe the spinner's bowl with the Acetone-soaked cleanroom wipe. Be sure to clean the surface of the bowl, the backside of the bowl, the edges of the bowl, and all surfaces of the bowl.
- 105. Once the bowl has been thoroughly cleaned and looks as good as (or better than) it did when you first grabbed it, place it back above the Solvent Hood.
- 106. Spray more Acetone onto the same cleanroom wipe you were using to clean the bowl and wipe down the spinner chamber, the lid, and the Stainless Steel surfaces of the coating system.
- 107. Once the system has been thoroughly cleaned, dispose of the cleanroom wipe you were using to clean into the Flammable/Solid Waste bin.
- 108. If you had an additional pair of gloves on (I usually do if I'm doing photoresist or if I'll be cleaning a spinner system), take the extra layer of gloves off without getting the other gloves dirty and place the dirty gloves into the Flammable/Solid Waste disposal bin.
- 109. You are now ready for the Exposure stage of the Photoresist Deposition process (4.3.2).
  - a. Note: Be sure to stay in the photolithography bay of the cleanroom since white light would expose the photoresist you just completed and your pattern will not turn out as expected.

#### 3.3.2 Photoresist Exposure

- 1. In the ASU NanoFab, the OAI 808 Mask Aligner is the tool we use for patterning and UV exposure of the photoresists we use.
  - a. Note: We have a HTG Mask Aligner in the ERC 444 cleanroom, but due to the broken Z-axis controls, we cannot use the aligner for anything except gap exposure. The Y-axis controls are also broken, so any alignment besides the first layer (not requiring alignment) is impossible.
- 2. Since we use the AZ nLOF 2020 photoresist and it requires a post-exposure bake to cross-link the polymer chains, set up a hot plate near you for use at 110°C for 60 seconds
  - a. Note: I like to use the built-in hot plate of the Brewer Science CEE Coater #1, which I also like to use for spinning the photoresist on. If this Spinner and Hot Plate were used for the photoresist and have not been changed, you can use the same Thermal program you used previously. If it is currently in use, simply find another hot plate to use and make sure you have a timer (cell phone/smart watch works well) ready to go.
- 3. Place a cleanroom wipe for wafer cooldown after the post-exposure bake. This can be placed either next to the hot plate or next to the Mask Aligner, either one works.
- 4. The OAI 808 Aligner requires training prior to use and uses a badge access to activate the vacuum lines. Scan your ASU ID on the "Login" badge portal and it should flash green lights when you have done so.
- 5. The interface box is located to the right of the machine. This should have been left on the screen that has "Mask Vacuum" in the bottom right-hand corner. If it is not on this screen, press the "Main Menu" button (upper left-hand side of the interface) and select "Manual Test" (middle left option).
- 6. There is a log book for the system on the rolling cart next to the machine. Fill in the information (name, date, start time, resist type, filter or no filter, calibrating gap or not calibrating gap, wafer size, wafer thickness, mask size, mask thickness, frontside alignment).
- 7. You will also need to record the lamp hours from the lamp's power box. This is located underneath the system on the left-hand side. Write this number into the log book.
- 8. The system now has to be prepped for your wafer and mask setup, so check what the current mask chuck and wafer chuck sizes are. If they do not match what you are working with, the following steps will dictate how to install the proper mask chuck and wafer chuck.
  - a. Note: Currently, we are using 4" wafers with a 5" mask plate. Typically, your mask plate is at least 1" larger than the wafer diameter.
- 9. Slide the wafer chuck out towards you.
- 10. Loosen the screws at the front and back of the wafer chuck (2 screws closest to you and 1 screw at the back of the wafer chuck).
- 11. Undo the vacuum line connections (3 of them) at the bottom/back right of the wafer chuck.
  - a. Note: They should read "BALL", "SUB", and "CONT"
- 12. Remove the wafer chuck by lifting it up off the posts.
- 13. Swap the previous wafer chuck with the correct wafer chuck (located on the top of the rack to your right against the wall).
  - a. Note: Be careful of the vacuum lines coming out of the vacuum chuck so that you do not pinch these. I typically will place the previous wafer chuck such that the vacuum lines are going through one of the slots in the wires of the rack and hanging beneath it.
- 14. Place the correct wafer chuck onto the posts the previous one was sitting on.
- 15. Screw the 2 screws closest to you first, but do not tighten completely (only tighten a few turns)
- 16. Screw the 1 screw furthest from you until you feel it contact the wafer chuck.

- 17. Finish screwing the 2 screws closest to you until you feel them contact the wafer chuck.
- 18. Tighten all of the screws, starting with the back one and finishing with the 2 front ones.
- 19. Install all 3 of the vacuum lines into their respective connections. The wafer chuck has now been installed.
  - a. Note: The vacuum lines will have a label ("CONT", "BALL", and "SUB") and the connection ports will have matching labels. Match the vacuum line to the matching connection port. These can get tangled, so I recommend starting from the back connection port and working your way to the front connection port.
- 20. Loosen the four spring-loaded screws on top of the mask chuck to uninstall it from its location on the stack.
- 21. Undo the vacuum line connection to the left of the mask chuck. (Follow the vacuum line from the mask chuck to find this connection)
- 22. Remove the mask chuck by sliding it off the rails until it is clear of the microscope/camera lenses (alignment scope system) and then lift it off the system once it is clear of the lenses.
- 23. Swap the previous mask chuck with the correct mask chuck (located on the bottom of the rack to your right against the wall).
- 24. Install the vacuum line into the port on the left of the mask chuck. The mask chuck has now been installed.
- 25. Lay the mask chuck upside down (you should see a few posts and a spring system for mask installment) on the rolling cart next to the system.
- 26. The system is now ready for you to install your mask. Place a cleanroom wipe next to your toolbox.
- 27. Grab your 5" clear mask plate from the mask holder/carrier and place it onto the cleanroom wipe.
  - a. Note: If the wrong mask is currently installed, remove the blue tape holding it to the mask plate and place the tape on the corners of the mask plate for later use.
  - b. Note: If the correct mask is currently installed, skip to Step 29.
- 28. Grab the correct mask from your toolbox and install it such that the ink is facing towards you. Secure it to the mask plate with the blue cleanroom tape your removed from the previous mask.
  - a. Note: Typically we use a right-read down mask style, meaning that the ink is on the side that text appears backwards on. This is why I will always include some kind of text in the mask design. (The text should read normally when it is installed into the system and about to come into contact with the wafer)
- 29. Place the mask (with mask and blue cleanroom tape on the side facing towards you) onto the mask chuck and push it against the posts on the corners of the mask chuck.
- 30. There should be a spring-loaded post at one end of the mask chuck. Press this lever/button such that the post comes into contact with the mask plate.
- 31. Press the "Mask Vacuum" button on the interface. Ensure that the vacuum level (gauge on the far left of the machine) is at least to -10 inHg (or lower).
  - a. Note: If the mask vacuum is not high enough, try pressing down on the edges of the mask plate and/or reseating the mask.
- 32. Press the "Level Screen" button (top left-hand side of the interface) to prepare for the next step on the interface.
- 33. Carefully flip the mask chuck such that the mask is on the bottom side and place it onto the rails you first uninstalled it from.

- 34. Slide the mask chuck onto the rails and align the spring-loaded screws with the holes in the structure. Tighten these screws once they are in position.
  - a. Note: Ensure the positioning is correct by checking that the vacuum line is coming out towards you and is on the left side of the mask chuck (closest to the connection port)
- 35. The following steps (36-58) should be repeated for every wafer in the batch that need to get exposed.
- 36. Slide the wafer chuck out towards you.
- 37. Grab the next wafer to be processed from its Wafer Box and place it on the wafer chuck.
- 38. Align the wafer on the wafer chuck by making sure the flat is pushed against the two pins closest to you and that the edge of the wafer is pushed against the pin on the right edge of the wafer.
- 39. Press the "Substrate Vacuum" button (upper-middle left-hand side of the interface). Ensure that the vacuum level (gauge on the far left of the machine is at least to -10 inHg (or lower).
  - a. Note: If the substrate vacuum is not high enough, try pressing down on the edges of the wafer and/or reseating the wafer. Also make sure the wafer is not sitting on top of the pins you pushed it up against.
- 40. Push the wafer chuck away from you and into the system's stack.
- 41. Check the Exposure Time (upper-center right-hand side of the interface) and if it isn't correct, Press the "Main Menu" button (upper left-hand side of the interface).
- 42. Press the "Process Settings" button (upper right-hand side of the interface).
- 43. Press the "Exposure Time" box (upper right-hand side of the interface).
- 44. Change the time to the required exposure time.
  - a. Note: We use 6.6 seconds for our system since it is set to ~10 mW/cm<sup>2</sup> and we require 66 mJ/cm<sup>2</sup> of energy.
- 45. Press the "Main Menu" button (upper left-hand side of the interface).
- 46. Press the "Level Screen" button (center right-hand side of the interface).
- 47. Press the "Level" button (bottom left-hand side of the interface) and wait for the wafer chuck to get up to the height of the mask.
- 48. Calibrate the gap between the wafer chuck and the mask FOR THE FIRST WAFER ONLY. (Skip if not the first wafer)
  - a. Press the "Cal Gap" button (lower left-hand side of the interface).
  - b. Grab the caliper, turn it on, and place it onto the mask chuck such that its pin is in the center of the mask and the sides are resting on the top of the mask chuck.
  - c. Change the movement increment to 10 µm by pressing on the increment number.
  - d. Lower the wafer chuck (by 10  $\mu m$  increments) until the reading on the caliper is no longer changing AND keep going until it is at least at 100  $\mu m$  past that point to make sure.
  - e. Press the "Origin" button on the caliper to return the reading to 0.000 mm.
  - f. Raise the wafer chuck slowly (by 10  $\mu m$  increments) until the caliper changes to any value besides 0.000 mm.
  - g. Change the movement increment to 3  $\mu m$  by pressing on the 10  $\mu m$  number.
  - h. Raise the wafer chuck slowly (by 3 µm increments) until the caliper reads -0.004 mm.
  - i. Record the number in the "Gap" reading in the log book.
  - j. Press the "Zero Gap" button (lower right-hand side of the interface)
  - k. Press the "Run Screen" button (upper left-hand side of the interface) to return to the Run Screen.

- 49. Line up the wafer to the mask by using the micromanipulators at the base of the wafer chuck. Make sure that the mask is in the correct location on the wafer.
- 50. Press the "Contact" button on the interface (upper-center left-hand side of the interface) and wait a few seconds for that to activate.
- 51. Press the "Hard Contact" button on the interface (upper-center right-hand side of the interface) and wait a few seconds for that to activate.
- 52. Press the "Cycle" button on the interface (lower right-hand side of the interface). The system will now move the UV lamp head over the wafer and mask, open the shutter for the specified duration, and return to its original position. It will also lower the wafer chuck back to its loading location and switch the interface to the "Level Screen" where you will see the "Substrate Vacuum" and "Level" buttons.
- 53. Slide the wafer chuck out towards you.
- 54. Press the "Substrate Vacuum" button (upper-center left-hand side of the interface) to release the vacuum from the wafer.
- 55. Grab the wafer from the wafer chuck and take it over the the Hot Plate which should be previously set up for you.
- 56. Place the wafer on the hot plate and start the timer (or press "Start Process" if you are using CEE Coater #1)
- 57. Once the timer has completed, remove the wafer from the hot plate and place it on a cleanroom wipe. This can be located either next to the hot plate or at the Mask Aligner. Leave the wafer on the cleanroom wipe for at least 60 seconds.
- 58. Remove the wafer from the cleanroom wipe and place it back into its slot in the Wafer Box.
- 59. Repeat Steps 36-58 for each wafer in the batch that you are working with.
- 60. Once you have completed processing on all wafers in the batch, push the wafer chuck away from you and back into the system (without a wafer on it).
- 61. Unscrew the 4 spring-loaded screws in the mask chuck.
- 62. Slide the mask chuck out on the rails until it is past the microscope/camera lenses.
- 63. Lift the mask chuck off the rails and flip the mask chuck such that the mask is facing toward the ceiling.
- 64. Place the mask chuck on the rolling cart next to the machine.
- 65. Press the "Main Menu" button (upper left-hand side of the interface).
- 66. Press the "Manual Test" button (center left-hand side of the interface).
- 67. Press the "Mask Vacuum" button (bottom right-hand side of the interface).
- 68. Pull the spring-loaded post (on the mask chuck) away from the mask.
- 69. Remove the mask from the mask chuck and place it back into the mask holder/carrier (mask and tape side facing down). Close the mask holder/carrier when it is in place and return this to the toolbox you brought into the cleanroom with you.
- 70. Flip the mask chuck to its normal orientation, place it back onto the rails it was on, and slide it all the way back to its location.
- 71. Tighten at least 2 of the spring-loaded screws down.
  - a. Note: I usually do either 2 opposite corners or all 4.
- 72. Press your ASU ID against the badge portal that says "Logout". It should flash green and then both the "Login" and "Logout" portals should be red.
- 73. Write the total time you exposed the wafers for (adding up all of the times you used for the batch you just completed) and write the end time into the log book.

74. You are now ready for the Developing process.

#### 3.3.3 Photoresist Developing

- 1. Our AZ nLOF 2020 photoresist has a recommended Developer solution of AZ 300 MIF. This chemical is available in both the ASU NanoFab and in the ERC 444 cleanroom. If you are doing your photoresist processing in the ASU NanoFab, it is recommended to just do this step in the NanoFab as well.
- 2. Find a clean working bench at a Base Hood to prepare your work station for this process at.
- 3. Place two cleanroom wipes on the bench, with one near a sink.
- 4. Grab a Pyrex dish from the supply rack (some say "300 MIF" on them and those are preferred to be used) and place it on the cleanroom wipe furthest from the sink.
  - a. Note: The other cleanroom wipe will be used for drying the wafers after the processing.
- 5. Place a timer next to the dish for 300 MIF. (The Stopwatch function will be needed)
  - a. Note: The AZ nLOF 2020 resist typically takes about 30 seconds for complete development.
- 6. Place your Wafer Box containing wafers needing to go through the developing process inside the Hood, next the the dish for 300 MIF.
- 7. Place your wafer tweezers or wand next to the Wafer Box.
- 8. Don the proper PPE required for chemical handling (apron, base gloves, & face shield).
  - a. Note: Technically this is only required when handling the chemicals, so they can be removed once you have put the 300 MIF bottle back in its storage location and have the Hazardous Waste bottle for 300 MIF (and the funnel) ready to go at the fume hood.
- 9. If you are more than a couple steps away from the storage location of the 300 MIF, grab a chemical transport container.
- 10. Grab the 300 MIF bottle from its storage location. (Place this into the chemical transport container if that is required here)
- 11. Bring the bottle of 300 MIF to the Base Hood you will be operating out of.
- 12. Open the cap to the bottle and pour 300 MIF into the dish until the chemical level is at least 1/2" deep. (This should be approximately 100-200 mL, depending on the size of the dish)
- 13. Return the cap to the bottle of 300 MIF.
- 14. Return the 300 MIF bottle to its storage location. (Use the chemical transport container if necessary).
- 15. Make sure there is a Hazardous Waste bottle for 300 MIF at the Base Hood as well. If this is not at the hood already, grab it from its storage location (using the chemical transport container if necessary) and transport it to the hood).
- 16. Make sure there is a funnel at the hood for ease of pouring into the Hazardous Waste bottle when completed.
  - a. Note: If you would like to remove your apron, face shield, and base gloves, you may do so at this point.
- 17. The ASU NanoFab has a sink and a drain attached to it and can be easily used to simply rinse the wafers once they are done developing. If you are in ERC 444 (or at Mayo Clinic), a bath setup will likely be needed for the water rinse step. For this bath setup, you should fill a dish (approximately the same size as the one for the 300 MIF solution) up to the same 1/2" level you

filled the 300 MIF dish up to (~100-200 mL depending on the dish). This will also require an additional cleanroom wipe.

- 18. The following steps (19-39) should be repeated for each wafer in the batch that require developing.
- 19. Start the water flow from the sink if you are using the sink for the rinse and not a bath.
- 20. Grab the next wafer to be processed from the Wafer Box in your dominant hand.
- 21. In your other hand, grab the timer and prepare to begin the Stopwatch.
- 22. Simultaneously, begin the Stopwatch as you place the wafer into the 300 MIF solution. Make sure the entire wafer gets submerged into the 300 MIF solution.
- 23. Using the tip of your tweezers (and holding vertically), grab the side of the wafer by the major flat (by pushing against the wafer) and slowly move the wafer up and down in the solution. Be sure to not let the wafer breach the surface of the solution.
  - a. Note: This is performed to agitate and create movement in the solution so that the chemical solution can access all the photoresist on the wafer. This motion can be replaced if there is a stirrer or other similar device to create this movement.
- 24. While the wafer is in the developer solution, you should be able to see the photoresist coming off to form the pattern that you created from the Mask Aligner.
- 25. Once the Stopwatch reaches 27 seconds, pull the wafer from the solution to check if it has completed its processing.
- 26. If the wafer has completed its processing (and you can see the pattern you used at the Mask Aligner), move the wafer to the water stream coming from the sink. If the wafer needs to continue processing, place it back into the solution and repeat Steps 23-26 until completed.
  - a. Note: If using the water bath instead of the sink, place the wafer into the water bath.
- 27. Rinse the front of the wafer (pattern) by running it across the stream of water a few times.
- 28. Rinse the back of the wafer (no pattern, label) by running it across the stream of water a few times.
- 29. Rinse the front of the wafer (pattern) again by running it across the stream of water a few times.
- 30. Place the wafer onto the cleanroom wipe dedicated to drying the wafers.
- 31. Stop the flow of water coming from the sink.
- 32. Grab the Nitrogen air gun and dry the wafer starting in the center.
- 33. Rotate your wrist (NOT your arm/elbow) and push the liquid towards the edges of the wafer until there is no more water on the front of the wafer.
- 34. Dry off your wafer tweezers away from your wafer (so that no liquid splashes onto your dried wafer).
- 35. Pick up your wafer from the cleanroom wipe and place it vertically back on the cleanroom wipe so that you can dry the back side of the wafer.
- 36. Dry the back side of the wafer in the same fashion you dried the front (start in the center and rotate your wrist to push the liquid to the edges of the wafer). The wafer does not have to be exactly vertical, but it just cannot be touching any of the faces (front or back) to the cleanroom wipe.
- 37. Lift the wafer off the cleanroom wipe and pass the Nitrogen air gun across the edge of the wafer (back and forth) across the entire wafer (rotating your wrist to access the rest of the wafer).
  - a. Note: This is hard to put in words, but basically you are just pushing any liquid from the edge of the wafer onto one of the surfaces (front or back) to be dried again.

- 38. Face the front of the wafer towards you and dry off any liquid that got pushed back onto the front of the wafer from the edge dry.
- 39. Place the completed wafer into the Wafer Box for later inspection.
- 40. Repeat Steps 19-39 for each wafer in the batch that you are completing through this stage of the process.
- 41. Once all wafers in the batch have completed their Development process, you can begin the teardown of the station. Start by moving the timer, Wafer Box, and wafer tweezers out of the way and onto another table top.
- 42. Don the PPE for Base chemical handling if you do not have it on anymore (face shield, apron, and base gloves).
- 43. Move the Hazardous Waste bottle for 300 MIF to a location in which you can pour the contents of your dish into it.
- 44. Remove the cap to the waste bottle and place the funnel into the bottle.
- 45. Carefully pour the contents of the dish of 300 MIF into the waste bottle through the funnel.
- 46. Once all the contents have been emptied into the waste bottle, spray/add water to the dish until the water is at least three-quarters as high as the chemical solution was.
- 47. Swirl the water around the dish so that the water can remove any residue 300 MIF solution from the dish.
- 48. Empty the dish into the waste bottle.
- 49. Spray/Add a little bit of water to the dish, swirl it around, and empty it into the waste bottle.
- 50. Spray/Add at least 100 mL of water to the dish, swirl it around, and empty it into the sink (down the drain).
- 51. Repeat Step 50 for rinsing the 300 MIF dish (Completed twice total).
- 52. Using the cleanroom wipe that the 300 MIF dish was sitting on, dry out the dish you just rinsed with water. Place this cleanroom wipe in the "Flammable/Solid Waste" bin.
- 53. Place this dish to the side for returning to its storage location later.
- 54. If you were using a dish for a water bath, swirl the contents around and empty it into the waste bottle for 300 MIF.
- 55. Spray/Add a little more water to the water dish, swirl it around, and empty it into the waste bottle for 300 MIF.
- 56. Spray/Add at least 100 mL of water to the dish, swirl it around, and empty it into the sink (down the drain).
- 57. Repeat Step 56 for rinsing the water dish (Completed twice total).
- 58. Using the cleanroom wipe that the water dish was sitting on, dry out the dish you just rinsed with water. Place this cleanroom wipe in the "Flammable/Solid Waste" bin.
- 59. Place this dish to the side for returning to its storage location later.
- 60. Tap the funnel on the side of the waste bottle a few times to get some last remaining drops off it.
- 61. Screw the cap back onto the waste bottle.
- 62. Move the funnel back to its original location.
- 63. Move the waste bottle to its original location (using the chemical transport container if necessary).
- 64. Remove the PPE you donned for the Developer chemical handling and place it back where you got it from.
- 65. Return the dishes back to their storage locations.
- 66. Make sure the water is not running or dripping from the faucet.

- 67. Using the cleanroom wipe you used for drying the wafers, wipe down the surface area of the hood that you just used for the processing. Grab more cleanroom wipes if necessary. Ensure that the fume hood surface looks as good (if not better) than when you started using it. Place any used cleanroom wipes in the "Flammable/Solid Waste" bin.
- 68. You have completed the Development stage of the process and your wafers are ready for inspection.

#### 3.3.4 Inspection

- 1. There are three methods of inspection we use to measure the quality of photoresist we get after spinning, exposure, and developing.
  - a. The first is a visual inspection using our eyes.
  - b. The second is a visual inspection using a microscope.
  - c. The third is a metric inspection using a measurement tool to get the thickness of the layer.
- 2. As a first inspection, look at the wafer and ensure you have gotten the correct pattern to what you were setting out to get from the photoresist/mask combination. Also inspect for defects like bubbles, dirt, debris, and any voids in the photoresist material. The color combination is also an effective tool to ensure there are not too many striations in the colors, meaning that something didn't quite come out as planned. Luckily, photoresist can always be reworked, so this entire process can be repeated if necessary.
- 3. The second inspection involves using a microscope to inspect any potential defects found as well as to check the correct exposure energy and developing time. Check the two squares used for alignment for checking for the correct exposure energy and development times, based on the connection at the corners. (Bridge versus gap). Using the microscope can also inspect the wafer for any mask issues that you did not already notice.
- 4. The third inspection is where we measure the thickness of the photoresist layer we just deposited onto the wafer. At ASU, we use a Reflectometer in the form of a Filmetrics F40 system (under yellow lights) or a Filmetrics F20 system (under white lights). The following processing steps will follow the technique we require for that process.
  - a. Note: We can program a new material into the Filmetrics Software if needed. A tutorial is being written about how to go about doing that. PI-2611 (soft-baked & cured), Parylene, AZ nLOF 2020, & AZ 15nXT are all present on the F20, soon to be added to the F40.
- 5. Ensure the PC at the F40 station is operational and move the mouse around if the monitor is blank.
- 6. Run a baseline test by grabbing the blank "Baseline" Silicon wafer with your Wafer Tweezers or Wand and placing it underneath the microscope of the system.
- 7. Once the wafer has been placed underneath the microscope, find an edge on the wafer to focus the optics onto.
- 8. Click "Baseline" on the F20 software and then click "OK".
- 9. The software will now ask you to tilt the object at 10°-80° to cancel out the background light it might see. Grab the Container the blank Silicon wafer was sitting on the place it next to the beam spot. Then grab the blank Silicon wafer and set it onto the container such that the flat part is

sitting on the base while the curved part is elevated and resting partially on the container. Click "OK" in the software to grab the measurement.

- a. Note: This is tricky because this system uses a microscope to perform the analysis. Be careful around the optics to not damage them. It may make it easier to drop the stage by a set amount to be able to run this test.
- 10. Grab one of the other wafers in the calibration stack and check the system to make sure it is reading correctly.
- 11. The software is now ready for processing, so start by loading the correct recipe.
  - a. Note: Load "AZ nLOF" from "Photoresists".
- 12. Once the recipe is selected, you will now have to select the correct nominal thickness. Click "Edit Recipe" to change the nominal thickness as well as the measurement type (Angstroms, nanometers, or micrometers).
- 13. The following Steps (14-19) should be repeated for every wafer that needs to be tested for thickness.
- 14. Place the wafer that needs to be inspected onto the microscope such that the center of the wafer is directly underneath the beam of the system. Focus the microscope onto a feature on the surface of the wafer.
- 15. Click the "Measure" button on the software to grab the measurement. Write this measurement into a table in your Cleanroom Notebook as the "Center" location.
- 16. Move the wafer (using the stage manipulator) to the right such that the left middle of the wafer is now underneath the microscope lens.
  - a. Note: For reference, I consider the major flat to be the bottom of the wafer and the opposite side to be the top of the wafer. This makes everything easier to think about in my mind. Orientation is a personal choice and does not matter, as long as it is consistent.
- 17. Click the "Measure" button on the software OR press the "Enter" key on the keyboard. Write this measurement into a table in your Cleanroom Notebook as the "Left Middle" location.
- 18. Move the wafer to the right such that the left side is now underneath the microscope lens.
  - a. Note: You do not want to capture the far edges of the wafer as this is where the edge bead has formed. Make sure this measurement is not on the edge bead section.
- 19. Click the "Measure" button on the software OR press the "Enter" key on the keyboard. Write this measurement into a table in your Cleanroom Notebook as the "Left" location.
- Continue the previous steps to get the "Right Middle", "Right", "Top Middle", "Top", "Bottom Middle", and "Bottom" measurements. Write each measurement into the table in your Cleanroom Notebook.
  - a. Note: These specific locations are important because they ensure consistency and they also cover enough sections of the wafer that any major defects can be determined simply by thickness variance. Gathering 9 points of data also helps in statistical verification as well as getting a surface profile of the wafer every time a process is completed.
- 21. Using your Wafer Tweezers or Wand, scoot the wafer off the base platform and load it back into the Wafer Box it came from.
- 22. Repeat Steps 14-21 for every wafer in the batch that is needing to be tested for thickness.
- 23. Once all wafers are completed, take the data from the Cleanroom Notebook you wrote it on and transfer it to an Excel sheet (or Google Sheet) such that it can be analyzed for averages, standard deviations, and other statistical calculations. Saving all data into a common location can

help with product tracing throughout the device's fabrication as well as ensuring that the processing steps remain consistent.

a. Note: Examples of the tables used in the Cleanroom Notebook as well as the Excel tables can be provided. This can all be recorded directly into an Excel Sheet or Google Sheet instead of onto a cleanroom notebook. I have to write the measurements onto a cleanroom notebook just because that is the nature of the lab in which the measurement tool is located in and I have not implemented a Google Sheet for this yet.

# **4** Substrate Surface Preparations

### 4.1 Equipment Needed

- Plasma Cleaner or RIE Machine
- Quartz ring (>4" diameter) for wafer containment
- Thickness Measurement tool (Filmetrics F20 Reflectometer or 3D Profilometer)

### 4.2 Materials Needed

- Wafer Tweezers or Wand (4")
- Wafer (3" or 4") Silicon w/ Polyimide
- Cleanroom Notebook
- Cleanroom Wipes
- Marker for wafer labeling

### 4.3 **Preparations Process**

#### 4.3.1 Inspection

 As with almost anything we are doing in this process, the wafers will need to be inspected prior to continuing in the processing. In this stage of the processing, however, it is crucial that we test both the Before thickness of the photoresist (and/or polyimide) as well as the After thickness of the photoresist (and/or polyimide). This will give us a better idea of the consistency we have in the process as well as the confidence of knowing that the photoresist layer is the appropriate thickness for the lift-off process (photoresist is approximately 10:1 versus the desired metal thickness, 7:1 is also acceptable)

- 2. Steps 3-22 should be repeated for both Before thickness measurements and for After thickness measurements.
- 3. At ASU, we use a Reflectometer in the form of a Filmetrics F20 system, so these processing steps will follow the technique we require for that process.
  - a. Note: We can program a new material into the Filmetrics Software if needed. A tutorial is being written about how to go about doing that. PI-2611 (soft-baked & cured), Parylene, AZ nLOF 2020, & AZ 15nXT are all present on the F20, soon to be added to the F40.
- 4. Ensure the PC at the F20 station is operational and move the mouse around if the monitor is blank.
- 5. Note: Steps 6-9 can be skipped if you have already used the system earlier in the day.
- 6. Run a baseline test by grabbing the blank Silicon wafer with your Wafer Tweezers or Wand and placing it underneath the beam of the system.
- 7. Click "Baseline" on the F20 software.
- 8. Click "OK" on the F20 software to grab the first baseline measurement.
- 9. The software will now ask you to tilt the object at 10°-80° to cancel out the background light it might see. Grab the Container the blank Silicon wafer was sitting on the place it next to the beam spot. Then grab the blank Silicon wafer and set it onto the container such that the flat part is sitting on the base while the curved part is elevated and resting partially on the container. Click "OK" in the software to grab the measurement.
- 10. The software is now ready for processing, so start by loading the correct recipe.
  - a. Note: Load "PI-2611 (Cured)" from "Others" for cured films. Load "PI-2611 (Soft-Baked)" from "Others" for soft-baked films.
- 11. Once the recipe is selected, you will now have to select the correct nominal thickness. Click "Edit Recipe" to change the nominal thickness as well as the measurement type (Angstroms, nanometers, or micrometers).
- 12. The following Steps (13-20) should be repeated for every wafer that needs to be tested for thickness.
- 13. Place the wafer that needs to be inspected onto the base such that the center of the wafer is directly underneath the beam of the system. This can be checked by placing your Wafer Tweezers or Wand underneath the beam and barely above your wafer to give yourself a better idea of where the beam is landing.
  - a. Note: Never touch your Tweezers or Wand directly onto the wafer surface!
- 14. Click the "Measure" button on the software to grab the measurement. Write this measurement into a table in your Cleanroom Notebook as the "Center" location.
- 15. Move the wafer (using your Wafer Tweezers or Wand's edge) to the right such that the left middle of the wafer is now underneath the beam.
  - a. Note: For reference, I consider the major flat to be the bottom of the wafer and the opposite side to be the top of the wafer. This makes everything easier to think about in my mind. Orientation is a personal choice and does not matter, as long as it is consistent.
- 16. Click the "Measure" button on the software OR press the "Enter" key on the keyboard. Write this measurement into a table in your Cleanroom Notebook as the "Left Middle" location.
- 17. Move the wafer to the right such that the left side is now underneath the beam.
  - a. Note: You do not want to capture the far edges of the wafer as this is where the edge bead has formed. Make sure this measurement is not on the edge bead section.

- 18. Click the "Measure" button on the software OR press the "Enter" key on the keyboard. Write this measurement into a table in your Cleanroom Notebook as the "Left" location.
- Continue the previous steps to get the "Right Middle", "Right", "Top Middle", "Top", "Bottom Middle", and "Bottom" measurements. Write each measurement into the table in your Cleanroom Notebook.
  - a. Note: These specific locations are important because they ensure consistency and they also cover enough sections of the wafer that any major defects can be determined simply by thickness variance. Gathering 9 points of data also helps in statistical verification as well as getting a surface profile of the wafer every time a process is completed.
- 20. Using your Wafer Tweezers or Wand, scoot the wafer off the base platform and load it back into the Wafer Box it came from.
- 21. Repeat Steps 13-20 for every wafer in the batch that is needing to be tested for thickness.
- 22. Once all wafers are completed, take the data from the Cleanroom Notebook you wrote it on and transfer it to an Excel sheet (or Google Sheet) such that it can be analyzed for averages, standard deviations, and other statistical calculations. Saving all data into a common location can help with product tracing throughout the device's fabrication as well as ensuring that the processing steps remain consistent.
  - a. Note: Examples of the tables used in the Cleanroom Notebook as well as the Excel tables can be provided. This can all be recorded directly into an Excel Sheet or Google Sheet instead of onto a cleanroom notebook. I have to write the measurements onto a cleanroom notebook just because that is the nature of the lab in which the measurement tool is located in and I have not implemented a Google Sheet for this yet.
- 23. Once you have completed the Before thickness measurements (Steps 3-22) and the entirety of section 4.3.2 (Surface Roughening), you will now have to repeat Steps 3-22 of this section in order to get the After thickness measurements.

#### 4.3.2 Surface Roughening

- 1. Polyimide has a natural surface roughness with features of about 1-2 nm in height due to the long-chain nature of the polymer. Prior to deposition of metal layers, it is helpful to roughen the surface a little more so that the feature sizes increase to about 5-6 nm in height. This can be accomplished through a plasma cleaning process using an ignited oxygen plasma.
  - a. Note: This is a recommended process from the datasheet that does not necessarily need to be done, but it does help the metal layers adhere to the surface of the polyimide. I will be doing this for every wafer we process at ASU from now on.
- 2. In the ASU NanoFab, we use a PlasmaTherm PT-790 Reactive Ion Etching Tool for this "plasma cleaning" procedure. There is a Harrington Plasma Cleaning system in our smaller cleanroom, but this has not been tested yet for this purpose.
- 3. The PT-790 system is left under vacuum when not in use, so it allows us to prepare the chamber for our process before we even add our samples. Since we will be using an Oxygen plasma cleaning process, the recipe is called "Clean" and is designed to clean the chamber of any particles leftover from previous runs.
  - a. Recipe: "Clean" 50 sccm  $O_2$  200 W Forward Power 40 mTorr Pressure 10 minutes for a complete clean, 1-2 minutes for a quick chamber prep

- b. Note: Each recipe has 2 minutes that are tacked on top of whatever the duration time is so that the machine can evaporate all of the water molecules from the air (humidity), hold the gas flow amounts steady, hold the vacuum pressure steady, and ignite the plasma.
- 4. Click on the "Log In" button on the upper right-hand side of the interface.
- 5. A pop-up window should appear with a list of usernames on the left and a password box in the upper right. Select your username credentials from the list on the left.
- 6. Enter your password into the box on the upper right-hand side of the pop-up window.
- 7. Click the "Log In" button below the password box to complete the Log In process.
- 8. Select the bottom left "Recipes" menu button, Click on "Clean" from the list of recipes and Click "Run Recipe" on the left-hand side of the software. This will bring up a pop-up window asking if you are sure you would like to run the specified recipe. Ensure that it is the correct recipe ("Clean") and Click "Yes" in the bottom of the pop-up window.
- 9. Another pop-up window will show up and will ask you to enter the run time for the recipe. Enter 2 minutes (default is 10 minutes) and hit Tab when done. Then select "Start Recipe" towards the bottom of the pop-up window and the system will begin to go through the steps of the recipe.
  - a. Note: This can be set to any amount, but I use 2 minutes just to make sure the gas is flowing, ignited, and interacting with everything for at least a minimal amount of time.
  - b. Note: Actual run time is approximately 5 minutes.
- 10. Once the recipe is complete and the system reads "Pumping Idle", Click on "Vent" to bring the system up to atmosphere.
  - a. Note: Vent time is approximately 2-3 minutes.
- 11. Once the system reads "Vented" and the chamber is filled in with a blue color (in the "Services" tab), the system is at atmosphere and you can open the chamber lid. Do so carefully but with force (the system was just under vacuum, so might be hard to open, but it cannot be slammed open)
  - a. Note: When the chamber lid is open, you want to make sure the lid is open for the least amount of time possible, so make sure your wafers/samples are ready to go before you open the lid.
- 12. Steps 13-28 below should be repeated for each wafer you are requiring a surface roughening on.
  - a. Note: It is highly recommended to do 1 wafer at a time for this process as it helps to ensure an even roughening across the entire wafer.
- 13. Place your sample(s) in the center of the platen. A quartz ring is available if it is desired to ensure the sample stays in the direct center.
  - a. Note: Doing 1 wafer at a time is highly recommended. Multiple wafers bring about inconsistent etching rates and some "dead zones". If doing multiple wafers at a time, a plan for rotating the wafers will be needed (stop etch, vent, rotate, pump, resume etch) in order to get a consistent etch result
  - b. Note: The quartz ring is recommended when it is only one wafer going through the process to keep the wafer in the center of the platen.
- 14. Once the sample(s) and/or quartz ring are on the platen inside the chamber, you may shut the lid.
- 15. With the lid shut, click "Pump Turbo" on the interface.
  - a. Note: This button can be found on any tab on the interface, but it is recommended to use the "Services" tab as this will show the user when the chamber is under vacuum

- 16. Once the "Pump Turbo" button has been selected, weight must be placed on the chamber lid (lean down onto the handle of the lid) in order to keep it sealed while the vacuum systems begins to pump the chamber down.
  - a. Note: This should be done until the chamber is no longer filled in with a blue color ("Services" tab) and when the chamber pressure drops from 760 Torr to 10.0 Torr.
- 17. Wait for the system to achieve a chamber pressure of 0 mTorr before proceeding to the recipe selection.
- 18. Click on the "Recipes" tab at the bottom of the interface once the system reads "Pumping Idle" and is at 0 mTorr.
- 19. The "Clean" recipe should be highlighted from the previous run (chamber preparations or previous sample). If this is not highlighted, for some reason, Click on the "Clean" recipe.
- 20. Click "Run Recipe" on the left-hand side of the interface.
- 21. A pop-up window should appear, asking for confirmation that you have selected the correct recipe. If the recipe it lists reads "Clean", you have the correct recipe and can Click "Continue". If not, Click "Close" and re-select the correct recipe.
- 22. Another pop-up window should appear, asking for the duration of the recipe. The default is set to 10 minutes. Change this to 1 minute and hit the "Tab" key on the keyboard to confirm the change.
- 23. Click the "Run Recipe" button once the time reads "1 minute".
- 24. While the recipe is starting, now would be a great time to fill out the run log.
  - a. Note: This should include the Wafer ID (I use batch number-wafer number, i.e. B7-026), the etch time (1 minute), etch rate (approximately 1,700 Angstroms/minute), the material(s) being etched (Polyimide / PR), the platen material (Graphite), the substrate material (Silicon), the chamber pressure used in the recipe (40 mTorr), and any notes about the run you may want to enter (I usually leave it blank)
  - Note: You will need to record the Forward Power (typically 150 W for the Clean recipe), the Reflected Power (typically 1 W for the Clean recipe), and the DC Offset (typically 417 V for the Clean recipe). Be sure to get actual values from the run that you enter into the chart.
- 25. Once the recipe has completed and the system reads "Pumping Idle", Click on "Vent" to vent the system to atmosphere.
- 26. Wait for the system to reach 760 Torr, for the chamber to be filled in with a blue color ("Services" tab), and for the system to read "Vented".
- 27. Once the system reads "Vented" and the chamber has been filled in with a blue color ("Services" tab), open the lid to the system.
- 28. Remove the sample that has just completed the Surface Roughening and place it in your Wafer Box.
- 29. Repeat Steps 13-28 until all wafers have been processed.
- 30. Once all wafers in the batch have been processed, ensure there are no wafers (or quartz rings) on the platen and close the lid to the chamber.
- 31. Click the "Pump Turbo" button on the left-hand side of the interface.
- 32. Apply pressure to the handle of the lid to ensure the lid is sealed properly.
  - a. Note: This should be done until the system reads 10.0 Torr instead of 760 Torr and the chamber is no longer filled in with a blue color ("Services" tab).
- 33. Wait for the system to read "Pumping Idle" and is at a pressure of 0 mTorr.
- 34. Click on the "Recipes" tab on the bottom of the interface.

- 35. Select the "Clean" recipe if it is not already selected.
- 36. Click on the "Run Recipe" button on the left-hand side of the interface.
- 37. A pop-up window should ask for confirmation that you are running the correct recipe. If this reads "Clean", then click "Continue".
- 38. Another pop-up window should ask for duration of the run. Set this to 2 minutes and hit the "Tab" key on the keyboard to confirm the change.
- 39. Click the "Run Recipe" button if the duration is set to 2 minutes.
- 40. Once the recipe has begun running, Click the "Log In" button on the top right of the interface.
- 41. A pop-up window should appear asking for username and password. Click the "Log Out" button on the right-hand side of the pop-up window.

# **5** Conductive Metal Deposition

### 5.1 Equipment Needed

- PVD Machine (E-Beam preferable, Thermal Evap acceptable, Sputter if nothing else)
- Thickness Measurement Tool (Filmetrics F20 Reflectometer or 3D Profilometer)

### 5.2 Materials Needed

- Wafer Tweezers or Wand (4")
- Wafer (3" or 4") Silicon w/ Polyimide & Photoresist
- Cleanroom Notebook

- Cleanroom Wipes
- Marker for wafer labeling
- Platinum pellets
- Titanium pellets (still need to see if this is needed or not)
- Source Crucibles (minimum 2 per material)
- Mounts or Blue Cleanroom Tape (for mounting the wafers to the platen/machine)
- Spare crystals (for thickness monitoring)

## 5.3 Deposition Process

#### 5.3.1 E-Beam Evaporation

- The metal layer we use for the electrodes is currently Platinum and there are 2 main ways to deposit this onto the polyimide on the wafer's surface. Sputtering would be an effective method to get a conformal coating, but the targets are very expensive and it is not entirely what we are wanting from a lift-off process. E-Beam Evaporation is the other technique and it provides a more cost-effective approach while maintaining a profile that works best for lift-off.
- Here at ASU, we have a Lesker PVD75 PRO (Lesker #4) that performs the E-Beam Evaporation procedure for us quite well. There is also another Lesker PVD75 (Lesker #3) that we used previously, but the automation in the PRO version works best for processing in a shared lab setting.
- 3. Before beginning this process, you will need to make sure that you have the metal crucibles that we use for the Lesker E-Beam systems. These are stored in the locker we have in ERC 444, which requires a combination to enter.
  - a. Note: Combination is the 4-digit protocol number for the IACUC protocol we have for window craniotomies on rats
- 4. At the Lesker #4 system, sign in to the software using the box in the upper left-hand side of the interface.
  - a. Note: If your credentials do not work, there is a username and password taped to the door behind the interface screen. Should be Username of "CSSER" and Password of "2014"
- 5. Once you are signed in, you should see some menu items pop up on the top and right-hand sides of the interface. Open the door and check the current program that is loaded to see if it has the correct Pocket selection
  - a. Note: Should be "Pt\_Ti\_10kV" with Pocket 1: SiO2, Pocket 2: Ti, Pocket 3: Pt, Pocket 4: Al.
- 6. Change the current program if necessary
  - a. To do this, select "Auto/Manual" from the interface behind the door with the left joystick (pushing it to the right to select).
  - b. Select "Manual" with the right joystick (highlighting it).
  - c. Push the right joystick to the right to log in.
  - d. Enter the code "2-0-3-1" to enter Manual mode.
  - e. Select "Set Process" with the left joystick (scroll down to find it and then push the joystick to the right to select).

- f. Select the correct process (Pt\_Ti\_10kV) required using the right joystick (moving it down to scroll through and right to select).
- g. Once this is selected, select "Set Data" with the left joystick (scroll to find it and then push the joystick to the right to select).
- h. Select the correct data set required (Pt\_10kV) using the right joystick (moving it down to scroll through and right to select).
- i. Go back to "Set Process" and make sure this stayed at the previous selection you made. Change this if it is not correct.
- j. Confirm that the correct process and data are displayed on the top of the interface behind the door.
- k. Select "Auto/Manual" with the left joystick (pushing it to the right to select).
- I. Select "Automatic" with the right joystick (highlighting it).
- m. Push the right joystick to the left to log out.
- 7. Check that the current pocket is Pocket 3 (Platinum) on the interface behind the door (top left-hand side of the interface). If this is not correct, change this in the main interface.
  - a. On the main interface (front of the door), select the "Deposition" tab in the top-center of the interface.
  - b. Select the text box that displays "Set Crucible".
  - c. Type "3" and hit the "Enter" key on the keyboard.
  - d. Wait until the indicator to the right of the text box ("Crucible in Position") is highlighted in green.
- 8. Click the "Vent" button on the right-hand side of the interface.
- 9. While the system is venting, go to the tablet on the table to the left of the system and start a process log under your name so that you can have it ready for entering in data from the run.
- 10. Once the system is vented, you should see "Recipe Complete" on the dialog box (should be in the bottom left-hand side of the interface). Close this dialog box. (Button is in the lower right-hand side of the dialog box)
- 11. Open the door to the chamber.
- 12. Using the Nilfisk vacuum, suck out any dust, debris, and clumped material from the chamber.
  - a. Note: Make sure you vacuum out the crucible pockets
- 13. On the main interface (front of the door), check the "Crystal Quality" in the bottom-right hand side of the "Deposition" tab (this is the crystal's life). If it is below 60-70%, inform the ASU NanoFab staff (Todd) that this will need to be replaced.
- 14. Load your crucible(s) with metal into the pockets in the platten at the bottom center of the chamber. If you are loading more than 1 metal, follow these steps:
  - a. Place the Platinum crucible into the platten. (This should be in Pocket 3, so check this on the interface behind the interface door before loading).
    - i. Note: The e-beam will evaporate a spot in the back-center of the crucible, so align this accordingly so that the beam will not reach bare crucible.
  - b. On the interface behind the door, select "Auto/Manual" using the left joystick (pushing it to the right to select).
  - c. Select "Manual" with the right joystick (highlighting it).
  - d. Push the right joystick to the right to log in.
  - e. Enter the code "2-0-3-1" to enter Manual mode.
  - f. Using the left joystick, select "Set Pocket" (pushing it to the right to select).

- g. Move the right joystick up to highlight "Pocket 2".
- h. Hold the right joystick to the right to move the crucible platten to Pocket 2.
  - i. Note: Continue holding the joystick until the crucible platten has completed its movement and has settled on Pocket 2.
- i. Vacuum out the new crucible pocket in the platten before adding your crucible of metal.
- j. Place the Titanium crucible into the platten. (This should be in Pocket 2, so confirm that is what the interface reads in the top left-hand corner of the interface behind the door)
  - i. Note: The e-beam will evaporate a spot in the back-center of the crucible, so align this accordingly so that the beam will not reach bare crucible.o
- k. On the main interface (front of the door), change the "Set Crucible" to "2" (under the "Deposition" tab).
  - i. Wait until the indicator to the right of the text box ("Crucible In Position") is highlighted in green before proceeding.
  - ii. Note: I do this so that it prevents an error from popping up on the interface behind the door. It is not necessarily required, but I would rather prevent any additional steps.
- I. Confirm that the correct process and data are displayed on the top of the interface behind the door.
- m. Select "Auto/Manual" with the left joystick (pushing it to the right to select).
- n. Select "Automatic" with the right joystick (highlighting it).
- o. Push the right joystick to the left to log out.
- 15. On the right-hand side of the main interface (front of door), select the "Run Recipe" button.
- 16. A pop-up window will appear on the left side of the interface. Find the "Sample Load Manual Operation Only" recipe, select it, and click "Start Process" in the lower left-hand side of the pop-up window.
  - a. Note: This process will home the wafer stage and then drop the shield so that the stage can be removed. DO NOT keep your hands inside the chamber while this process is running to ensure that you do not injure your hands from the moving metal shield.
- 17. Once the shield has dropped down, the recipe will "Pause" itself and wait for you to remove the wafer stage, add your samples, and return the wafer stage (with samples attached) to its position in the chamber. To remove the wafer stage, simply lift it up and off the pins it sets on and slide it out from the pins, down the chamber, and out of the chamber.
  - a. Note: Be very careful when removing the wafer stage to not knock it into the crystal sensor (right side of the chamber). Doing so could cause damage to the crystal and could potentially require replacement by ASU NanoFab staff (Todd)
- 18. Place the wafer stage on the table (I typically use the one behind Lesker #4 and to the left of Lesker #1) with the pins facing the table (upside down from what it was in the chamber).
- 19. Install your wafers onto the wafer stage. The process will slightly vary based on your size of wafers and the quantity you have in the batch, but the following steps should be a baseline:
  - a. Assuming 4" (100 mm) wafers and 4 wafers per batch, the wafers will be placed in a square surrounding the center of the wafer stage.
  - b. Place the wafers on the stage such that their major flats are facing towards the outside of the stage and not towards each other.
  - c. Ensure the wafers are as close as possible without touching or overlapping.

- d. Using the Kapton tape (found in the toolbox), secure at least 2 sides of each wafer to the stage.
  - i. Note: I use 3 pieces of tape and ignore the sections of the wafers closest to the center of the stage. Total of 12 pieces of tape for all 4 wafers in the batch.
- e. Once all wafers in the batch have been secured, carefully & slowly tilt the stage to the vertical orientation to see if any wafers move in the process.
  - i. Note: I do this as a safety check to make sure I won't have a wafer fall from the stage and break inside the chamber.
- 20. Grab the loaded wafer stage and move it back into the chamber. (Remember to return it to its correct orientation before attempting to load it back into the chamber).
- 21. Slowly and carefully put the stage into the chamber, ensuring that you are avoiding the crystal sensor, and raise it up so that it can rest on the pins it started on.
- 22. Ensure the stage is on the pins and secure before letting go of the stage.
- 23. On the main interface (front of door), select the "Resume" button on the pop-up recipe window to return the shield to its normal protective location.
- 24. Once the shield is back into position, the recipe pop-up window should display "Recipe Complete". Click the "Close" button to close this pop-up window.
- 25. Click the "Vacuum" tab from the top of the interface.
- 26. Close the chamber door and hold it closed with your hands with weight into it (either body weight or muscle).
  - a. Note: Keep this closed until you see the "Vac Lock Switch" activate and highlight in green.
- 27. Click the "Pump" button on the right-hand side of the interface.
- 28. Once you see the "Vac Lock Switch" highlighted in green, you can release the door.
  - a. Note: There should also be an audible "click" when this happens, but be sure that it was your system and not another in the lab if you are basing your procedure off of the audible sound.
- 29. The system will now pump down until it reaches 5e-5 Torr (50 μTorr). It is recommended to stand by the system until it reaches this vacuum level just in case it runs into an error.
- 30. Once the system has reached its minimum pump down level, a number will be displayed in the pop-up recipe window. This number is the "Pump Time Remaining" and should be recorded in the process log on the tablet.
- 31. Enter the log information into the process log on the tablet.
  - Note: If using Titanium/Platinum stack, the following is a sample for how to fill out the log. If only using Platinum, Material 2 becomes Material 1 and there is no other material to use.
    - i. Material 1: Titanium || Pocket: 2 || Target Thickness: 50 Å (depends on what you are trying to achieve) || Rate: 1 Å/s (depends on what you are trying to achieve)
    - Material 2: Platinum || Pocket: 3 || Target Thickness: 1000 Å (depends on what you are trying to achieve) || Rate: 1 Å/s (depends on what you are trying to achieve)
    - iii. Pump Time Remaining: record the number from the interface
    - iv. Pump Pressure: Leave blank until deposition has begun.
- 32. Double click on the Pressure reading in the bottom center of the interface to enlarge this to an almost full-screen reading. This will allow us to read the current pressure from outside of the cleanroom so that we can know if the system is ready when we come back.

- 33. The system will continue to pump down indefinitely, so we can leave the cleanroom and return after about 1 hour in order to let the system pump down to at least 5e-6 Torr (5 µTorr).
  - a. Note: From what I have seen so far, the lower that pressure is, the more consistent the deposition will be. Typically 1 hour results in a pressure of 5e-6 or 6e-6 while 2 hours results in 1e-6. The wait time all depends on when the pump started and what the schedule of the machine looks like when you start it.
- 34. Assuming that the machine has reached the desired starting pressure, return to the system in the cleanroom and close the enlarged window that is displaying the current chamber pressure.
- 35. Enter the current pressure reading into the process log in the tablet next to the system.
  - a. Note: If you are doing a Titanium deposition before Platinum deposition, always record the pressure at which the Platinum deposition started and not when the Titanium deposition started.
- 36. Go to the "Deposition" tab on the main interface (front of door) and ensure that the "Set Crucible" setting is the correct Pocket number.
  - a. Note: This should be "2" if Titanium will be deposited first and should be "3" if you are only depositing Platinum.
- 37. Start the graph charting of your materials by clicking on the "Graph" icon.
  - a. Note: The charting is an optional step and is not needed, but I like to do this just to monitor the system's performance.
- 38. Select the following data to be plotted and click the "Add to Graph" button in the left-middle side of the graph's pop-up window.
  - a. Shutter  $\rightarrow$  Substrate Shutter
  - b. Crystal  $\rightarrow$  Xtal1 Rate
  - c. Crystal  $\rightarrow$  Xtal1 Thickness
- 39. Click "Start Graph" in the lower left-hand side of the graph's pop-up window to begin plotting the data.
- 40. Click "Hold on Top" in the lower left-hand side of the graph's pop-up window to keep it in front of the Lesker software.
  - a. Note: This step can be done later so that you do not run into problems with screen real-estate when you begin the deposition process, but I prefer to do it earlier so that I don't forget it.
- 41. Monitoring the heating of the chamber/substrate is not directly monitored, but the following steps will allow you to see the heating of the pin side of the wafer stage (not the wafer side of the wafer stage):
  - a. Click on a new "Graph" icon from the "Deposition" tab of the main interface.
  - b. Select the "Heating" option from the drop-down list and click the "Temperature" option from the list.
  - c. Click "Add to Graph" in the left-middle of the graph's pop-up window
  - d. Click "Start Graph" in the bottom left-hand side of the graph's pop-up window to begin plotting the data.
  - e. Click "Hold on Top" to keep the graph's pop-up window in front of the Lesker software.
    - i. Note: This step can be done later so that you do not run into problems with screen real-estate when you begin the deposition process, but I prefer to do it earlier so that I don't forget it.
- f. Position the 2 graph pop-up windows such that you can see both graphs and so that you can still see the E-Beam power percentage as well as the current rate, thickness, and other live metrics.
- 42. If you are running a Titanium/Platinum combination deposition, the following steps (Steps 43-49) should be repeated for Platinum after your initial Titanium deposition.
- 43. Now that your graphs are in place and your crucible platten is in the correct pocket, we are ready to begin our deposition. Click on "Run Recipe" from the right-middle side of the main interface.
  - a. Note: This process will differ for multiple metals, so I will write the process for one single metal deposition and I will add Notes whenever the processing would differ for Titanium.
- 44. From the pop-up window that should appear, select "Deposition Platinum".
  - a. Note: This would be "Deposition Titanium" if you are running Titanium first.
- 45. Another pop-up window will appear on the upper right-hand side of the interface. This will be where you enter your pocket number, target metal thickness, and target deposition rate. Enter the following values (current process values, subject to change):
  - a. Platinum
    - i. Pocket: 3
    - ii. Target Thickness: 1.0 (in kÅ)
    - iii. Target Deposition Rate: 1.0 (in Å/s)
  - b. Titanium
    - i. Pocket: 2
    - ii. Target Thickness: 0.05 (in kÅ)
    - iii. Target Deposition Rate: 1.0 (in Å/s)
- 46. Click "Continue Process" in the lower left-hand side of the pop-up window that you just entered your values into.
- 47. The Lesker #4 system will automate the deposition process for you, so all you need to do while it runs is monitor the data and watch the crucible and wafers for any potentially mishaps. The following are some examples of what I am looking for during the deposition:
  - a. Soak 1 (initial setpoint of E-Beam energy to melt part of the metal in the crucible to prepare for the deposition)
    - i. Look to make sure the beam is centered in a location on your crucible that will not be contacting bare crucible.
  - b. Soak 2 (final setpoint of E-Beam energy to get the majority of the metal in the crucible into the liquid stage to prepare for deposition)
    - i. Sometimes this is set very low, so you may need to adjust the "Rate" value you entered during the automated run. (Do this by entering a new value into the "Target Rate" text box in the bottom-middle section of the interface.
    - ii. Also make sure that the E-Beam's coverage area will not be touching bare crucible.
  - c. Ramp to Target Deposition Rate (ramps up/down the E-Beam energy setpoint to get the deposition rate to +/- 15% of the Target Deposition Rate and holds for 20 seconds)
    - i. May need to adjust the Target Deposition Rate if this will reach the end of the timer before it will reach the Target Rate. (Move it down to 0.75 or even 0.6 if needed, then ramp it up to the correct rate as it reaches the new Target Rates).
    - ii. Keep checking the melted area in the crucible to make sure it isn't going over the crucible and isn't going to contact bare crucible.

- d. Deposition (opens the Substrate Shutter and begins the deposition on your wafers)
  - i. Watch your wafers to make sure they are not falling off the wafer stage and ensure they are getting metal deposited on them.
  - ii. Make sure the wafer stage is rotating to make sure you are getting an even deposition.
  - iii. Check to be sure that the crucible melt isn't contacting bare crucible and isn't spilling over.
  - iv. Watch the rate and thickness readings on the graph to be sure they are following the correct pattern.
  - v. Watch the heating of the chamber (if you started this graph) to check the substrate heating that is happening due to the deposition.
- e. Ramp Down (turns the E-Beam energy down until it is turned off)
  - i. Make sure the melted part of the crucible is cooling down (the crucible platten is water cooled, so it should be cooling down fairly rapidly).
  - ii. Make sure the Substrate Shutter has closed.
  - iii. Ensure that the Rate you were following has dropped to 0 and isn't fluctuating too much.
- 48. Once your deposition process has completed, the process pop-up window (lower left-hand side of the main interface) should display "Recipe Complete". Close the recipe pop-up window.
- 49. Regardless of the metal you just deposited, wait at least 5-10 minutes for the chamber to cool down further before continuing with your processing.
  - a. Note: ~5 minutes is adequate for Titanium while ~10 minutes is adequate for Platinum.
- 50. If you just deposited Titanium and now require a Platinum deposition, repeat Steps 43-49, but for a Platinum deposition instead of Titanium.
- 51. Once all deposition steps are completed and the appropriate cool down time has been spent, we will now need to Vent the system to atmosphere and remove our samples and materials from the system. Start this process by selecting "Vent" from the right-middle side of the main interface.
- 52. While the system is venting, you can select "Stop Graph" on your open graph(s) and close the window(s) they were displayed in.
- 53. When the recipe pop-up window displays "Recipe Complete", that means the system is vented to atmosphere. You may open the chamber door now.
- 54. We will now need to remove our crucible(s) from the platten. If you only used Platinum, you will only have one crucible to remove. If you need to remove Platinum and Titanium, the following steps should guide you through that process:
  - a. Remove the Platinum crucible from the pocket you can see (Pocket 3) and place it in the plastic crucible storage box we use.
  - b. Open the door the interface is on to reveal the pocket interface.
  - c. On the interface behind the door, select "Auto/Manual" using the left joystick (pushing it to the right to select).
  - d. Select "Manual" with the right joystick (highlighting it).
  - e. Push the right joystick to the right to log in.
  - f. Enter the code "2-0-3-1" to enter Manual mode.
  - g. Using the left joystick, select "Set Pocket" (pushing it to the right to select).
  - h. Move the right joystick up to highlight "Pocket 2".
  - i. Hold the right joystick to the right to move the crucible platten to Pocket 2.

- i. Note: Continue holding the joystick until the crucible platten has completed its movement and has settled on Pocket 2.
- j. Remove the Titanium crucible from the pocket you can see (Pocket 2) and place it in the plastic crucible storage box we use.
- k. Move the right joystick down to highlight "Pocket 3".
- I. Hold the right joystick to the right to move the crucible platten to Pocket 3.
  - i. Note: Continue holding the joystick until the crucible platten has completed its movement and has settled on Pocket 3.
- m. Select "Auto/Manual" with the left joystick (pushing it to the right to select).
- n. Select "Automatic" with the right joystick (highlighting it).
- o. Push the right joystick to the left to log out.
- p. Close the door so that the main interface can be seen again.
- 55. On the main interface, click on "Run Recipe".
- 56. In the pop-up window that appears, select "Sample Unload Manual Operation Only" and click on "Start Recipe".
  - a. Note: This process will home the wafer stage and then drop the shield so that the stage can be removed. DO NOT keep your hands inside the chamber while this process is running to ensure that you do not injure your hands from the moving metal shield.
- 57. Once the shield has dropped down so that you can see the wafer stage, you can remove the wafer stage in the same manner that you did previously. (Lift up and off the pins, move out away from the pins, move down into the chamber, and move out of the chamber)
  - a. Note: Be careful when you are removing the wafer stage so that you do not knock into the crystal sensor. Also, you have wafers with fresh metal deposition on them, so make sure that your hands are not on the wafers when you are removing the wafer stage AND make sure that the wafers do not scrape against anything in the chamber or on the table you will set the wafer stage on.
- 58. Place the wafer stage on the nearby table such that you can see the wafers on top and the pins are contacting the table surface.
- 59. Remove the wafers from the wafer stage using a razor blade to remove the Kapton tape from the edges of the wafers.
  - a. Note: Be very careful with the blade to not touch the wafers. I will typically peel up about half of the tape that is contacting the wafer stage and peel the rest by hand. Also be careful of where the blade is at all times in order to minimize your risk of getting injured from the blade.
- 60. Place the wafers into the Wafer Box they came from.
- 61. Grab the unloaded wafer stage and move it back into the chamber. (Remember to return it to its correct orientation before attempting to load it back into the chamber).
- 62. Slowly and carefully put the stage into the chamber, ensuring that you are avoiding the crystal sensor, and raise it up so that it can rest on the pins it started on.
- 63. Ensure the stage is on the pins and secure before letting go of the stage.
- 64. On the main interface (front of door), select the "Resume" button on the pop-up recipe window to return the shield to its normal protective location.
- 65. Once the shield is back into position, the recipe pop-up window should display "Recipe Complete". Click the "Close" button to close this pop-up window.
- 66. Click the "Vacuum" tab from the top of the interface.

- 67. Close the chamber door and hold it closed with your hands with weight into it (either body weight or muscle).
  - a. Note: Keep this closed until you see the "Vac Lock Switch" activate and highlight in green.
- 68. Click the "Pump" button on the right-hand side of the interface.
- 69. Once you see the "Vac Lock Switch" highlighted in green, you can release the door.
  - a. Note: There should also be an audible "click" when this happens, but be sure that it was your system and not another in the lab if you are basing your procedure off of the audible sound.
- 70. The system will now pump down until it reaches 5e-5 Torr (50 µTorr). You do not have to stand next to the system as it pumps down to its minimum pressure since you have completed your run.
- 71. Log out of the Lesker software.
  - a. Note: The Pump down recipe will still continue to run since it was started when you were logged in.
- 72. Select "Run Complete" on the tablets so that other users know that you have completed your processing.
- 73. Your wafers will now need to be inspected and tested before you leave the cleanroom.

#### 5.3.2 Inspection

- 1. There are three methods of inspection we use to measure the quality of metal layer(s) we get after deposition.
  - a. The first is a visual inspection using our eyes.
  - b. The second is a visual inspection using a microscope.
  - c. The third is a metric inspection using a measurement tool to get the resistance of a known distance across the metal.
- 2. As a first inspection, look at the wafer and ensure you have gotten the correct pattern to what you were setting out to get from the photoresist/mask combination. Also inspect for defects like color patterns, burnt sections, peeling, or cracked metal layers. Any errors found cannot be reworked without etching (which may sacrifice biocompatibility), so let's hope they aren't catastrophic errors and that most of the devices will not be affected.
- 3. The second inspection involves using a microscope to inspect any potential defects found as well as to check for the presence of any micro cracks in the metal layer(s). Larger sections of metal depositions are more prone to micro cracks than smaller and more intricate sections, so find those larger sections to see if there are any cracks that are immediately apparent. You can also play with the focus, going in and out from the normal focus to see if there are barely visible lines, which would indicate that micro cracks could appear during sonication.
- 4. The third inspection is where we measure the resistance of the metal layer(s) across a known distance using the Four-Point Probe station inside the ASU NanoFab cleanroom. This system consists of a multimeter unit and a probe unit.
  - a. Note: There are instructions for operation on top of the multimeter unit just in case you need them in the cleanroom.
- 5. Turn on the Keithley multimeter (bottom far left)
- 6. Press the  $\Omega$ 4 button (middle-right underneath the display)
- 7. Press the Rate button (bottom middle-right) to go from "Med" to "Slow"

- 8. Check the interface to make sure it reads "Auto" (right side of display) and press the "Auto" button (right side of buttons, between the arrows) if this does not display "Auto"
- 9. Wait for 20+ minutes for the Keithley to warm up before using.
  - a. Note: I typically turn the Keithley on as soon as the Platinum begins depositing, NOT the Titanium.
- 10. The following steps (Steps 11-33) should be repeated for each wafer in the batch that just completed metal deposition.
- 11. Remove the next wafer to test from the Wafer Box and place it on the glass plate underneath the probe unit of the system.
- 12. Move the wafer (and glass plate) around until you have found your first testing site.
  - a. Note: I use the large testing pads from the "Metrigraphics Electrodes" mask design exclusively, so my first testing site is bottom pad, top middle.
- 13. Slowly push the handle of the probe unit to the left (clockwise) until it bottoms out (DO NOT push this rapidly). This will make contact with the probe to the metal.
- 14. Wait for the reading on the Keithley multimeter to settle and record the value.
  - a. Note: This should be around 1  $\Omega$  for a 1,000 Å Platinum deposition, regardless of Titanium presence.
- 15. Slowly push the handle of the probe unit to the right (counterclockwise) until it bottoms out (DO NOT push this rapidly). This will release contact from the probe to the metal.
  - a. Note: ALWAYS do this before trying to move the wafer in order to prevent scratching the wafer surface.
- 16. Move the wafer (and glass plate) around until you have found your second testing site.
- a. Note. My second testing site is bottom pad, top left.
- 17. Repeat Steps 13-15 for this measurement point.
- 18. Move the wafer (and glass plate) around until you have found your third testing site.
  - a. Note: My third testing site is bottom pad, top right.
- 19. Repeat Steps 13-15 for this measurement point.
- 20. Move the wafer (and glass plate) around until you have found your fourth testing site.
  - a. Note: My fourth testing site is bottom pad, bottom middle.
- 21. Repeat Steps 13-15 for this measurement point.
- 22. Move the wafer (and glass plate) around until you have found your fifth testing site.
  - a. Note: My fifth testing site is top pad, bottom middle.
- 23. Repeat Steps 13-15 for this measurement point.
- 24. Move the wafer (and glass plate) around until you have found your sixth testing site.
  - a. Note: My sixth testing site is top pad, bottom left.
- 25. Repeat Steps 13-15 for this measurement point.
- 26. Move the wafer (and glass plate) around until you have found your seventh testing site.
  - a. Note: My seventh testing site is top pad, bottom right.
- 27. Repeat Steps 13-15 for this measurement point.
- 28. Move the wafer (and glass plate) around until you have found your eighth testing site.
  - a. Note: My eighth testing site is top pad, top middle.
- 29. Repeat Steps 13-15 for this measurement point.
- 30. (Optional) If you would like to measure a ninth time (to line up with your measurement points from the Reflectometer), move to that location.

- a. Note: My ninth testing site is top pad, far top and left of middle (to the immediate left of the "Top" label).
- 31. (Optional unless Step 30 is completed) Repeat Steps 13-15 for this final measurement point.
- 32. Once all of the measurements have been taken for this wafer, remove it from the glass plate.
  - a. Note: BE CAREFUL of the probe tips to make sure that they do not scratch the surface of the wafer when removing from the testing apparatus.
- 33. Place the wafer that has just completed testing back into the Wafer Box it came from.
- 34. Repeat Steps 11-33 for each wafer in the batch that has just completed the metal deposition stage of the processing.
- 35. Once all wafers in the batch are complete, turn the Keithley multimeter unit Off (button is far left bottom of the unit).

# 6 Lift-Off for Conductive Metal Patterning

# 6.1 Equipment Needed

- Pyrex dish (>4" diameter) (Qty: 4)
- Sonicator OR Nitrogen bubblers
- Hot Plate or Oven (80 C & 23C Optional)
- Nitrogen air gun (for wafer drying)
- Microscope (for inspection)
- Camera (for microscopic imaging)
- Fume Hood
- Timer

## 6.2 Materials Needed

- Wafer Tweezers or Wand (4")
- Wafer (3" or 4") Silicon w/ Polyimide, Photoresist, & Conductive Metal
- Cleanroom Notebook
- Cleanroom Wipes
- Marker for wafer labeling
- AZ 400T Stripper (~200-400 mL per batch)
- Isopropyl Alcohol (~100-200 mL per batch)
- DI Water (~100-200 mL per batch)
- Waste bottles for all chemicals (~3)

# 6.3 Lift-Off Process

### 6.3.1 Photoresist Stripper & Cleaning Baths

- It is best to do the Lift-Off process all in one day so that the photoresist is fresh and will come off easily. Any time that I waited to remove the resist resulted in either a failed lift-off or a poor quality of metal layer since sonication lasted longer than it should have. Try to do Sections 3-6 in the same day (Photoresist Deposition for Metal Patterning → Substrate Surface Preparations → Conductive Metal Deposition → Lift-Off for Conductive Metal Patterning).
  - Note: If you are trying to get the cleanest wafer fabrication possible, I would also begin the Insulation Deposition process (Section 7) as soon as the Lift-Off is complete. However, this requires pre-planning in order to make sure there is a student worker in the ASU NanoFab with you for Parylene deposition (and many hours in a cleanroom in a single day).
- At ASU, we can do the Lift-Off Process in any cleanroom, but I typically use the ERC 444 cleanroom as it has many more dishes available to us and also does not cost us to be inside. I will use the "Piranha" fume hood as it has the most surface area available for our bath-style of lift-off processing.
  - a. Note: Be aware that the transfer between the ASU NanoFab and ERC 444 can potentially introduce humidity effects to the wafers. We have not had to worry about this in the past, but it is something to keep in mind as we continue processing advancement just to keep the cleanest process possible and introduce the fewest sources of potential error.
- 3. Once inside ERC 444, turn on the IsoTherm Oven (between the Mask Aligner and the Evaporator) and ensure that it is set to 100°C.
  - a. Note: This will be used for a Dehydration bake after the wafers have completed the lift-off process, so I do this first just to ensure that it is at temperature when we are ready to use it.
- 4. Open the sash on the "Piranha" fume hood so that you will have enough room to work underneath.
  - a. Note: NEVER place this in the "Red" section on the colored label on the sides of the sash.
- 5. Place your Wafer Box and wafer tweezers (or wand) into the "Piranha" hood so that you have them readily available.
- 6. Have your cleanroom notebook and/or process sheet handy to write down the times it took each wafer for the lift-off process as well as any comments you observed in the process.
- 7. Grab the Marker from the toolbox and place it near your cleanroom notebook/process sheet as you will likely need to re-write the labels on the back of the wafers after the Lift-Off process.
- 8. Have a Timer (or watch) handy near/inside the "Piranha" hood to record the times of some of the baths.
  - a. Note: The sonicator will time the actual stripping process for us, but it is always nice to have something to watch when doing the IPA and DI Water baths.
- 9. Place 5 cleanroom wipes in the front of the "Piranha" hood and place 1 cleanroom wipe in the back of the "Piranha" hood.
  - a. Note: The 5 cleanroom wipes will be for the baths and the 1 cleanroom wipe at the back will be for photographs before and after processing.
- 10. Place the Wafer Box on the side edge of the cleanroom wipe in the back so that it doesn't blow away during our Nitrogen drying step.

- 11. Grab one of the Nitrogen air guns from on top of the work benches behind you (center of the room) and move one to the left side of the "Piranha" fume hood.
  - a. Note: I will usually stick this air gun between the "Vacuum" and "Air" valves on the left side of the fume hood so that it stays put without putting too much strain on the line the air gun is attached to.
- 12. Grab 4 dishes from the drying rack above the sink and place one on each cleanroom wipe, leaving one wipe dedicated to the drying stage of the process.
  - a. Note: All of these dishes need to be at least 4" in diameter, preferably around 5" in diameter. Currently, there are only 2 dishes of this size, so using dishes that are 6" or 7" will work.
- 13. The 2 dishes closest to the sink and sonicator are what I use for the AZ 400T Photoresist Stripper solutions ("Dirty" is closest and "Clean" is further) as they will be taken from the hood and placed in the sonicator for processing.
- 14. The next dish in the line (to the left of the "Clean" 400T dish) would be for the IPA rinse.
- 15. The final dish in the line (to the left of the IPA rinse dish) would be for the DI Water rinse.
- 16. The empty cleanroom wipe to the left of the DI Water rinse dish would then be for the Nitrogen air gun drying.
- 17. Grab some small empty dishes from the sink area and place them on the top left and bottom left side of the empty cleanroom wipe.
  - a. Note: I do this to prevent the empty cleanroom wipe from moving during the air drying process.
- 18. Using the Marker or a pen (doesn't matter which is used), mark the cleanroom wipes corresponding to the chemicals you will be adding to their respective dishes. Add your name and phone number just in case you have to leave the cleanroom.
  - a. The order of cleanroom wipe labels going from left to right will read as the following:
    i. [N2 Dry] → [DI Water Rinse] → [IPA Rinse] → ["Clean" 400T] → ["Dirty" 400T]
  - b. Note: I will label the back of the cleanroom wipes (towards the back of the fume hood and the Wafer Box) and not the front so that the label will remain just in case the front gets any water from the sonicator on it (which will happen).
- 19. Check the water level in the sonicator to make sure there is at least a little water inside the bath. Add more water (using the plastic pitcher next to the sonicator) if there is hardly any inside the bath.
  - a. Note: You want at least a couple inches of water to make sure the dishes will not touch the metal bottom of the sonicator. Overfilling the water will mean that your dishes will likely need to use more 400T (to completely cover the wafers during processing) due to their buoyancy inside the water of the bath.
- 20. Turn the sonicator on.
- 21. The sonicator should start with the "Bath Temp" mode highlighted. Press the "Reset" button to ensure you are not heating the solution while in the sonicator.
- 22. Press the "Mode" button on the sonicator to move to the "Set Temp" mode. Press the "Reset" button to ensure you are not heating the solution while in the sonicator.
  - a. Note: If the process is not working as intended (aka if the photoresist is older than 24 hours), heat will be REQUIRED for the processing to take place. If it is needed, use the temperature of "69" (°C) to heat the bath.

- i. Sidebar: Maximum temperature of the sonicator bath is 69°C and the recommended from the AZ nLOF 2020 datasheet is 80°C. The temperature was not picked due to the sexual innuendo that is typically referenced with the number.
- b. ALTERNATIVE: Still need to try a heat-only Lift-Off process with no sonication and only manual agitation. For this process, use a "Set Temp" of "69" (°C) and a "Set Time" of "00" (minutes). While on the "Set Temp" mode is when you would press the "Start" button to begin heating, which should turn the "Heat On" light on. This process will require separate timing and manual agitation with your wafer tweezers, so be sure that these are ready near the sonicator before you place the dish of 400T (with a wafer in the dish) into the sonicator bath. Be aware that this process will likely take at least 10 minutes with manual agitation in the "Dirty" bath and another 5-10 minutes with manual agitation in the "Clean" bath, but the time could be even longer potentially. (Your hands may cramp and get sore from manually agitating for that long, so just do a 1-2 minute cycle of agitate/swirl/... etc.)
- 23. Press the "Mode" button on the sonicator again to move to the "Set Time" mode. Press the "Set" button until you reach "05" (minutes).
- 24. Ensure that the "Heat On" light in the Mode list is NOT on.
  - a. Note: If you require heat to remove the photoresist, this light SHOULD be on.
- 25. Leave the sonicator on the "Set Time" mode as this is the mode you need to be in when you press the "Start" button to begin sonication.
  - a. Note: This would be the "Set Temp" mode if you are heating the bath to begin heating.
- 26. Grab the dish you intend to add DI Water into and take it to the sink to add DI Water from the faucet on the left (not the normal Hot/Cold faucet). Place this dish back on its respective cleanroom wipe once you have added about 0.5-1 inch of DI Water to the dish.
  - a. Note: This water level needs to be high enough that you can manually agitate the wafers and keep them completely submerged, but you also don't want to use too much.
- 27. Grab the bottle of Isopropyl Alcohol from the cabinet underneath and to the left of the "Piranha" fume hood and place it inside the "Piranha" fume hood.
- 28. Fill the dish you are intending for your IPA Rinse with the Isopropyl Alcohol.
  - a. Note: Fill this dish to the same level that you have your DI Water dish's filled to and be careful to not spill any IPA in other dishes or on the working surface of the fume hood.
- 29. Place the bottle of Isopropyl Alcohol back in its storage location once you have filled your dish to the desired level.
- 30. The next step would be to add the AZ 400T Stripper solution to both the "Clean" and "Dirty" dishes. Read the notes below for handling precautions. If desired, don the proper PPE necessary for handling at this point.
  - a. BE AWARE that the SDS calls to take precautions when handling as it is a Base. (Recommended to have an Apron, Base (rubber) gloves, and a Face Shield) However, we do not currently have rubber gloves that would suffice to this duty (very tattered and hole-filled rubber gloves), so simply being careful when moving the bottle and when pouring is all I have done previously.
  - b. Note: If we would like to start saving some of the 400T solution for future processes (if we are doing a lot of batches back-to-back-to-back, this is recommended), we can start collecting "Dirty" 400T solution and re-using it for future batches, as long as we pour fresh

"Clean" 400T each time, the process will still function as intended. If we do decide to keep some 400T to be re-used for the "Dirty" bath, we NEED to keep it separate from any other chemical in the cleanroom, which means that we need to label the hazardous waste bottle in such a way that no one but us can use it. (i.e. "JMBC USE ONLY - DO NOT USE FOR 400T WASTE")

- 31. Grab the 400T bottle from the bottom cabinet to the left of the open chemical bench (far right from the sink, but the left door of that section) and place it in the "Piranha" fume hood.
- 32. Carefully pour the 400T solution into the "Clean" and "Dirty" dishes, ensuring that you have at least 0.5" of solution in the dish. (Closer to 1" will guarantee that you will cover everything when in the sonicator)
  - a. Note: The "Dirty" dish can be filled with "old" solution if needed, but the "Clean" dish must be fresh.
- 33. Place the 400T bottle back in its storage location once you have filled both dishes to the desired level.
  - a. Note: I typically only wear the PPE during chemical handling and pouring, so I remove the apron, gloves, and face shield now. However, be aware that the transfer of 400T dishes from the fume hood to the sonicator technically counts as chemical handling. In other words, if you would prefer to be as safe as possible, you would want to keep the PPE attire on until you are done with the 400T completely (~2 hours of total processing or 6+ times where you don the PPE and then remove it shortly thereafter)
- 34. The following Steps (35-80) should be repeated for each wafer in the batch that require Lift-Off processing.
- 35. Grab the next wafer needed to be processed from the Wafer Box with your wafer tweezers.
- 36. Place this wafer on the empty cleanroom wipe in the back of the "Piranha" fume hood (next to the Wafer Box) so that you can take a picture of the wafer. Grab an image of the wafer and be sure to angle your phone/hand in such a way that you don't see much of anything in the fume hood reflected back on the surface of the wafer.
- 37. Grab the wafer off the cleanroom wipe and flip it over so that you can see the back of the wafer and the label you wrote on it during the Substrate Preparations Section of the process.
- 38. Grab an image of the back of the wafer so that you can know which wafer you just took a picture of when looking back at your photos.
- 39. Carefully dip the wafer into the "Dirty" 400T bath and push the wafer down until it is fully submerged.
  - a. Note: Be gentle with the wafer, but do not let it stick above the surface of the solution.
- 40. Move the "Dirty" 400T bath from the "Piranha" fume hood to the sonicator bath.
  - a. Note: This is what would require PPE if you are wanting to be extra precautious, but do not try to don it after you have placed the wafer in the solution if you do not already have it on.
- 41. Press the "Start" button on the sonicator to begin the ultrasonic vibrations.
  - a. Note: If you are doing a heat-only Lift-Off process, you will not have to Start the sonicator as it should have already been heated up. Instead, Start the Timer to monitor how long the wafer is in the heated solution and begin your manual agitation process.
- 42. Continually watch the wafer in the sonicator to see how far along it is in the process.
  - a. Note: DO NOT stand above the sonicator and watch the wafer for the entire duration as the fumes from the 400T solution will likely make you dizzy after a few minutes of inhaling

them. Instead, just check on the wafer every minute or so, or watch the wafer from an angle that is at the level of the sonicator's walls.

- 43. The photoresist (and metal it is attached to) will slowly "dissolve" away and you will be left with the pattern that you intended with the mask you designed (or are using). Once the process has "dissolved" approximately 80-90% of the photoresist it needs to, allow the process to go on for 1 minute more before pressing the "Stop" button on the sonicator (if necessary).
  - a. Note: Sometimes this process can take longer than 5 minutes, so pressing the "Start" button on the sonicator to restart the 5 minute process is required to continue the processing. If this is the case, be sure that you record that 5 minutes have already passed in your cleanroom notebook/process sheet so that you can keep track of the processing time required for each wafer.
- 44. Remember the time that it took for the wafer to process in the "Dirty" bath as you will need to write this down after you have started the next sonication process.
- 45. Move the "Dirty" 400T dish from the sonicator to the "Piranha" fume hood and place it back on its cleanroom wipe.
- 46. Manually agitate the wafer (same process as from Developing) for approximately 20-30 seconds in order to move the "dissolved" photoresist off the surface of the wafer and into the bath solution.
- 47. Remove the wafer from the "Dirty" 400T solution and hold it vertically above the solution to allow it to drip into it.
- 48. Once the drips have slowed to at least once per second (should take approximately 1-5 seconds), transfer the wafer to the "Clean" 400T dish and carefully dip the wafer into the solution and push the wafer down until it is fully submerged.
  - a. Note: Be gentle with the wafer, but do not let it stick above the surface of the solution.
- 49. Move the "Clean" 400T bath from the "Piranha" fume hood to the sonicator bath.
  - a. Note: This is what would require PPE if you are wanting to be extra precautious, but do not try to don it after you have placed the wafer in the solution if you do not already have it on.
- 50. Press the "Start" button on the sonicator to begin ultrasonic vibrations.
  - a. Note: If you are doing a heat-only Lift-Off process, you will not have to Start the sonicator as it should have already been heated up. Instead, Start the Timer to monitor how long the wafer is in the heated solution and begin your manual agitation process.
- 51. The "Clean" 400T solution process is required just to remove the last remaining photoresist from the wafer and clean up the surface from any leftover artifacts from the Lift-Off process, so a time of 3-5 minutes is all that is needed here.
  - a. Note: The time the "Clean" bath will take is also going to depend on the length of time that the "Dirty" bath took. If the "Dirty" bath only took 5 minutes, a time of 3 minutes is sufficient here. If the "Dirty" bath took 20 minutes, then a time of 5 minutes would be minimum and more would ensure the entire amount of photoresist has been "dissolved" away.
- 52. Once the time selected for the "Clean" 400T bath is complete, press the "Stop" button on the sonicator (if necessary).
- 53. Move the "Clean" 400T dish from the sonicator to the "Piranha" fume hood and place it back on its cleanroom wipe.
- 54. Manually agitate the wafer (same process as from Developing) for approximately 20-30 seconds in order to move the "dissolved" photoresist off the surface of the wafer and into the bath solution.

- 55. Remove the wafer from the "Clean" 400T solution and hold it vertically above the solution to allow it to drip into it.
- 56. Once the drips have slowed to at least once per second (should take approximately 1-5 seconds), transfer the wafer to the IPA Rinse dish and carefully dip the wafer into the solution and push the wafer down until it is fully submerged.
- 57. Manually agitate the wafer (same process as from Developing) for approximately 20-30 seconds.
- 58. Remove the wafer from the IPA Rinse solution and hold it vertically above the solution to allow it to drip into it.
- 59. Once the drips have slowed to at least once per second (should take approximately 1-5 seconds), transfer the wafer to the DI Water Rinse dish and carefully dip the wafer into the solution and push the wafer down until it is fully submerged.
- 60. Manually agitate the wafer (same process as from Developing) for approximately 20-30 seconds.
- 61. Remove the wafer from the DI Water Rinse solution and hold it vertically above the solution to allow it to drip into it.
- 62. Once the drips have slowed to at least once per second (should take approximately 1-5 seconds), transfer the wafer to the empty "N2 Dry" cleanroom wipe and lay it down flat such that the top of the wafer is facing up towards you.
- 63. Place the wafer tweezers to the side, but near the cleanroom wipe the wafer is on.
- 64. Grab the Nitrogen air gun from its location on the left of the "Piranha" fume hood.
- 65. Carefully spray the center of the wafer with the Nitrogen air gun and slowly rotate your wrist (and only your wrist) around in a circular pattern to blow all of the liquid off the wafer.
  - a. Note: The air guns we have in the ERC 444 cleanroom can be extra powerful, so be mindful of how much pressure you are applying behind the trigger. Full blast is not recommended for drying out the wafers since this could potentially be way too much power.
  - b. Also: When rotating your wrist, do it in such a way that rotates the air gun's nozzle around in a circle without moving it in the vertical direction. In other words, be mindful of where the nozzle is at all times so that you do not touch it onto the wafer and you do not move it too far away from the wafer (where it could cause the wafer to levitate - and no, that isn't as cool as it sounds)
- 66. Push all of the liquid off the edge of the wafer and onto the cleanroom wipe, but be careful when doing this as the wafer will want to lift off the ground as you get closer to the edge of the wafer.
- 67. Grab your wafer tweezers in your non-dominant hand (aka the hand not holding the air gun).
- 68. Holding the tweezers outside of the "Piranha" fume hood and directed away from your wafer, dry the tweezers off using the Nitrogen air gun. Rotate your tweezers to dry the entire surface.
  - a. Note: DO NOT perform this task above or next to your wafer as you will get drips from your tweezers onto your wafer, causing you to have to re-dry your wafer. I will turn 90° to the left (or right) and do this to the side of the "Piranha" fume hood.
- 69. Using the dry tweezers, grab the wafer from the major flat, rotate your wrist such that the wafer is vertical and the back/bottom side of the wafer is facing your dominant side, and place the "top" of the wafer (furthest part of the wafer from the major flat) on the cleanroom wipe (edge of the wafer only).
  - a. Note: I am right-handed, so the air gun will be in my right hand when doing these steps. I will place the wafer with the patterned side facing towards the Piranha waste secondary

container and the bottom side facing towards the DI Water Rinse dish, within the "Piranha" fume hood. Hold this in the opposite direction if you are left-handed.

- 70. Carefully spray the center of the bottom of the wafer with the Nitrogen air gun and slowly rotate your wrist (and only your wrist) around in a circular pattern to blow all of the liquid off the wafer.
  - a. CAUTION: Make sure you are firmly holding the wafer with your wafer tweezers the entire time you are drying the bottom side of the wafer. I have broken a few wafers because I was not clamped on the wafer with the tweezers.
  - b. Note: The air guns we have in the ERC 444 cleanroom can be extra powerful, so be mindful of how much pressure you are applying behind the trigger. Full blast is not recommended for drying out the wafers since this could potentially be way too much power.
  - c. Also: When rotating your wrist, you will be doing it in the vertical axis instead of the horizontal axis this time, so be aware of where the nozzle of the air gun is in relation to the bottom side of the wafer.
- 71. Once the bottom of the wafer is completely dry, pick the wafer up off the cleanroom wipe and hold it in the same position it was just in on the cleanroom wipe (vertical with the major flat behind held at the top and the "top" of the wafer pointed toward the fume hood surface).
- 72. Gently air dry the sides of the wafer by activating the air gun and moving the air gun in a side-to-side manner across the edge of the wafer.
- 73. While you are moving the air gun side-to-side, rotate the wafer (using your entire arm this time) so that you can dry off all of the edges of the wafer.
  - a. Note: This will take some practice to get the hang of since it requires simultaneous movements in different directions and orientations. If needed, ask Carrie Sinclair (ASU NanoFab staff) to help demonstrate this motion for you as she is the expert that I learned it from.
- 74. Once all of the edges of the wafer have been dried, rotate the wafer such that the patterned side is facing towards you.
- 75. Gently air dry the patterned face, pushing any liquid off the front surface of the wafer that may have appeared from the edge drying process.
  - a. CAUTION: Make sure you maintain a firm grasp on the wafer using the wafer tweezers during this step as this is another stage in which I have broken a few wafers during. Too firm of a grasp will cause the wafer to slip easier though, so be mindful of how firm you are holding it.
- 76. When the front (patterned) face of the wafer is completely dry again, DO NOT place it back on the cleanroom wipe as it will be wet from the drying process. Place this wafer on the empty cleanroom wipe in the back of the "Piranha" fume hood (next to the Wafer Box) so that you can take a picture of the completed wafer. Grab an image of the wafer and be sure to angle your phone/hand in such a way that you don't see much of anything in the fume hood reflected back on the surface of the wafer.
  - a. Note: I like to inspect all of my wafers under the microscope (Section 6.3.2) as soon as they are done with the Lift-Off process so that I know if the process was successful or if any stage of the process needs to be adjusted. If you see debris on the wafer still, increase the time you have the proceeding wafers in the "Clean" 400T bath under sonication. If the Platinum has cracked or appears to be tearing off, decrease the time you have the proceeding wafers in the "Dirty" 400T bath under sonication. If it looks like

there are "dry spots" on your wafer, increase the time you have proceeding wafers in the IPA Rinse and DI Water Rinse baths and ensure the manual agitation efforts are not bringing the wafer up above the surface of the solution. You can take a picture of the wafer after it has been inspected under the microscope.

- 77. Grab the wafer off the cleanroom wipe and flip it over so that you can see the back of the wafer.
- 78. Using the Marker, re-write the label that used to be on the back of the wafer. Place the Marker back where you grabbed it from once you are done writing.
  - a. Note: This should have disappeared from the bath process we just performed.
- 79. Grab an image of the back of the wafer so that you can know which wafer you just took a picture of when looking back at your photos.
- 80. Place the wafer back in the Wafer Box it came from.
- 81. Repeat Steps 34-80 for each wafer in the batch that require Lift-Off processing.
- 82. Once all wafers in the batch are complete through the bath processing, it is time for the dehydration bake in the IsoTherm oven. Move the Wafer Box from the "Piranha" fume hood to the top surface of the oven.
- 83. Open the door to the oven.
- 84. Place all of the wafers that just completed bath processing onto a shelf in the oven.
  - Note: You will need to remove the wafers shortly, so place them in such a way that you don't have to maneuver around other wafers to get the the ones you are trying to remove.
     In other words, limit your placement to 2-3 wafers per shelf and place them in the front of the shelf, closest to the door for easy removal.
- 85. Start your timer for 20-30 minutes.
  - a. Note: The time is not really important here as long as it is for longer than 60 seconds. I usually shoot for 30 minutes just to ensure everything has evaporated from the surface of the wafer as well as anything that may have gotten absorbed during the processing.
- 86. Close the door to the oven.
  - a. Note: You can close the door before starting the timer if you would like, as an exactly precise oven time is not as necessary at this stage of the process.
- 87. While the timer is running, move the cleanroom wipe you used for pictures in the "Piranha" fume hood to the top of the oven for use in cooling the wafers down.
  - a. Note: If the top surface is too cluttered, you can place this cleanroom wipe on the table surface in the center of the room behind you (next to the green working mat).
- 88. When the dehydration bake timer expires, Open the door to the oven.
  - a. Note: I would continue on and complete Steps 94-135 before returning to remove the wafers from the oven so that you can manage your time appropriately without waiting around.
- 89. Remove all of the wafers you placed in the oven for dehydration and place them on the cleanroom wipe you just placed for cool down.
- 90. Close the door to the oven after all wafers have been removed.
- 91. Start a timer for at least 1 minute to allow the wafers time to cool down to room temperature again.
  - a. Note: The time is very arbitrary here, so as long as it is longer than 60 seconds, it does not need to be precise.
- 92. Turn the IsoTherm oven Off (if it was Off when you came in, otherwise you can leave it On if it was already On when you entered the cleanroom)

- 93. When the timer expires (if you actually set a timer for this, otherwise just when you feel the wafers have cooled down enough), move the wafers from the cleanroom wipe to the Wafer Box they came from.
  - a. Note: Skip to Step 136 if you have already completed Steps 94-135 during the dehydration bake waiting time.
- 94. Grab the Waste bottle for 400T Stripper from the secondary containers below the "Piranha" fume hood and place it inside the "Piranha" fume hood, behind the line of dishes.
  - a. BE AWARE that the SDS calls to take precautions when handling as it is a Base. (Recommended to have an Apron, Base (rubber) gloves, and a Face Shield) However, we do not currently have rubber gloves that would suffice to this duty (very tattered and hole-filled rubber gloves), so simply being careful when moving the bottle and when pouring is all I have done previously.
- 95. Open the 400T Waste bottle and place the cap on the counter of the fume hood.
- 96. Grab the waste funnel from the secondary containers below the "Piranha" fume hood and place it inside the opened 400T Waste bottle.
- 97. Carefully grab the "Dirty" 400T dish and empty its contents into the 400T Waste bottle, through the funnel.
  - a. Note: Be careful to not pour this too quickly as this could cause it to spill.
- 98. Using the "D" labeled knob on the front right of the "Piranha" fume hood, dispense about 100 mL of DI Water into the dish that contained the "Dirty" 400T solution.
  - a. The "Dirty" dish will likely have metal deposits coating the bottom of the dish, so be sure the rotate the dish around when dispensing the DI Water so that the stream of water can move the deposits off the bottom of the dish.
- 99. Swirl the DI Water around in the dish and empty it into the 400T Waste bottle, through the funnel.
  - a. Note: Be careful to not spill this when swirling and to not pour this too quickly as this could cause it to spill.
- 100. Dispense more DI Water, such that it barely covers the bottom of the dish.
- 101. Swirl the DI Water around in the dish and empty it into the 400T Waste bottle, through the funnel.
  - a. Note: Be careful to not spill this when swirling and to not pour this too quickly as this could cause it to spill.
- 102. Move the dish that was used for the "Dirty" 400T solution over to the sink.
- 103. Dispense at least 100 mL of DI Water into the dish.
- 104. Swirl the DI Water around in the dish and empty it down the drain.
- 105. Dispense at least 100 mL more of DI Water into the dish.
- 106. Swirl the DI Water around in the dish and empty it down the drain.
- 107. Grab the cleanroom wipe that the "Dirty" 400T dish was sitting on and use it to wipe the dish dry.
- 108. Place this dish on the drying rack behind the sink.
- 109. Dispose of the cleanroom wipe used to dry the dish in the "Solid Chemical Waste" bin near the fume hoods.
- 110. Repeat Steps 97-109, but for the "Clean" 400T dish.
- 111.Remove the funnel from the 400T Waste bottle and place it in the location you found it in the secondary containers below the "Piranha" fume hood.
- 112. Place the cap to the 400T Waste bottle back on the bottle and screw it down tightly.

- 113. Move the 400T Waste bottle back to its location in the secondary containers beneath the "Piranha" fume hood.
  - a. Note: At this point, any PPE you have donned can be removed. Leave your nitrile gloves on your hands and your safety goggles/glasses covering your eyes while still in the cleanroom though.
- 114. Grab the IPA Waste bottle (or the IPA/Acetone Waste bottle) from the secondary container beneath the "Piranha" fume hood and place it inside the "Piranha" fume hood, behind the dwindling line of dishes.
- 115. Open the IPA Waste bottle (or the IPA/Acetone Waste bottle) and place the cap on the counter of the fume hood.
- 116. Grab the waste funnel from the secondary containers below the "Piranha" fume hood and place it inside the opened IPA Waste bottle (or IPA/Acetone Waste bottle).
- 117. Repeat Steps 97-109, but for the IPA Rinse dish and the IPA Waste bottle (or the IPA/Acetone Waste bottle).
- 118. Carefully grab the DI Water Rinse dish and empty its contents into the IPA Waste bottle (or IPA/Acetone Waste bottle), through the funnel.
  - a. Note: This dish will contain trace amounts of IPA, so it should NOT be emptied down the drain like normal DI Water can be.
- 119. Using the "D" labeled knob on the front right of the "Piranha" fume hood, dispense enough DI Water to barely cover the bottom of the dish.
  - a. Note: We just want to make sure that all IPA and other solutions are out of the dish.
- 120. Move the dish that was used for the DI Water Rinse bath over to the sink.
- 121. Dispense at least 100 mL of DI Water into the dish.
- 122. Swirl the DI Water around in the dish and empty it down the drain.
- 123. Dispense at least 100 mL more of DI Water into the dish.
- 124. Swirl the DI Water around in the dish and empty it down the drain.
- 125. Grab the cleanroom wipe that the DI Water Rinse dish was sitting on and use it to wipe the dish dry.
- 126. Place this dish on the drying rack behind the sink.
- 127. Dispose of the cleanroom wipe used to dry the dish in the "Solid Chemical Waste" bin near the fume hoods.
  - a. Note: Again, this is because trace amounts of IPA or other solution could be on the cleanroom wipe.
- 128. Remove the funnel from the IPA Waste bottle (or IPA/Acetone Waste bottle) and place it in the location you found it in the secondary containers below the "Piranha" fume hood.
- 129. Place the cap to the IPA Waste bottle (or IPA/Acetone Waste bottle) back on the bottle and screw it down tightly.
- 130. Move the IPA Waste bottle (or IPA/Acetone Waste bottle) back to its location in the secondary containers beneath the "Piranha" fume hood.
- 131. Remove the Marker and any other tools or supplies you have inside the "Piranha" fume hood.
- 132. Use the cleanroom wipe you used for pictures (or a new wipe if still being used for the dehydration bake) to wipe the working surface that you used of the "Piranha" fume hood.
  - a. Note: If anything is exceptionally dirty or not coming off (regardless of whether you created the dirtiness or not), use IPA and/or Acetone on the cleanroom wipe to clean up the bench surface.

- 133. Dispose of the cleanroom wipe you just used to wipe the fume hood's working surface in the "Solid Chemical Waste" bin near the fume hoods.
- 134. Turn off the sonicator by pressing the "Power" switch.
  - Note: If you heated the water bath in the sonicator at all, you will need to dump out the heated water (down the drain is fine) and refill the bath using a full plastic pitcher of DI Water. Be sure to allow time to let the bath cool down before adding more water to it (at least 10 minutes).
- 135. Close the sash to the "Piranha" fume hood (or at least push it down the majority of the way).
  - a. Note: If you skipped ahead to clean up the fume hood area while waiting for the dehydration bake to be completed, go back to complete Steps 88-93.
- 136. Make sure everything is off (IsoTherm Oven, sonicator, etc.) and that everything is out of the "Piranha" fume hood.
- 137. If you have not done so already, move the Wafer Box over to the microscope station so that you can complete Section 6.3.2 (Inspection).
  - a. Note: If you completed this during your processing, you are done with this section.

#### 6.3.2 Inspection

- 1. There are two methods of inspection we use to measure the quality of lift-off and metal layer(s) we get after the bath process of Lift-Off.
  - a. The first is a visual inspection using our eyes.
  - b. The second is a visual inspection using a microscope.
  - c. There are also optional tests to determine the exact thickness of metal and the surface profile of the wafer, using a DekTak or a Zygo Profilometer.
- 2. As a first inspection, look at the wafer and ensure you have gotten the correct pattern to what you were setting out to get from the photoresist/mask combination. Also inspect for defects like color patterns, burnt sections, peeling, or cracked metal layers. Any errors found cannot be reworked without etching (which may sacrifice biocompatibility), so let's hope they aren't catastrophic errors and that most of the devices will not be affected.
- 3. The second inspection involves using a microscope to inspect any potential defects found as well as to check for the presence of any micro cracks in the metal layer(s). After the Lift-Off process is when any small-scale cracks would appear, partially due to the ultrasonic vibrations of sonication, so under a microscope is where you will see the extent that these cracks reach. You will also need to inspect the traces to see if any of the metal has peeled up on the edges or if there are any missing or separated connections (obvious usually). This is where we will find mask errors that resolved, lift-off errors that appeared, and the overall metal quality.
- 4. The optional tests I mentioned are going to help us maintain an accurate measurement protocol throughout our processing, but are unnecessary since there is a calibrated quartz crystal that guides us on our metal thickness consistency.
  - a. The DekTak (ASU NanoFab has an easy-to-use model and ERC 444 has an ancient model) is going to give us results on the hill, valley, or step distance between one or more layers by dragging a stylus across the surface and returning the height value where it encountered resistance. This is great for measuring the thickness of a metal layer, but can also slightly damage (scratch/indent) metal or polymer layers like we use for these

devices. The elasticity of our polymers also makes data from those layers a little unpredictable since the stylus can actually push into the material a little easier than harder materials, like metal or Silicon.

- b. The Zygo Optical profilometer allows the same process the DekTak does to be completed (surface profile including hills, valleys, and steps), but over a much larger area and without direct contact onto the wafer's surface. This sounds like it would be the preferred methodology, but transparent films make it difficult to get quality data out of the system and sometimes a direct stylus approach will give you consistent data (when you feel the optical data is not consistent enough to be trusted).
  - i. Note: Mayo only has a Zygo, so they do not have a stylus-based profilometer. This is noteworthy because we can use either if we get inconsistent results with one to double check our results.

# 7 Conductive Metal Testing

## 7.1 Equipment Needed

- Micro Probe Station (at least 4 micromanipulators)
- Four-Point Probe Station
- Multimeter OR Data Acquisition Device (DAQ) OR Source Measure Unit (SMU)
- Microscope
- Camera (for microscopic imaging)

## 7.2 Materials Needed

- Wafer Tweezers or Wand (4")
- Wafer (3" or 4") Silicon w/ Polyimide & Conductive Metal
- Cleanroom Notebook
- Cleanroom Wipes
- Marker for wafer labeling
- Spare probe tips (Tungsten, Gold, or Stainless Steel likely)
- Calibration samples (Aluminum or Titanium or equivalent to ensure system is within range)

# 7.3 Testing Process

### 7.3.1 Four-Point Probe Testing (Stationary)

- The Four-Point Probe in the ASU NanoFab is quite helpful in getting general metal resistance results, which can be translated into sheet resistance and resistivity measurements. However, this needs to be done over a fixed (short) distance and the probes used are typically quite large. So this test will only give you the resistance of large metal pads (requires an area of at least 6-10 mm.
- 2. Turn on the Keithley multimeter (bottom far left)
- 3. Press the Ω4 button (middle-right underneath the display)
- 4. Press the Rate button (bottom middle-right) to go from "Med" to "Slow"
- 5. Check the interface to make sure it reads "Auto" (right side of display) and press the "Auto" button (right side of buttons, between the arrows) if this does not display "Auto"
- 6. Wait for 20+ minutes for the Keithley to warm up before using.

- a. Note: I typically turn the Keithley on as soon as the Platinum begins depositing, NOT the Titanium.
- 7. The following steps (Steps 8-30) should be repeated for each wafer in the batch that just completed metal deposition.
- 8. Remove the next wafer to test from the Wafer Box and place it on the glass plate underneath the probe unit of the system.
- 9. Move the wafer (and glass plate) around until you have found your first testing site.
  - a. Note: I use the large testing pads from the "Metrigraphics Electrodes" mask design exclusively, so my first testing site is bottom pad, top middle.
- 10. Slowly push the handle of the probe unit to the left (clockwise) until it bottoms out (DO NOT push this rapidly). This will make contact with the probe to the metal.
- 11. Wait for the reading on the Keithley multimeter to settle and record the value.
  - a. Note: This should be around 1  $\Omega$  for a 1,000 Å Platinum deposition, regardless of Titanium presence.
- 12. Slowly push the handle of the probe unit to the right (counterclockwise) until it bottoms out (DO NOT push this rapidly). This will release contact from the probe to the metal.
  - a. Note: ALWAYS do this before trying to move the wafer in order to prevent scratching the wafer surface.
- 13. Move the wafer (and glass plate) around until you have found your second testing site.
  - a. Note. My second testing site is bottom pad, top left.
- 14. Repeat Steps 10-12 for this measurement point.
- 15. Move the wafer (and glass plate) around until you have found your third testing site.
  - a. Note: My third testing site is bottom pad, top right.
- 16. Repeat Steps 10-12 for this measurement point.
- 17. Move the wafer (and glass plate) around until you have found your fourth testing site.
  - a. Note: My fourth testing site is bottom pad, bottom middle.
- 18. Repeat Steps 10-12 for this measurement point.
- 19. Move the wafer (and glass plate) around until you have found your fifth testing site.
  - a. Note: My fifth testing site is top pad, bottom middle.
- 20. Repeat Steps 10-12 for this measurement point.
- 21. Move the wafer (and glass plate) around until you have found your sixth testing site.
  - a. Note: My sixth testing site is top pad, bottom left.
- 22. Repeat Steps 10-12 for this measurement point.
- 23. Move the wafer (and glass plate) around until you have found your seventh testing site.
  - a. Note: My seventh testing site is top pad, bottom right.
- 24. Repeat Steps 10-12 for this measurement point.
- 25. Move the wafer (and glass plate) around until you have found your eighth testing site.
  - a. Note: My eighth testing site is top pad, top middle.
- 26. Repeat Steps 10-12 for this measurement point.
- 27. (Optional) If you would like to measure a ninth time (to line up with your measurement points from the Reflectometer), move to that location.
  - a. Note: My ninth testing site is top pad, far top and left of middle (to the immediate left of the "Top" label).
- 28. (Optional unless Step 30 is completed) Repeat Steps 10-12 for this final measurement point.
- 29. Once all of the measurements have been taken for this wafer, remove it from the glass plate.

- a. Note: BE CAREFUL of the probe tips to make sure that they do not scratch the surface of the wafer when removing from the testing apparatus.
- 30. Place the wafer that has just completed testing back into the Wafer Box it came from.
- 31. Repeat Steps 8-30 for each wafer in the batch that has just completed the metal deposition stage of the processing.
- 32. Once all wafers in the batch are complete, turn the Keithley multimeter unit Off (button is far left bottom of the unit).

### 7.3.2 **Probe Station Testing (Micromanipulators)**

- In order to test the full path length of the wires or electrode arrays that we design, a micro-probe station will be necessary to perform these tests. The system requires at least 2 micromanipulators, a microscope, a vacuum system, and a device to supply a current and record a voltage/resistance. In the lab at ASU, we have such a system set up in the dark room of ERC 320. Here we have a Keithley SMU to generate our supplied signal and measure our returned signal through the wire/electrode. The following steps will be specific to that system, but can be adjusted for different setups as well.
- 2. Turn on the Keithley 2636A Source Measurement Unit.
- 3. Press the "SRC" key for "Channel A".
- 4. Enter 100 mV using the dial.
  - a. Note: If this is not reading in Volts or milliVolts, you will have to press the "SRC" key again to get the system to switch modes.
- 5. Press the "Enter" key.
- Press the "MEAS" key for "Channel A" until you see the Current option (should be "A" or "mA" or "μA").
  - a. Note: Available measurement options are Voltage, Current, Power, and Resistance.
- 7. Press the "LMT" key for "Channel A".
- 8. Highlight the "Current Limit" option and push the dial in to "Select" the option.
- 9. Enter 100  $\mu$ A using the dial.
  - a. Note: This will likely be in mA, so you will enter 0.1 mA.
- 10. Press the "Enter" key.
- 11. Press the "Exit" key until you return to the main screen.
- 12. Press the "SRC" key for "Channel B".
- 13. Enter 0.00 mV using the dial.
  - a. Note: If this is not reading in Volts or milliVolts, you will have to press the "SRC" key again to get the system to switch modes.
  - b. Also: We are using Channel B as our "Sink" for the current to flow towards.
- 14. Press the "Enter" key.
- 15. Press the "MEAS" key for "Channel B" until you see the Current option (should be "A" or "mA" or "μA").
  - a. Note: Available measurement options are Voltage, Current, Power, and Resistance.
- 16. Press the "LMT" key for "Channel B".

- 17. Highlight the "Current Limit" option and push the dial to "Select" the option.
- 18. Enter 100  $\mu$ A using the dial.
  - a. Note: This will likely be in mA, so you will enter 0.1 mA.
- 19. Press the "Enter" key.
- 20. Press the "Exit" key until you return to the main screen.
- 21. Your Keithley SMU is now set up to test your probe contact quality and can be easily switched to get your resistance measurement. In the main area of 320 (outside of the dark room curtains), there is a Vacuum switch in the corner of the desk space closest to the dark room (by the COMSOL PC). Open this switch so that the handle is parallel with the line it is coming from (turn it clockwise towards you).
  - a. Note: Once the vacuum is activated, you should hear a hissing sound coming from the dark room, this is a good thing because it means you have vacuum running to your system!
- 22. Place your Wafer Box containing the samples you need to test along with your wafer tweezers inside the dark room, near/underneath the probe station.
  - a. Note: Be aware that the top surface of the probe station is where you will be moving the micromanipulators and the microscope head will also be moving, so it is not a safe place for a Wafer Box. Tweezers are fine up there though.
- 23. Ensure that the microscope is as high as it can go using the lever on the right side.
  - a. Note: The lever should be up and not down.
- 24. Turn on the light for the microscope using the switch on the light box.
  - a. Note: Follow the steel cable coming from the top of the microscope to the light box in order to find what this looks like.
- 25. The following steps (26-68) should be repeated for each wafer in the batch that needs to be tested.
- 26. Grab the next wafer in the batch to be tested from the Wafer Box.
- 27. Place the wafer on the platform underneath the microscope.
  - a. Note: I always place the flat towards myself for ease of orientation and because the microscope has a lot more room to move in the X-direction (left and right) than it does in the Y-direction (closer and further).
- 28. Flip the vacuum switch on the bottom right side of the probe station.
  - a. Note: The hissing should go away or get much quieter. This means your wafer now has a vacuum applied to it. If the hissing got louder, flip the switch in the other direction.
- 29. The following steps (30-63) should be repeated for each wire/array you are wanting to test on the current wafer.
- 30. Using the stage manipulators (underneath where the wafer is now), move the stage such that the wire/array you are wanting to test next is as centered underneath the microscope as it can be.
  - a. Note: DO NOT move these manipulators PAST their movement regions (or at least not too far). The manipulators can get stuck or damaged if they are pushed too far in or out.
- 31. Release the suction on the LEFT micromanipulator and twist/rotate it clockwise so that it is placed directly over the left contact point of the wire/array you are wishing to test.
  - a. Note: It is NOT recommended to lift this up off the top of the probe station as this could damage the tip of the probe on the micromanipulator.
  - b. Also: Make sure the height of the probe tip will not come into contact with the surface of the wafer (or anything else) as this will also damage the tip of the probe. You do not have

to align the Z-height of the wafer at this point, but just make sure it is high enough that it will not contact/scratch the surface.

- 32. Activate the suction on the LEFT micromanipulator once it is in place.
- 33. Release the suction on the RIGHT micromanipulator and twist/rotate it counterclockwise so that it is placed directly over the right contact point of the wire/array you are wishing to test.
  - a. Note: It is NOT recommended to lift this up off the top of the probe station as this could damage the tip of the probe on the micromanipulator.
  - b. Also: Make sure the height of the probe tip will not come into contact with the surface of the wafer (or anything else) as this will also damage the tip of the probe. You do not have to align the Z-height of the wafer at this point, but just make sure it is high enough that it will not contact/scratch the surface.
- 34. Activate the suction on the RIGHT micromanipulator once it is in place.
- 35. Lower the microscope head down using the lever on the right of the probe station.
  - a. Note: The lever should be down now.
- 36. Lower the Z-height of the LEFT micromanipulator probe (righty-tighty on the top screw) until it is very close to the surface of the wafer, but not touching.
  - a. Note: You can use your eyes for determining this height.
- 37. Lower the Z-height of the RIGHT micromanipulator probe (righty-tighty on the top screw) until it is very close to the surface of the wafer, but not touching.
  - a. Note: You can use your eyes for determining this height.
- 38. Move the microscope head (using the dials in the back of the probe station) in the X-direction and in the Y-direction until it is aligned to the LEFT contact point of the wire/array you are wishing to test.
  - a. Note: It does not matter which side you start on, it is actually recommended that you start on whatever side the microscope is closest to. In other words, the RIGHT and LEFT sides can be switch depending on preference and ease of adjustments.
- 39. Focus the microscope on the LEFT contact point you are trying to touch the probe tip onto.
  - a. Note: You should be able to see the probe tip (or shadow of your probe tip) in your view. If not, it is likely that your probe tip is either too high above the surface of the wafer or it is not aligned to your view. If it is too high: lower the probe tip a little further. If it isn't aligned: release the suction on the micromanipulator, twist/rotate the micromanipulator until the probe tip is in the view of the microscope, and activate the suction on the micromanipulator.
- 40. Lower the Z-height of the LEFT micromanipulator until the probe is very close to touching, but not quite touching yet.
- 41. Make any adjustments in the X-direction and Y-direction on the LEFT micromanipulator to center the probe tip on the contact point you are trying to touch the probe tip onto.
- 42. SLOWLY lower the Z-height of the LEFT micromanipulator so that the probe tip touches the surface of the wafer and push it a touch further.
  - a. Note: There is a feeling to performing this action that you will just have to get experience finding. The probe tip will contact the surface of the wafer and will push forward slightly, meaning that you are in contact with the surface. With Platinum, you will want to push the probe tip a little further in than the initial contact point.

- 43. CAUTION: You now have a probe tip touching the surface of the wafer. ANYTHING you do until that probe is released could potentially move the tip and scratch the surface of the wafer. Be very cautious about your movements.
- 44. Carefully move the microscope head (using the dials in the back of the probe station) in the X-direction (mostly) and hopefully just slight adjustments in the Y-direction until it is aligned to the RIGHT contact point of the wire/array you are wishing to test.
  - a. Note: It does not matter which side you start on, it is actually recommended that you start on whatever side the microscope is closest to. In other words, the RIGHT and LEFT sides can be switch depending on preference and ease of adjustments.
- 45. Focus the microscope on the RIGHT contact point you are trying to touch the probe tip onto.
  - a. Note: You should be able to see the probe tip (or shadow of your probe tip) in your view. If not, it is likely that your probe tip is either too high above the surface of the wafer or it is not aligned to your view. If it is too high: lower the probe tip a little further. If it isn't aligned: release the suction on the micromanipulator, twist/rotate the micromanipulator until the probe tip is in the view of the microscope, and activate the suction on the micromanipulator.
- 46. Lower the Z-height of the RIGHT micromanipulator until the probe is very close to touching, but not quite touching yet.
- 47. Make any adjustments in the X-direction and Y-direction on the RIGHT micromanipulator to center the probe tip on the contact point you are trying to touch the probe tip onto.
- 48. SLOWLY lower the Z-height of the RIGHT micromanipulator so that the probe tip touches the surface of the wafer and push it a touch further.
  - a. Note: There is a feeling to performing this action that you will just have to get experience finding. The probe tip will contact the surface of the wafer and will push forward slightly, meaning that you are in contact with the surface. With Platinum, you will want to push the probe tip a little further in than the initial contact point.
- 49. CAUTION: You now have both probe tips in contact with the surface of the wafer. ANYTHING you do until the probes are released could potentially move the tips and scratch the surface of the wafer. Be very cautious about your movements.
- 50. On the Keithley, you will need to activate both of the channels. Press the "Channel B" button (bottom right) to activate your Current Sink.
- 51. Press the "Channel A" button (bottom right) to activate your Current Source.
- 52. The Current measurement you should be getting on the Keithley should match almost exactly. This is due to Kirchhoff's Current Law and the conservation of current.
  - a. Note: If the current readings do not match across both Channel A and Channel B, your probe tips are not in quality contact with the Platinum on your wafer. Slowly lower your Z-heights of BOTH micromanipulators (CAREFULLY), one at a time, to see if this will create a connection. If this does not work, you will either have to lift the probe tips and try again in a slightly different spot (lots of work since you have to move the microscope head to the other side again) or you can consider your wire/array to be not conductive.
- 53. Since the Current readings match, you can switch the measurement type on "Channel A" to Resistance by pressing the "MEAS" button for "Channel A" until you get " $\Omega$ " or "k $\Omega$ " or "M $\Omega$ ".
  - a. Note: For 70 mm trace lengths, you should be getting k $\Omega$  and for short wires (< ~20 mm trace lengths) you should be getting  $\Omega$ .
- 54. Record this measurement in your notebook/spreadsheet.

- 55. Press the "Channel A" button (bottom right) to deactivate your Current Source.
- 56. Press the "Channel B" button (bottom right) to deactivate your Current Sink.
- 57. Raise the Z-height on the LEFT micromanipulator (lefty-loosey) until it reaches a height you are comfortable moving the wafer out from underneath.
  - a. Note: The higher the safer.
- 58. Raise the Z-height on the RIGHT micromanipulator (lefty-loosey) until it reaches a height you are comfortable moving the wafer out from underneath.
  - a. Note: The higher the safer.
- 59. Raise the microscope head by raising the lever on the right side of the probe station.
- 60. Release the suction on the LEFT micromanipulator and rotate/twist it counterclockwise until it is clear of the wafer surface.
- 61. Activate the suction on the LEFT micromanipulator.
- 62. Release the suction on the RIGHT micromanipulator and rotate/twist it clockwise until it is clear of the wafer surface.
- 63. Activate the suction on the RIGHT micromanipulator.
- 64. Repeat Steps 30-63 for each wire/array you are wanting to test on the wafer that is currently on the probe station stage.
- 65. Once you have tested all of the wires/arrays you planned on testing, you can go through the process of removing the wafer. Using the stage manipulators, move the stage back into the centered position.
- 66. Flip the vacuum switch (bottom right of probe station) to release the vacuum holding the wafer to the stage.
- 67. Remove the wafer from the stage using your wafer tweezers.
- 68. Place the completed wafer back into its Wafer Box.
- 69. Repeat Steps 26-68 for each wafer that needs to be tested.
- 70. Once all wafers have been tested, turn off the Keithley.
- 71. Turn off the light source for the microscope.
  - a. Note: Follow the metal wire coming from the top of the microscope to find this light source box.
- 72. In the main lab area of 320 (outside of the dark room), deactivate the vacuum line by pushing the switch counterclockwise toward the wall.
  - a. Note: This switch is located next to the COMSOL PC in the corner of the bench top if you forgot.
- 73. Remove your Wafer Box and wafer tweezers as well as any other supplies you had in the probe station area.

# 8 **Pre-Insulation Surface Preparation**

### 8.1 Equipment Needed

- Plasma Cleaner or RIE Machine
- Quartz ring (>4" diameter) for wafer containment
- Thickness Measurement tool (Filmetrics F20 Reflectometer or 3D Profilometer)

## 8.2 Materials Needed

- Wafer Tweezers or Wand (4")
- Wafer (3" or 4") Silicon w/ Polyimide
- Cleanroom Notebook
- Cleanroom Wipes
- Marker for wafer labeling

## 8.3 **Preparations Process**

#### 8.3.1 Inspection

- As with almost anything we are doing in this process, the wafers will need to be inspected prior to continuing in the processing. In this stage of the processing, however, it is crucial that we test both the Before thickness of the polyimide as well as the After thickness of the polyimide. This will give us a better idea of the consistency we have in the process.
- 2. Steps 3-22 should be repeated for both Before thickness measurements and for After thickness measurements.
- 3. At ASU, we use a Reflectometer in the form of a Filmetrics F20 system, so these processing steps will follow the technique we require for that process.
  - a. Note: We can program a new material into the Filmetrics Software if needed. A tutorial is being written about how to go about doing that. PI-2611 (soft-baked & cured), Parylene, AZ nLOF 2020, & AZ 15nXT are all present on the F20, soon to be added to the F40.
- 4. Ensure the PC at the F20 station is operational and move the mouse around if the monitor is blank.
- 5. Note: Steps 6-9 can be skipped if you have already used the system earlier in the day.
- 6. Run a baseline test by grabbing the blank Silicon wafer with your Wafer Tweezers or Wand and placing it underneath the beam of the system.
- 7. Click "Baseline" on the F20 software.
- 8. Click "OK" on the F20 software to grab the first baseline measurement.
- 9. The software will now ask you to tilt the object at 10°-80° to cancel out the background light it might see. Grab the Container the blank Silicon wafer was sitting on the place it next to the beam spot. Then grab the blank Silicon wafer and set it onto the container such that the flat part is sitting on the base while the curved part is elevated and resting partially on the container. Click "OK" in the software to grab the measurement.
- 10. The software is now ready for processing, so start by loading the correct recipe.
  - a. Note: Load "PI-2611 (Cured)" from "Others" for cured films. Load "PI-2611 (Soft-Baked)" from "Others" for soft-baked films.
- 11. Once the recipe is selected, you will now have to select the correct nominal thickness. Click "Edit Recipe" to change the nominal thickness as well as the measurement type (Angstroms, nanometers, or micrometers).
- 12. The following Steps (13-20) should be repeated for every wafer that needs to be tested for thickness.
- 13. Place the wafer that needs to be inspected onto the base such that the center of the wafer is directly underneath the beam of the system. This can be checked by placing your Wafer Tweezers or Wand underneath the beam and barely above your wafer to give yourself a better idea of where the beam is landing.
  - a. Note: Never touch your Tweezers or Wand directly onto the wafer surface!
- 14. Click the "Measure" button on the software to grab the measurement. Write this measurement into a table in your Cleanroom Notebook as the "Center" location.

- 15. Move the wafer (using your Wafer Tweezers or Wand's edge) to the right such that the left middle of the wafer is now underneath the beam.
  - a. Note: For reference, I consider the major flat to be the bottom of the wafer and the opposite side to be the top of the wafer. This makes everything easier to think about in my mind. Orientation is a personal choice and does not matter, as long as it is consistent.
- 16. Click the "Measure" button on the software OR press the "Enter" key on the keyboard. Write this measurement into a table in your Cleanroom Notebook as the "Left Middle" location.
- 17. Move the wafer to the right such that the left side is now underneath the beam.
  - a. Note: You do not want to capture the far edges of the wafer as this is where the edge bead has formed. Make sure this measurement is not on the edge bead section.
- 18. Click the "Measure" button on the software OR press the "Enter" key on the keyboard. Write this measurement into a table in your Cleanroom Notebook as the "Left" location.
- Continue the previous steps to get the "Right Middle", "Right", "Top Middle", "Top", "Bottom Middle", and "Bottom" measurements. Write each measurement into the table in your Cleanroom Notebook.
  - a. Note: These specific locations are important because they ensure consistency and they also cover enough sections of the wafer that any major defects can be determined simply by thickness variance. Gathering 9 points of data also helps in statistical verification as well as getting a surface profile of the wafer every time a process is completed.
- 20. Using your Wafer Tweezers or Wand, scoot the wafer off the base platform and load it back into the Wafer Box it came from.
- 21. Repeat Steps 13-20 for every wafer in the batch that is needing to be tested for thickness.
- 22. Once all wafers are completed, take the data from the Cleanroom Notebook you wrote it on and transfer it to an Excel sheet (or Google Sheet) such that it can be analyzed for averages, standard deviations, and other statistical calculations. Saving all data into a common location can help with product tracing throughout the device's fabrication as well as ensuring that the processing steps remain consistent.
  - a. Note: Examples of the tables used in the Cleanroom Notebook as well as the Excel tables can be provided. This can all be recorded directly into an Excel Sheet or Google Sheet instead of onto a cleanroom notebook. I write the measurements onto a cleanroom notebook just because that is the easiest and I have not implemented a Google Sheet for this yet.
- 23. Once you have completed the Before thickness measurements (Steps 3-22) and the entirety of section 8.3.2 (Surface Roughening), you will now have to repeat Steps 3-22 of this section in order to get the After thickness measurements.

### 8.3.2 Surface Roughening

 Polyimide has a natural surface roughness with features of about 1-2 nm in height due to the long-chain nature of the polymer. Prior to deposition of any additional layers, it is helpful to roughen the surface a little more so that the feature sizes increase to about 5-6 nm in height. This can be accomplished through a plasma cleaning process using an ignited oxygen plasma.

- a. Note: This is a recommended process from the datasheet that does not necessarily need to be done, but it does help further layers to adhere better to the existing substrate. I will be doing this for every wafer we process at ASU from now on.
- 2. In the ASU NanoFab, we use a PlasmaTherm PT-790 Reactive Ion Etching Tool for this "plasma cleaning" procedure. There is a Harrington Plasma Cleaning system in our smaller cleanroom, but this has not been tested yet for this purpose.
- 3. The PT-790 system is left under vacuum when not in use, so it allows us to prepare the chamber for our process before we even add our samples. Since we will be using an Oxygen plasma cleaning process, the recipe is called "Clean" and is designed to clean the chamber of any particles leftover from previous runs.
  - a. Recipe: "Clean" 50 sccm  $O_2$  200 W Forward Power 40 mTorr Pressure 10 minutes for a complete clean, 1-2 minutes for a quick chamber prep
  - b. Note: Each recipe has 2 minutes that are tacked on top of whatever the duration time is so that the machine can evaporate all of the water molecules from the air (humidity), hold the gas flow amounts steady, hold the vacuum pressure steady, and ignite the plasma.
- 4. Click on the "Log In" button on the upper right-hand side of the interface.
- 5. A pop-up window should appear with a list of usernames on the left and a password box in the upper right. Select your username credentials from the list on the left.
- 6. Enter your password into the box on the upper right-hand side of the pop-up window.
- 7. Click the "Log In" button below the password box to complete the Log In process.
- 8. Select the bottom left "Recipes" menu button, Click on "Clean" from the list of recipes and Click "Run Recipe" on the left-hand side of the software. This will bring up a pop-up window asking if you are sure you would like to run the specified recipe. Ensure that it is the correct recipe ("Clean") and Click "Yes" in the bottom of the pop-up window.
- 9. Another pop-up window will show up and will ask you to enter the run time for the recipe. Enter 2 minutes (default is 10 minutes) and hit Tab when done. Then select "Start Recipe" towards the bottom of the pop-up window and the system will begin to go through the steps of the recipe.
  - a. Note: This can be set to any amount, but I use 2 minutes just to make sure the gas is flowing, ignited, and interacting with everything for at least a minimal amount of time.
  - b. Note: Actual run time is approximately 5 minutes.
- 10. Once the recipe is complete and the system reads "Pumping Idle", Click on "Vent" to bring the system up to atmosphere.
  - a. Note: Vent time is approximately 2-3 minutes.
- 11. Once the system reads "Vented" and the chamber is filled in with a blue color (in the "Services" tab), the system is at atmosphere and you can open the chamber lid. Do so carefully but with force (the system was just under vacuum, so might be hard to open, but it cannot be slammed open)
  - a. Note: When the chamber lid is open, you want to make sure the lid is open for the least amount of time possible, so make sure your wafers/samples are ready to go before you open the lid.
- 12. Steps 13-28 below should be repeated for each wafer you are requiring a surface roughening on.
  - a. Note: It is highly recommended to do 1 wafer at a time for this process as it helps to ensure an even roughening across the entire wafer.
- 13. Place your sample(s) in the center of the platen. A quartz ring is available if it is desired to ensure the sample stays in the direct center.

- a. Note: Doing 1 wafer at a time is highly recommended. Multiple wafers bring about inconsistent etching rates and some "dead zones". If doing multiple wafers at a time, a plan for rotating the wafers will be needed (stop etch, vent, rotate, pump, resume etch) in order to get a consistent etch result
- b. Note: The quartz ring is recommended when it is only one wafer going through the process to keep the wafer in the center of the platen.

14. Once the sample(s) and/or quartz ring are on the platen inside the chamber, you may shut the lid.15. With the lid shut, click "Pump Turbo" on the interface.

- a. Note: This button can be found on any tab on the interface, but it is recommended to use the "Services" tab as this will show the user when the chamber is under vacuum
- 16. Once the "Pump Turbo" button has been selected, weight must be placed on the chamber lid (lean down onto the handle of the lid) in order to keep it sealed while the vacuum systems begins to pump the chamber down.
  - a. Note: This should be done until the chamber is no longer filled in with a blue color ("Services" tab) and when the chamber pressure drops from 760 Torr to 10.0 Torr.
- 17. Wait for the system to achieve a chamber pressure of 0 mTorr before proceeding to the recipe selection.
- 18. Click on the "Recipes" tab at the bottom of the interface once the system reads "Pumping Idle" and is at 0 mTorr.
- 19. The "Clean" recipe should be highlighted from the previous run (chamber preparations or previous sample). If this is not highlighted, for some reason, Click on the "Clean" recipe.
- 20. Click "Run Recipe" on the left-hand side of the interface.
- 21. A pop-up window should appear, asking for confirmation that you have selected the correct recipe. If the recipe it lists reads "Clean", you have the correct recipe and can Click "Continue". If not, Click "Close" and re-select the correct recipe.
- 22. Another pop-up window should appear, asking for the duration of the recipe. The default is set to 10 minutes. Change this to 1 minute and hit the "Tab" key on the keyboard to confirm the change.
- 23. Click the "Run Recipe" button once the time reads "1 minute".
- 24. While the recipe is starting, now would be a great time to fill out the run log.
  - a. Note: This should include the Wafer ID (I use batch number-wafer number, i.e. B7-026), the etch time (1 minute), etch rate (approximately 1,700 Angstroms/minute), the material(s) being etched (Polyimide / PR), the platen material (Graphite), the substrate material (Silicon), the chamber pressure used in the recipe (40 mTorr), and any notes about the run you may want to enter (I usually leave it blank)
  - Note: You will need to record the Forward Power (typically 150 W for the Clean recipe), the Reflected Power (typically 1 W for the Clean recipe), and the DC Offset (typically 417 V for the Clean recipe). Be sure to get actual values from the run that you enter into the chart.
- 25. Once the recipe has completed and the system reads "Pumping Idle", Click on "Vent" to vent the system to atmosphere.
- 26. Wait for the system to reach 760 Torr, for the chamber to be filled in with a blue color ("Services" tab), and for the system to read "Vented".
- 27. Once the system reads "Vented" and the chamber has been filled in with a blue color ("Services" tab), open the lid to the system.

- 28. Remove the sample that has just completed the Surface Roughening and place it in your Wafer Box.
- 29. Repeat Steps 13-28 until all wafers have been processed.
- 30. Once all wafers in the batch have been processed, ensure there are no wafers (or quartz rings) on the platen and close the lid to the chamber.
- 31. Click the "Pump Turbo" button on the left-hand side of the interface.
- 32. Apply pressure to the handle of the lid to ensure the lid is sealed properly.
  - a. Note: This should be done until the system reads 10.0 Torr instead of 760 Torr and the chamber is no longer filled in with a blue color ("Services" tab).
- 33. Wait for the system to read "Pumping Idle" and is at a pressure of 0 mTorr.
- 34. Click on the "Recipes" tab on the bottom of the interface.
- 35. Select the "Clean" recipe if it is not already selected.
- 36. Click on the "Run Recipe" button on the left-hand side of the interface.
- 37. A pop-up window should ask for confirmation that you are running the correct recipe. If this reads "Clean", then click "Continue".
- 38. Another pop-up window should ask for duration of the run. Set this to 2 minutes and hit the "Tab" key on the keyboard to confirm the change.
- 39. Click the "Run Recipe" button if the duration is set to 2 minutes.
- 40. Once the recipe has begun running, Click the "Log In" button on the top right of the interface.
- 41. A pop-up window should appear asking for username and password. Click the "Log Out" button on the right-hand side of the pop-up window.

# 9 Insulation Deposition

# 9.1 Equipment Needed

- Parylene Coater (SCS brand)
- Balance (to weigh Parylene dimer)
- Aluminum Foil (~4" x ~8" strips are what we use here)
- Weight boat (for Parylene dimer)
- Aluminum Forming rod (we have one here to shape the Aluminum boat, I can get dimensions if needed)
- Thickness Measurement Tool (Filmetrics F20 Reflectometer or 3D Profilometer)
- Microscope (for inspection)
- Camera (for microscopic imaging)

## 9.2 Materials Needed

- Wafer Tweezers or Wand (4")
- Wafer (3" or 4") Silicon w/ Polyimide & Conductive Metal
- Cleanroom Notebook
- Cleanroom Wipes
- Marker for wafer labeling
- Parylene-C dimer (pellets)
- Razor blade (to scrape residue off cold-trap of Parylene coater)

## 9.3 Deposition Process

#### Preface

- We have two different insulation materials at this point in our fabrication process: Polyimide and Parylene. However, we have already gone over the deposition process for Polyimide, so this section will be solely based on the deposition of Parylene as the insulation layer.
- If you are wishing to use Polyimide as your insulating layer, please refer to Section 2 and follow the steps for spin-coating Polyimide.
- Based on a paper I have seen (<u>Ceyssens, F. & Puers, R. (2015) J. Neur, Eng.</u>), we may be altering the curing temperatures depending on the layer of Polyimide you deposit, but that can be adjusted in the Process Sheets document and not the Procedures document.
  - Note: Future adjustment may be 205°C for the maximum curing temperature for the first (substrate) layer of Polyimide while the normal 350°C will remain the maximum curing temperature for the second (insulation) layer of Polyimide. Due to this change in curing profiles, this will also eliminate the need for a "Descum" or "Surface Roughening" of the substrate layer prior to deposition of the insulation layer since it let to a mechanical peel test failure for the group that published the above paper.

### 9.3.1 Coater Loading

- 1. The ASU NanoFab has an SCS Coater machine that is dedicated to the deposition of Parylene-C. This machine is managed by the cleanroom staff and therefore you will have to reserve time on the machine using the scheduling tool on the ASU NanoFab website.
- 2. BEFORE you enter the cleanroom, you will have to make sure you have some crucial items with you (or in your toolbox). [Read: I have forgotten multiple items before and my deposition was severely delayed because of it]. Those items are listed below:
  - a. Wafers (in a Wafer Box)
  - b. Wafer Tweezers
  - c. Parylene-C Dimer (pre-weighed for accurate results)
    - Note: The scale in the ASU NanoFab has been broken for a while, so use the scale in 454 to measure your Parylene-C amounts. <u>You will get approximately</u> 2.3 μm per 1 gram of dimer.
  - d. Aluminum Foil strip
    - i. Note: This should be the appropriate size for the tube, so either use the strips currently in the box for measurement or figure out how wide they need to be and use a ruler each time.
  - e. Kapton Tape
    - i. Note: This is important to keep the wafers in the position you placed them in to prevent a catastrophe like I encountered
- 3. Once you arrive at the system, deactivate the E-Stop button (twist and pull towards you).
- 4. Press the "Main Power" button (top left of the button interface).
- 5. Behind the machine is the chiller. Flip the "Power" switch on the left side of this device to activate the cold trap chiller.
  - a. Note: ASU NanoFab recommends that this chiller runs for at least 20-30 minutes in order to reach its operating temperature. By the time you are done with the system setup, this time should be close enough.
- 6. On the button interface, flip the switch from "Vacuum" to "Hold" and then to "Vent".
  - a. Note: You can do this all in one fell swoop or hold at the "Hold" option and then continue to "Vent". Up to your preference.
- 7. Once the system reaches atmosphere (is no longer increasing), remove the cold trap from its location and place it in the holder on the side of the machine.
  - a. Note: The cold trap looks like a long metal rod/tube that is in the back right corner of the top of the machine.
- 8. Inspect the cold trap to ensure that the previous user cleaned it completely.
  - a. Note: If the cold trap still has any debris on it, clean it off with a razor blade (which can be found in the cabinet beneath the system).
- 9. Open the lid of the "Parylene Waste" bucket that is below the cold trap.
- 10. Spray the cold trap with de-adhesion solution, located in the spray bottle behind the machine on top of the chiller. When spraying the cold trap, coat the entire surface with the de-adhesion solution.
  - a. Note: I forget the mixture off the top of my head, but this is listed in the User Manual the ASU NanoFab staff wrote, sitting on top of the chiller.

- b. Also: You will need to let all of the de-adhesion solution drip off the cold trap and into the "Parylene Waste" bucket before you place it back into its location in the machine, so just let it drip while you complete the rest of the set up.
- 11. Check the temperature of the Vaporizer in the indicators on the button interface panel to be sure this temperature is below 40°C.
  - a. CAUTION: If this is above 40°C, DO NOT PROCEED as it will likely be too hot to touch.
- 12. Open the cabinet on the bottom of the machine.
- 13. Grab the boat-forming tube and place it on the table behind you.
- 14. Grab the aluminum foil piece from your toolbox.
  - a. Note: This should hopefully already be cut to size, but if you have a full roll, tear off a piece approximately 4" wide.
- 15. Face the shiny side of the aluminum foil towards yourself.
- 16. Place the boat-forming tube on the aluminum foil such that it is along the length of the foil (long side to long side) and allow an end of the boat-forming tube to go past the aluminum foil by approximately 1-2 inches.
- 17. Wrap the aluminum foil around the edge of the boat-forming tube, covering the opening to the boat-forming tube
  - a. Note: When performing this step, be sure to press the aluminum foil against the edges of the boat-forming tube so that you get a creased circle remaining in the aluminum foil shape.
- 18. Wrap the aluminum foil around the boat-forming tube such that it conforms to the shape of the boat-forming tube.
- 19. Press all of the aluminum foil against the boat-forming tube so that it is as flush with the surface as it possibly can be.
- 20. Remove the boat-forming tube from the aluminum foil boat.
  - a. Note: It should retain the shape you just formed it into.
- 21. Grab your Parylene-C dimer from your toolbox.
  - a. Note: WRITE DOWN the amount of Parylene-C dimer you are using so that you can reference it when you record your thickness measurements after the deposition.
  - b. Also: I like to pre-weigh my dimer and place it into 15 mL centrifuge tubes so that we can just pour it from the tubes and re-use it after we are done. If you do not have any pre-weighed dimer, you will have to grab the weigh boat from the cabinet below the machine and use the scale by the entrance to the cleanroom (if it is actually working). You will get approximately 2.3 µm per 1 gram of dimer.
- 22. Pour the Parylene-C dimer into the aluminum foil boat.
  - a. Note: Pour the dimer into the boat in a way that spreads it around evenly within the boat. Do not clump it in one area and leave it like that.
- 23. Carefully bring the full aluminum foil boat over to the open cabinet beneath the machine.
- 24. Remove the latch above the Vaporizer tube.
- 25. Open the Vaporizer tube and ensure there is nothing inside.
  - a. Note: If there is still a boat inside, remove the boat carefully and place it in the "Metal Waste" bucket behind you and next to the Lesker #4 system.
  - b. Side Note: This shouldn't happen, but I want to add this just in case. If there is a boat inside the Vaporizer and it still has Parylene-C dimer in it (and you didn't put it there),

someone is somehow still using the system - consult the ASU NanoFab staff before proceeding.

- 26. Carefully slide your full aluminum foil boat about 3/4 of the way into the Vaporizer tube.
  - a. Note: When you are sliding it into the tube, watch the edges of the boat to make sure they are maintaining their shape and going into the tube smoothly.
- 27. You should have part of the aluminum foil boat sticking out of the tube. Using your thumb and index finger, make an indent in the top of the boat form.
  - a. Note: This indent is going to be how you remove the boat from the tube after the deposition has completed, so be sure that it is wide enough for you to grab onto with your fingers. Because the boat is mostly in the tube already, this should not deform your boat very much except at the place you are indenting.
- 28. Push the boat all the way into the Vaporizer tube so that the edge of the boat is flush with the edge of the tube.
- 29. Close the opening to the Vaporizer tube.
- 30. Secure the latch over the Vaporizer tube opening.
- 31. Return the boat-forming tube to the cabinet it came from.
  - a. Note: If you needed to use a razor blade or the weigh boat at any point, return it back to the cabinet at this time.
- 32. Return your Parylene-C dimer storage capsule.
  - a. Note: This would be the 15 mL centrifuge tube you had a pre-weighed amount of dimer in.
- 33. Close the cabinet door and secure the latch.
- 34. Open the main chamber lid by using a twisting motion on the handles.
  - a. Note: This may require brute strength if the system has not seen vacuum grease in a while. Do not be afraid to ask for help to employ the two-person technique instead.
  - b. Two-Person Technique: One person on each handle. Both lift up at the same time to lift the chamber lid up. Carefully clear the chamber lid of the stage once it has been lifted from its location.
- 35. Once the chamber lid has been released of the suction, you may lift the chamber up and off the system.
  - a. Note: BE CAREFUL around the stage so that you do not damage the chamber or the stage.
- 36. Flip the chamber lid upside-down and place it on top of the chiller.
  - a. Note: The chamber lid is oddly shaped, so make sure this is in a stable position on top of the chiller before releasing it.
- 37. If the rubber seal is still stuck to the chamber, remove it carefully and place it around the bottom of the chamber lid.
  - a. Note: If you have to place the rubber seal around the bottom of the chamber lid, make sure the seal wraps all the way around the bottom of the chamber lid and is contacting the whole lid (and not hanging into the chamber lid).
- 38. Check the rubber seal to make sure it does not have cracks or debris and also press it down against the bottom of the chamber lid to make sure it is securely on the chamber lid.
- 39. Grab the Nilfisk vacuum and clean out the chamber lid, wafer stage, and the entire chamber.
- 40. Grab your Wafer Box containing the wafers in need of Parylene deposition and open it up.
- 41. Grab your wafer tweezers from your toolbox.

- 42. Place your wafers onto the wafer stage in the chamber of the system.
  - a. Note: There is a batch stage, but I would not recommend using it unless we have the baffle rod to disperse the Parylene throughout the system. Last I checked, Todd was unable to find this baffle rod, so DO NOT use this batch stage without that baffle rod.
  - b. Also: A maximum of 4 100 mm (4") wafers can fit on this wafer stage. DO NOT overfill the system.
- 43. Grab your Kapton tape from your toolbox.
- 44. Grab some scissors.
  - a. Note: You can find scissors near Lesker #1's table or in the clear plastic box near Lesker #4.
- 45. Cut 8 pieces of Kapton tape, approximately 1/4" in length.
- 46. Place Kapton tape on the Left and Right (1 piece for each side) of each wafer on the wafer stage.
  - a. Note: We do this so that the wafers do not move at all during the process. This also gives us an opportunity to do a thickness measurement using the DekTak if we wanted to confirm our Reflectometer results.
- 47. Close the Wafer Box.
- 48. Grab the chamber lid and return it to the orientation it was in when you first removed it.
  - a. Note: This should be correct-side up and window facing towards you.
- 49. Place the chamber lid onto the chamber and ensure that it is completely flush with the chamber surface.
  - a. Note: Twist it around to make sure it can rotate freely if you wish to check this.
- 50. Return the cold trap to its location inside the machine.
- 51. Flip the switch from "Vent" to "Hold".
- 52. Hold the cold trap in its place and maintain that hold until the system is under vacuum.
- 53. Flip the switch from "Hold" to "Vacuum".
  - a. Note: Continue holding the cold trap when you flip the switch.
- 54. Once the vacuum is reading below 850 vacuum units, you can release your hold on the cold trap.
  - a. Note: It happens pretty quickly, so I usually release around 800 vacuum units, just to be safe.
- 55. The system will now reduce the vacuum reading. You will be waiting until the system reaches its vacuum set point before beginning your process.
  - a. Note: This set point is 30 vacuum units in the ASU NanoFab.
- 56. Return your wafer tweezers to your toolbox.
- 57. Return your Wafer Box to its storage location.
  - a. Note: I put mine in the toolbox, so either do that or place it on the table behind you if you carried it into the cleanroom.
- 58. Return the scissors to where you got them from.
- 59. Return the Kapton tape to your toolbox.
- 60. Grab the Log Book, located on top of the chiller.
- 61. Write your name, the time and date, the amount of Parylene-C dimer you used, and the anticipated time you will be removing the wafers from the system.
- 62. Return the Log Book to the top of the chiller.
- 63. Once the system reaches the vacuum set point, press the "Start" button (bottom right of button interface) to begin the process.
- 64. The green light of the "Start" button should illuminate to notify you that you have a process running.
- 65. You can now grab your belongings and exit the cleanroom.
  - a. Note: The green light of the "Start" button will flash when the deposition is complete, so that is how you know when you need to return to the cleanroom.

#### 9.3.2 Coater Unloading

- 1. BEFORE you enter the cleanroom, check to be sure that you have a flashing green light on the "Start" button of the machine. You can see this from the windows outside of the cleanroom.
- 2. Once you enter the cleanroom, go to the machine and press the "Start" button to turn off the flashing light.
- 3. Flip the switch from "Vacuum" to "Hold".
- 4. Turn off the chiller, located behind the system.
- 5. Flip the switch from "Hold" to "Vent".
  - a. Note: Allow the vacuum reading to stop increasing before you do anything else.
- 6. Once the chamber has reached atmosphere, open the main chamber lid by using a twisting motion on the handles.
  - a. Note: This may require brute strength if the system has not seen vacuum grease in a while. Do not be afraid to ask for help to employ the two-person technique instead.
  - b. Two-Person Technique: One person on each handle. Both lift up at the same time to lift the chamber lid up. Carefully clear the chamber lid of the stage once it has been lifted from its location.
- 7. Once the chamber lid has been released of the suction, you may lift the chamber up and off the system.
  - a. Note: BE CAREFUL around the stage so that you do not damage the wafers or the chamber/stage.
- 8. Flip the chamber lid upside-down (once clear of your wafers) and place it on top of the chiller.
  - a. Note: The chamber lid is oddly shaped, so make sure this is in a stable position on top of the chiller before releasing it.
- 9. If the rubber seal is still stuck to the chamber, remove it carefully and place it around the bottom of the chamber lid.
  - a. Note: If you have to place the rubber seal around the bottom of the chamber lid, make sure the seal wraps all the way around the bottom of the chamber lid and is contacting the whole lid (and not hanging into the chamber lid).
- 10. Check the rubber seal to make sure it does not have cracks or debris and also press it down against the bottom of the chamber lid to make sure it is securely on the chamber lid.
- 11. Open the cabinet below the chamber.
- 12. Grab a razor blade from the cabinet.
- 13. CAREFULLY use the razor blade to remove the Kapton tape from the sides of your wafers.
- 14. Put the razor blade back in the cabinet when you are done removing your wafers from the stage.
- 15. Grab your wafer tweezers from your toolbox.
- 16. Grab your Wafer Box and open it.
- 17. Move each of your wafers from the stage into their respective slots in your Wafer Box.

- 18. Close the Wafer Box and put it back where you got it (toolbox or table).
- 19. Put your wafer tweezers back in your toolbox.
- 20. Grab the chamber lid and return it to the orientation it was in when you first removed it.
  - a. Note: This should be correct-side up and window facing towards you.
- 21. Place the chamber lid onto the chamber and ensure that it is completely flush with the chamber surface.
  - a. Note: Twist it around to make sure it can rotate freely if you wish to check this.
- 22. Check the temperature of the Vaporizer in the indicators on the button interface panel to be sure this temperature is below 40°C.
  - a. CAUTION: If this is above 40°C, DO NOT PROCEED as it will likely be too hot to touch.
- 23. Remove the latch above the Vaporizer tube.
- 24. Open the opening to the Vaporizer tube.
- 25. Remove the aluminum foil boat from the Vaporizer tube.
  - a. Note: All of your Parylene dimer should be gone. Ensure that this is the case. (If not, the process did not run and your wafers did not get coated).
  - b. Also: If you have trouble removing the aluminum foil boat and end up pushing it back into the tube, you can grab some of the sample tweezers (not wafer tweezers) from the toolbox to grab the wafer boat out of the tube.
- 26. Place the used aluminum foil boat into the "Metal Waste" bucket behind you (next to Lesker #4).
- 27. Close the opening to the Vaporizer tube.
- 28. Secure the latch over the Vaporizer tube opening.
- 29. Remove the cold trap from its place in the top of the system and place it in the holder to the right of the system.
- 30. Grab the razor blade from the cabinet below the chamber.
- 31. Use the razor blade to scrape off ALL of the Parylene deposit that has gathered on the cold trap.
  - a. Note: The cold trap will likely still be very cold, so be sure to take breaks if your hands are getting too cold. Also remember to wipe off the flakes from your razor blade often to make sure you are not getting build-up on the blade.
- 32. If you are having a hard time getting some of the deposits off the cold trap, you can use the spray bottle of de-adhesion solution to coat the cold trap again to help you remove those tricky deposits.
- 33. Once all of the deposits and debris is removed from the cold trap, place the cold trap back into its position in the system.
- 34. Flip the switch from "Vent" to "Hold".
- 35. Check to be sure that all wafers are removed from the stage, the aluminum foil boat is not in the Vaporizer tube, the Vaporizer tube is closed and latched, the cold trap is in place, and the chamber lid is securely on the chamber surface.
- 36. Hold the cold trap in its place and maintain that hold until the system is under vacuum.
- 37. Flip the switch from "Hold" to "Vacuum".
  - a. Note: Continue holding the cold trap when you flip the switch.
- 38. Once the vacuum is reading below 850 vacuum units, you can release your hold on the cold trap.
  - a. Note: It happens pretty quickly, so I usually release around 800 vacuum units, just to be safe.
- 39. The system will now reduce the vacuum reading. You will be waiting until the system reaches a satisfactory vacuum level for you to shut the system down under.

- a. Note: This recommended vacuum level is 100 vacuum units in the ASU NanoFab. However, if the system is under 200, you can still shut the system down (especially if the pressure is increasing instead of decreasing like it should be).
- 40. Once the system reaches the desired vacuum level, push the E-Stop button.
- 41. This will shut down the power to the system and leave it under a decent level of vacuum.
- 42. You can now clean up your belongings and you are done with the operation of this machine. I would recommend proceeding to section 9.3.3 "Inspection" before you leave the cleanroom so that you can get a measurement of the thickness of your newly deposited layer.

#### 9.3.3 Inspection

- 1. There are three methods of inspection we use to measure the quality of Parylene we get after spinning, baking, and curing.
  - a. The first is a visual inspection using our eyes.
  - b. The second is a visual inspection using a microscope.
  - c. The third is a metric inspection using a measurement tool to get the thickness of the layer.
- 2. As a first inspection, just looking at the wafer will show us some large defects if they are present. This would include bubbles, tears, voids, and colored swirls. The bubbles typically indicate dirt and debris beneath the layer or an adhesion problem while tears and voids indicate that the layer wasn't complete. Bubbles can continue into further processing (unless they are excessively large or covering sections of a device) while voids and tears means the entire wafer will have to be reworked to prevent additional defects (unless the tear or void is outside of the region that contains a device). Colored swirls on the other hand, are natural of the Parylene material based on the frequency and wavelength of white and filtered light. However, if these swirls are too prevalent and too close together, then the measurement techniques will have a very hard time differentiating between the thicknesses in those areas. This is a very subjective determination, but also why the process has been set in the manner that it is listed above, to remove as many inconsistencies as possible.
- 3. The second inspection involves using a microscope to inspect the open areas with no defects to ensure the entire material is as uniform as possible. This also gives us the chance to investigate the defects we found from the first inspection and see if they are actual defects or just blemishes.
- 4. The third inspection is where we measure the thickness of the Parylene layer we just spun onto the wafer. At ASU, we use a Reflectometer in the form of a Filmetrics F20 system, so these processing steps will follow the technique we require for that process.
  - a. Note: We can program a new material into the Filmetrics Software if needed. A tutorial is being written about how to go about doing that. PI-2611 (soft-baked & cured), Parylene, AZ nLOF 2020, & AZ 15nXT are all present on the F20, soon to be added to the F40.
- 5. Ensure the PC at the F20 station is operational and move the mouse around if the monitor is blank.
- 6. Run a baseline test by grabbing the blank Silicon wafer with your Wafer Tweezers or Wand and placing it underneath the beam of the system.
- 7. Click "Baseline" on the F20 software.
- 8. Click "OK' to grab the background measurement.

- 9. The software will now ask you to tilt the object at 10°-80° to cancel out the background light it might see. Grab the Container the blank Silicon wafer was sitting on the place it next to the beam spot. Then grab the blank Silicon wafer and set it onto the container such that the flat part is sitting on the base while the curved part is elevated and resting partially on the container. Click "OK" in the software to grab the measurement.
- 10. The software is now ready for processing, so start by loading the correct recipe.
  - a. Note: Load "Parylene" and set "Platinum" as your substrate material. This will help you get accurate thickness readings without bringing the thickness of your polyimide into question (and creating a harder stack for the Reflectometer to analyze).
- 11. Once the recipe is selected, you will now have to select the correct nominal thickness. Click "Edit Recipe" to change the nominal thickness as well as the measurement type (Angstroms, nanometers, or micrometers).
- 12. The following Steps (13-20) should be repeated for every wafer that needs to be tested for thickness.
- 13. Place the wafer that needs to be inspected onto the base such that the center of the wafer is directly underneath the beam of the system. This can be checked by placing your Wafer Tweezers or Wand underneath the beam and barely above your wafer to give yourself a better idea of where the beam is landing.
  - a. Note: Never touch your Tweezers or Wand directly onto the wafer surface!
- 14. Click the "Measure" button on the software to grab the measurement. Write this measurement into a table in your Cleanroom Notebook as the "Center" location.
- 15. Move the wafer (using your Wafer Tweezers or Wand's edge) to the right such that the left middle of the wafer is now underneath the beam.
  - a. Note: For reference, I consider the major flat to be the bottom of the wafer and the opposite side to be the top of the wafer. This makes everything easier to think about in my mind. Orientation is a personal choice and does not matter, as long as it is consistent.
- 16. Click the "Measure" button on the software OR press the "Enter" key on the keyboard. Write this measurement into a table in your Cleanroom Notebook as the "Left Middle" location.
- 17. Move the wafer to the right such that the left side is now underneath the beam.
  - a. Note: You do not want to capture the far edges of the wafer as this is where the edge bead has formed. Make sure this measurement is not on the edge bead section.
- 18. Click the "Measure" button on the software OR press the "Enter" key on the keyboard. Write this measurement into a table in your Cleanroom Notebook as the "Left" location.
- 19. Continue the previous steps to get the "Right Middle", "Right", "Top Middle", "Top", "Bottom Middle", and "Bottom" measurements. Write each measurement into the table in your Cleanroom Notebook.
  - a. Note: These specific locations are important because they ensure consistency and they also cover enough sections of the wafer that any major defects can be determined simply by thickness variance. Gathering 9 points of data also helps in statistical verification as well as getting a surface profile of the wafer every time a process is completed.
- 20. Using your Wafer Tweezers or Wand, scoot the wafer off the base platform and load it back into the Wafer Box it came from.
- 21. Repeat Steps 13-20 for every wafer in the batch that is needing to be tested for thickness.
- 22. Once all wafers are completed, take the data from the Cleanroom Notebook you wrote it on and transfer it to an Excel sheet (or Google Sheet) such that it can be analyzed for averages,

standard deviations, and other statistical calculations. Saving all data into a common location can help with product tracing throughout the device's fabrication as well as ensuring that the processing steps remain consistent.

a. Note: Examples of the tables used in the Cleanroom Notebook as well as the Excel tables can be provided. This can all be recorded directly into an Excel Sheet or Google Sheet instead of onto a cleanroom notebook. I have to write the measurements onto a cleanroom notebook just because that is the nature of the lab in which the measurement tool is located in and I have not implemented a Google Sheet for this yet.

# **10 Photoresist Deposition for Electrode Contacts**

## **10.1 Equipment Needed**

- Spinner
- Mask Aligner (i-line UV) w/ Z-axis calibration (~66 mJ/cm<sup>2</sup>)
- Fume Hood (for developer)
- Pyrex dish (>4" diameter) (Qty: 2)
- Hot Plate or Oven (110C)
- Nitrogen air gun (for wafer drying)
- Microscope (for inspection)
- Camera (for microscopic imaging)
- Timer

- Thickness Measurement Tool (Filmetrics F20 Reflectometer or 3D Profilometer)
- Refrigerator for storage of AZ nLOF 2020 Photoresist

- Wafer Tweezers or Wand (4")
- Wafer (3" or 4") Silicon w/ Polyimide, Conductive Metal, & Parylene
- Cleanroom Notebook
- Cleanroom Wipes
- Marker for wafer labeling
- Photoresist (Pick one or more)
  - AZ nLOF 2020 Photoresist (~2 mL per wafer)
  - AZ 4330 Photoresist (~2 mL per wafer)
  - Thick Photoresist (positive or negative, depends on masks created)
- 5" Soda Lime Glass mask plate (Clear for Mylar films or complete Mask)
- Blue Cleanroom Tape, Low-Tack or Medium-Tack (for Mylar masks)
- Developer Solution (Pick one or more)
  - AZ 300MIF Developer Solution (~100-200 mL per batch)
  - Accompanying Developer Solution for the Thick Photoresist)
- DI Water (~100-200 mL per batch)
- Waste bottles for all chemicals (~3)

## **10.3 Deposition Process**

#### 10.3.1 Photoresist Spinning

- The process for spinning photoresist is going to depend on the type of spinner system you are using, so these steps will serve as a guideline for what we do here at ASU. Since we typically use the ASU NanoFab for these steps of the process, I will be following the procedure I would use in that cleanroom.
  - a. Note: The mask aligner we have in our cleanroom does not currently have control of the Z-axis nor the Y-axis due to some broken micro-manipulators.
- 2. The ASU NanoFab has 2 Brewer Science CEE Coater systems for spinning use. One of them has a hot plate included and the other does not. I will be walking through the operation of the coater that includes the hot plate (#1) so that the controls are known. If you are using the other coater system (#2), there is a hot plate to be used next to the coater system and just has to be preset to the specified temperature.
- 3. Touch anywhere on the interface screen of the Brewer Science CEE Coater #1 to wake it up from its "Sleep" mode.
- 4. If the software is not running (Windows background), double-click on the CEE Software shortcut in the upper right-hand side of the background.
- 5. Touch the "Log In" button in the upper right-hand side of the software interface.
- 6. Use the password "recipe" for normal use (spinning and hot plate)

- a. Note: There is another password that can be used for higher functionality, including the ability to run the spinner with the lid up. This is useful for cleaning the coater once you have completed the spinning process. This password is "secret".
- 7. Once the password has been entered and accepted, click on the "Thermal" button to enter the controls of the hot plate.
  - a. Note: The only two button options here are "Spin" or "Thermal"
- 8. Touch the "Load Recipe" button near the top of the interface and select the recipe you would like to use.
  - a. When using the AZ nLOF 2020 resist, we require the use of a 110°C temperature for 60 seconds, so the "110C 60s" recipe works the best for us here.
- 9. Regardless of the current temperature of the hot plate, press the "Start Process" button to begin a cycle of the hot plate.
  - a. Note: This is just to get the hot plate temperature up to where we need it to go.
- 10. A pop-up window will appear if the temperature is outside of the setpoint's range asking if you would like to continue even though the temperature is out of range. Select "Yes" and allow the cycle to run.
- 11. While the temperature of the included hot plate is getting to its setpoint, you can begin to set the spinning system up. Lift the lid to the spinner chamber to allow for the tools to be installed.
  - a. Note: The process for the temperature to get up to setpoint will alarm after 1 minute since it completed the process it was running. Make sure to hit "OK" on the interface once this starts alarming. This may be after you have installed everything or during your installation process, so just be aware you will need to complete this before the annoying alarm destroys your sanity.
- 12. Grab a bowl from the top of the Solvent Hood (to the right of the spinner systems table) and place it inside the spinning chamber.
  - a. Note: There are 2 types of bowls to choose, 1 with a lip and 1 without a lip. If you are only doing 1-2 wafers, the bowl with a lip is best for easy cleaning after spinning. If you are processing more than 2 wafers (normal batch is 4 wafers), use the bowl without a lip so that everything can go down the drain once it reaches the bowl. The bowl without a lip likely requires the use of the "secret" password for the cleaning process.
  - b. Note: Make sure the entire bowl is flush with the chamber and is not sitting on the Stainless Steel rim surrounding the spinning chamber.
- 13. Grab the chuck toolbox and open it up (located underneath the spinning systems).
- 14. Select the corresponding wafer chuck and centering tool based on the size of wafers you are using.
  - a. Note: We typically use a 4" wafer, so the 4" centering tool and the 2.5" wafer chuck (requires screw) are what we typically use.
- 15. Install the wafer chuck with the post aligned with the slot in the wafer chuck. Screw this in using the allen wrench (red handle in the chuck toolbox)
  - a. Note: This is hard to describe in words, but you need to make sure the openings in the wafer chuck line up with the things sticking out of the spinner post. Not that this additional description actually helps, but it will make sense when you see the system, hopefully.
- 16. Place the centering tool next to the system for later use.
- 17. Select the "Run" tab on the bottom left-hand side of the interface.
  - a. Note: This step cannot be completed until the Thermal process has completed.

- 18. Select the "Spin" button from the center of the screen.
- 19. Press the "Load Recipe" button on the top left-hand side of the interface and load the recipe needed from the list.
  - a. When using the AZ nLOF 2020 photoresist, we typically use a 3,000 rpm spin speed and a 30 second duration. The "3000 30s KN" recipe includes a 500 rpm (5s) spreading step followed by the 3,000 rpm spin step (30s) and a 500 rpm (5s) cooldown step. This is the preferred recipe. You can save this off as another recipe name if you would like to have one you can always find. Be aware that this is a public system for anyone in the cleanroom to use, so whatever name you pick for a new recipe name would have to be unique and descriptive.
- 20. You can press the "Edit Recipe" button to check that the recipe is correct if you would like.
  - a. Note: Press the "Back" button on the lower right-hand side of the interface to return to the previous screen.
- 21. The system has now been set up and we can begin to process the wafers. Grab 2 cleanroom wipes and place them next to the spinning system.
  - a. Note: 1 of these cleanroom wipes will be used for cleaning the spinning system once you have completed your processing. The other cleanroom wipe will be used for the Mask Aligner system and for cooldown after the PEB (Post-Exposure Bake after the exposure in the Mask Aligner).
- 22. Grab the bottle of photoresist you are using and place it next to the spinning system, on the upper right edge of the cleanroom wipes.
  - a. Note: We store AZ nLOF 2020 in the refrigerator in ERC 320, so you should have placed this bottle inside of the toolbox you are taking into the cleanroom with you. The ASU NanoFab supplies resists such as AZ 3312, AZ 4330, and a couple others. They also supply HMDS if that is required for your processing. Anything else will have to be purchased when we run out and will be stored in our lab space.
- 23. Move the centering tool to the upper left edge of the cleanroom wipes next to the spinning system.
- 24. Grab the Wafer Box containing the wafers you will be processing and place it on the left side of the cleanroom wipes next to the spinning system.
- 25. Ensure the Wafer Tweezers are present next to the spinning system as well.
- 26. Open the bottle of photoresist and set the cap to the side.
- 27. Grab a "Large Pipette" from the bins between the spinning systems.
- 28. Squeeze the bulb of the pipette until you cannot squeeze it further.
- 29. Place the squeezed pipette into the bottle of photoresist and release the bulb once the tip has been fully submerged into the photoresist.
- 30. Open the lid to the spinning bowl.
- 31. Dispense the entirety of the pipette into the bowl in a circular fashion.
  - a. Note: We need to create a Solvent-rich environment for the photoresist to spin consistently every time, so this should be distributed throughout the entire bowl as evenly as possible.
  - b. Note: This is the first coating of two to ensure a complete environment.
- 32. Empty the entire pipette into the bowl, keep the bulb depressed, place it back into the bottle of photoresist, and release the bulb once the tip has been fully submerged into the photoresist.
- 33. Dispense the entirety of the pipette into the bowl in a circular fashion.

- a. Note: We need to create a Solvent-rich environment for the photoresist to spin consistently every time, so this should be distributed throughout the entire bowl as evenly as possible.
- b. Note: This is the second coating of two to ensure a complete environment.
- 34. Empty the entire pipette into the bowl, keep the bulb depressed, place it back into the bottle of photoresist, and release the bulb once the tip has been fully submerged into the photoresist.
- 35. Close the lid to the spinning chamber.
- 36. Steps 37-73 below should be repeated for every wafer in the batch you are trying to process.
- 37. Open the lid to the spinning chamber.
- 38. Grab the next wafer to be processed in your dominant hand.
- 39. Grab the centering tool in the other hand.
- 40. Place the centering tool around the wafer chuck such that its ridge is in contact with the bottom and side of the wafer chuck.
  - a. Note: Do not release the centering tool from its location until the vacuum has been established on the wafer.
- 41. Place the wafer onto the wafer chuck and push it against the centering tool with the edge of your tweezers.
  - a. Note: Maintain the same contact position with the centering tool during this time.
- 42. Using the tweezers hand, Press the "Hold" button on the interface.
- 43. This should illuminate the "Substrate Present" box in blue (upper right-hand side of the interface). Once that is illuminated in blue, you can remove the centering tool from its contact position on the wafer chuck.
- 44. Place the centering tool back on its place on the cleanroom wipes (upper left-hand side).
- 45. Press the "Start Centering" button on the bottom left-hand side of the interface.
- 46. Check to be sure the wafer is completely centered on the wafer chuck. If this is not the case, Press the "Release" button, remove the wafer from the wafer chuck, and repeat Steps 38-46 until the wafer is centered on the wafer chuck.
- 47. Grab the bottle of photoresist in one hand.
- 48. Grab the pipette from the bottle of photoresist in your dominant hand and pull it from the bottle such that the tip is above the photoresist level, but still within the opening of the bottle.
  - a. Note: The photoresist will be dripping from the pipette, so ensure these drops make it back into the bottle of photoresist.
- 49. Once the dripping has slowed to at least a couple seconds between drops, move the pipette from the bottle to the bowl in a swift (but cautious!) movement.
- 50. Once the dripping has slowed to at least a few seconds between drops, move the pipette to the center of the wafer and begin the dispensing process.
  - a. Note: The pipette should be held at approximately a 45° angle from vertical and the tip should be as close to the wafer as possible without touching the wafer or the photoresist.
- 51. To dispense the photoresist evenly and consistently, squeeze the bulb of the pipette with enough pressure that you have a steady and constant flow of photoresist coming out of the tip of the pipette.
- 52. Continue to dispense the photoresist until you have at least a half dollar-sized amount on the wafer or until you have a minimal amount of photoresist remaining in the pipette (and/or start seeing bubbles coming out of the pipette).

- 53. If any bubbles exist in the photoresist that was dispensed, now is the only time you have to get rid of them. Completely depress the bulb of the pipette (emptying any contents into the spinning bowl) and move the pipette to the surface of the photoresist, next to/on top of the bubble. Release the bulb slowly to get the bubble to go up into the pipette tip. Once the bubble has been sucked up into the pipette tip, stop releasing the bulb. Move the pipette from the wafer surface to the spinning bowl and squeeze the bubb completely to expel the bubble from the pipette. Repeat this step until all bubbles have been eliminated from the photoresist puddle.
- 54. Completely depress the bulb to the pipette (emptying any contents inside into the spinning bowl), keep it depressed, move it back to the bottle of photoresist, place the tip into the photoresist liquid, and release the bulb once the tip has gone entirely below the surface of the photoresist.
  - a. Note: DO NOT do this step if there are no more wafers to process. Instead, place the emptied pipette onto the cleanroom wipe for disposal later.
- 55. Place the bottle of photoresist back on its place on the cleanroom wipes (upper right-hand side).
- 56. Close the lid to the spinning chamber.
- 57. Ensure the "Lid Closed" and "Substrate Present" boxes on the interface are both illuminated in blue.
- 58. Press the "Start Process" button on the bottom left-hand side of the interface to begin the spinning process.
- 59. Wait for the process to complete. Once the process is complete, the system will alarm. Press the "OK" button when this alarm goes off.
- 60. After the alarm has been acknowledged, press the "Run" tab on the bottom left-hand side of the interface.
- 61. Select the "Thermal" option.
- 62. Ensure that the temperature is illuminated with a green background, denoting that the temperature is within its setpoint range.
- 63. Open the lid to the spinning chamber.
- 64. Grab the wafer with your Wafer Tweezers in your dominant hand and remove it from the spinning chamber.
- 65. Using your other hand, Press the "Start Process" button on the bottom left-hand side of the interface.
  - a. Note: The hot plate in this system has a 5 second step before it adds vacuum so that the wafer can be placed on the hot plate and adjusted to the center position before it adds a vacuum under the wafer and starts the 60 second timer.
- 66. Open the lid to the Hot Plate chamber with your free hand and place the wafer onto the hot plate such that the 2 standing posts are keeping it centered.
  - a. Note: This lid will not stay up, so you must keep it held with one of your hands while you are placing the wafer onto the hot plate.
- 67. Ensure the wafer achieves a vacuum (gets sucked down onto the hot plate) and close the lid to the Hot Plate chamber.
  - a. Note: If there is a previous wafer cooling down on the cleanroom wipe, remove it from the wipe and place it back into the Wafer Box.
- 68. Wait for the timer to get close to expiring (approximately 1-3 seconds remaining) and open the lid to the Hot Plate chamber.
- 69. The wafer should lift up off the hot plate for 5 seconds after the 60 second timer expires, so utilize this time to grab the wafer from the hot plate.

- 70. Place the wafer onto the cleanroom wipe below.
- 71. Close the lid to the Hot Plate chamber.
- 72. The system should alarm once the entire recipe has completed, so hit the "OK" button once this goes off to acknowledge the alarm.
- 73. Keep the wafer on the cleanroom wipe for at least 60 seconds for it to cool down before placing it back into the Wafer Box.
- 74. Repeat Steps 37-73 for all wafers in the batch.
- 75. Once all of the wafers have finished spinning and baking, you then begin the process of returning the system back to how you found it. While the last wafer is cooling down on the cleanroom wipe, you can prepare the system for cleaning. Do this by grabbing the Acetone and Isopropyl Alcohol (IPA) bottles from the General Waste Bin station (or the Solvent Hood).
- 76. Move the pipette from the cleanroom wipe to the Flammable/Solid Waste bin.
  - a. Note: if this still remains full inside the bottle of photoresist, empty its contents into the spinner's bowl completely and then place directly into the Flammable/Solid Waste bin.
- 77. Return the cap to the photoresist bottle and place it back where it came from.
  - a. Note: This should be the toolbox you came in with if it is AZ nLOF 2020, but it could be the dry box in the cleanroom if you were using one of the ASU NanoFab's provided photoresists.
- 78. Ensure the "secret" login information was used so that you have access to the Diagnostics page of the software.
  - a. Note: You can manually clean the spinning bowl and the system, but that just requires some cleanroom wipes and Acetone/IPA combination. I will outline the steps for cleaning when a whole batch was completed.
- 79. Click on the "Diag" tab at the bottom center of the interface.
- 80. Select the "Spin Diagnostics" option.
- 81. Press the spin speed and change it from 250 rpm to 1,000 rpm.
- 82. Press the ramp rate and change it from 1,000 rpm/s to 250 rpm/s.
- 83. Open the lid to the spinning chamber.
- 84. Grab a blank Silicon wafer (not part of a batch) with your dominant hand.
- 85. Grab the centering tool with your other hand.
- 86. Place the centering tool into its contact point with the wafer chuck.
- 87. Place the wafer onto the wafer chuck and push it against the centering tool with the edge of your wafer tweezers.
- 88. Without moving the centering tool from its location, use your tweezer hand to press the "Yes" button under the "Vacuum" section of the interface (center-bottom left-hand side).
- 89. Once you see a checkbox ("Wafer Present?") appear above the button you just pressed, you can remove the centering tool from the spinning chamber.
- 90. Ensure there are no wafers still cooling down on the cleanroom wipe before proceeding to the next step.
- 91. Spray Acetone onto the wafer currently on the chuck until the entire wafer has a coating of Acetone on it.
- 92. Press the "Start" button under the "Spin" settings of the interface to begin spinning.
- 93. Spray Acetone onto the center of the spinning wafer for 5 seconds.
- 94. Continue spraying Acetone onto the center of the spinning wafer and also spray IPA onto the center of the wafer for 5 seconds.

- 95. Stop spraying the Acetone onto the spinning wafer and continue spraying IPA onto the center of the spinning wafer for 5 seconds.
- 96. Stop spraying the IPA onto the spinning wafer and wait until all of the rainbow colored streaks disappear from the wafer.
- 97. Wait another 15 seconds after the rainbow colored streaks disappear.
- 98. Press the "Stop" button until the "Spin" setting of the interface to stop spinning.
- 99. Press the "No" button under the "Vacuum" section of the interface to release the vacuum on the wafer.
- 100. Grab the wafer off the wafer chuck and place it back into the Wafer Box.
- 101. Grab the allen wrench from the chuck toolbox.

is easy to find.

- 102. Unscrew the wafer chuck and remove it from the system, placing it back in the chuck toolbox.a. Note: Make sure you keep track of the screw and place it in a spot inside the toolbox that
- 103. Spray Acetone onto one of the cleanroom wipes (preferably the top one since it will need to be disposed of already).
- 104. Wipe the spinner's bowl with the Acetone-soaked cleanroom wipe. Be sure to clean the surface of the bowl, the backside of the bowl, the edges of the bowl, and all surfaces of the bowl.
- 105. Once the bowl has been thoroughly cleaned and looks as good as (or better than) it did when you first grabbed it, place it back above the Solvent Hood.
- 106. Spray more Acetone onto the same cleanroom wipe you were using to clean the bowl and wipe down the spinner chamber, the lid, and the Stainless Steel surfaces of the coating system.
- 107. Once the system has been thoroughly cleaned, dispose of the cleanroom wipe you were using to clean into the Flammable/Solid Waste bin.
- 108. If you had an additional pair of gloves on (I usually do if I'm doing photoresist or if I'll be cleaning a spinner system), take the extra layer of gloves off without getting the other gloves dirty and place the dirty gloves into the Flammable/Solid Waste disposal bin.
- 109. You are now ready for the Exposure stage of the Photoresist Deposition process (4.3.2).
  - a. Note: Be sure to stay in the photolithography bay of the cleanroom since white light would expose the photoresist you just completed and your pattern will not turn out as expected.

#### 10.3.2 Photoresist Exposure

- 1. In the ASU NanoFab, the OAI 808 Mask Aligner is the tool we use for patterning and UV exposure of the photoresists we use.
  - a. Note: We have a HTG Mask Aligner in the ERC 444 cleanroom, but due to the broken Z-axis controls, we cannot use the aligner for anything except gap exposure. The Y-axis controls are also broken, so any alignment besides the first layer (not requiring alignment) is impossible.
- 2. Since we use the AZ nLOF 2020 photoresist and it requires a post-exposure bake to cross-link the polymer chains, set up a hot plate near you for use at 110°C for 60 seconds
  - a. Note: I like to use the built-in hot plate of the Brewer Science CEE Coater #1, which I also like to use for spinning the photoresist on. If this Spinner and Hot Plate were used for the photoresist and have not been changed, you can use the same Thermal program you

used previously. If it is currently in use, simply find another hot plate to use and make sure you have a timer (cell phone/smart watch works well) ready to go.

- 3. Place a cleanroom wipe for wafer cooldown after the post-exposure bake. This can be placed either next to the hot plate or next to the Mask Aligner, either one works.
- 4. The OAI 808 Aligner requires training prior to use and uses a badge access to activate the vacuum lines. Scan your ASU ID on the "Login" badge portal and it should flash green lights when you have done so.
- 5. The interface box is located to the right of the machine. This should have been left on the screen that has "Mask Vacuum" in the bottom right-hand corner. If it is not on this screen, press the "Main Menu" button (upper left-hand side of the interface) and select "Manual Test" (middle left option).
- 6. There is a log book for the system on the rolling cart next to the machine. Fill in the information (name, date, start time, resist type, filter or no filter, calibrating gap or not calibrating gap, wafer size, wafer thickness, mask size, mask thickness, frontside alignment).
- 7. You will also need to record the lamp hours from the lamp's power box. This is located underneath the system on the left-hand side. Write this number into the log book.
- 8. The system now has to be prepped for your wafer and mask setup, so check what the current mask chuck and wafer chuck sizes are. If they do not match what you are working with, the following steps will dictate how to install the proper mask chuck and wafer chuck.
  - a. Note: Currently, we are using 4" wafers with a 5" mask plate. Typically, your mask plate is at least 1" larger than the wafer diameter.
- 9. Slide the wafer chuck out towards you.
- 10. Loosen the screws at the front and back of the wafer chuck (2 screws closest to you and 1 screw at the back of the wafer chuck).
- 11. Undo the vacuum line connections (3 of them) at the bottom/back right of the wafer chuck.
  - a. Note: They should read "BALL", "SUB", and "CONT"
- 12. Remove the wafer chuck by lifting it up off the posts.
- 13. Swap the previous wafer chuck with the correct wafer chuck (located on the top of the rack to your right against the wall).
  - a. Note: Be careful of the vacuum lines coming out of the vacuum chuck so that you do not pinch these. I typically will place the previous wafer chuck such that the vacuum lines are going through one of the slots in the wires of the rack and hanging beneath it.
- 14. Place the correct wafer chuck onto the posts the previous one was sitting on.
- 15. Screw the 2 screws closest to you first, but do not tighten completely (only tighten a few turns)
- 16. Screw the 1 screw furthest from you until you feel it contact the wafer chuck.
- 17. Finish screwing the 2 screws closest to you until you feel them contact the wafer chuck.
- 18. Tighten all of the screws, starting with the back one and finishing with the 2 front ones.
- 19. Install all 3 of the vacuum lines into their respective connections. The wafer chuck has now been installed.
  - a. Note: The vacuum lines will have a label ("CONT", "BALL", and "SUB") and the connection ports will have matching labels. Match the vacuum line to the matching connection port. These can get tangled, so I recommend starting from the back connection port and working your way to the front connection port.
- 20. Loosen the four spring-loaded screws on top of the mask chuck to uninstall it from its location on the stack.

- 21. Undo the vacuum line connection to the left of the mask chuck. (Follow the vacuum line from the mask chuck to find this connection)
- 22. Remove the mask chuck by sliding it off the rails until it is clear of the microscope/camera lenses (alignment scope system) and then lift it off the system once it is clear of the lenses.
- 23. Swap the previous mask chuck with the correct mask chuck (located on the bottom of the rack to your right against the wall).
- 24. Install the vacuum line into the port on the left of the mask chuck. The mask chuck has now been installed.
- 25. Lay the mask chuck upside down (you should see a few posts and a spring system for mask installment) on the rolling cart next to the system.
- 26. The system is now ready for you to install your mask. Place a cleanroom wipe next to your toolbox.
- 27. Grab your 5" clear mask plate from the mask holder/carrier and place it onto the cleanroom wipe.
  - a. Note: If the wrong mask is currently installed, remove the blue tape holding it to the mask plate and place the tape on the corners of the mask plate for later use.
  - b. Note: If the correct mask is currently installed, skip to Step 29.
- 28. Grab the correct mask from your toolbox and install it such that the ink is facing towards you. Secure it to the mask plate with the blue cleanroom tape your removed from the previous mask.
  - a. Note: Typically we use a right-read down mask style, meaning that the ink is on the side that text appears backwards on. This is why I will always include some kind of text in the mask design. (The text should read normally when it is installed into the system and about to come into contact with the wafer)
- 29. Place the mask (with mask and blue cleanroom tape on the side facing towards you) onto the mask chuck and push it against the posts on the corners of the mask chuck.
- 30. There should be a spring-loaded post at one end of the mask chuck. Press this lever/button such that the post comes into contact with the mask plate.
- 31. Press the "Mask Vacuum" button on the interface. Ensure that the vacuum level (gauge on the far left of the machine) is at least to -10 inHg (or lower).
  - a. Note: If the mask vacuum is not high enough, try pressing down on the edges of the mask plate and/or reseating the mask.
- 32. Press the "Level Screen" button (top left-hand side of the interface) to prepare for the next step on the interface.
- 33. Carefully flip the mask chuck such that the mask is on the bottom side and place it onto the rails you first uninstalled it from.
- 34. Slide the mask chuck onto the rails and align the spring-loaded screws with the holes in the structure. Tighten these screws once they are in position.
  - a. Note: Ensure the positioning is correct by checking that the vacuum line is coming out towards you and is on the left side of the mask chuck (closest to the connection port)
- 35. The following steps (36-58) should be repeated for every wafer in the batch that need to get exposed.
- 36. Slide the wafer chuck out towards you.
- 37. Grab the next wafer to be processed from its Wafer Box and place it on the wafer chuck.
- 38. Align the wafer on the wafer chuck by making sure the flat is pushed against the two pins closest to you and that the edge of the wafer is pushed against the pin on the right edge of the wafer.

- 39. Press the "Substrate Vacuum" button (upper-middle left-hand side of the interface). Ensure that the vacuum level (gauge on the far left of the machine is at least to -10 inHg (or lower).
  - a. Note: If the substrate vacuum is not high enough, try pressing down on the edges of the wafer and/or reseating the wafer. Also make sure the wafer is not sitting on top of the pins you pushed it up against.
- 40. Push the wafer chuck away from you and into the system's stack.
- 41. Check the Exposure Time (upper-center right-hand side of the interface) and if it isn't correct, Press the "Main Menu" button (upper left-hand side of the interface).
- 42. Press the "Process Settings" button (upper right-hand side of the interface).
- 43. Press the "Exposure Time" box (upper right-hand side of the interface).
- 44. Change the time to the required exposure time.
  - a. Note: We use 6.6 seconds for our system since it is set to ~10 mW/cm<sup>2</sup> and we require 66 mJ/cm<sup>2</sup> of energy.
- 45. Press the "Main Menu" button (upper left-hand side of the interface).
- 46. Press the "Level Screen" button (center right-hand side of the interface).
- 47. Press the "Level" button (bottom left-hand side of the interface) and wait for the wafer chuck to get up to the height of the mask.
- 48. Calibrate the gap between the wafer chuck and the mask FOR THE FIRST WAFER ONLY. (Skip if not the first wafer)
  - a. Press the "Cal Gap" button (lower left-hand side of the interface).
  - b. Grab the caliper, turn it on, and place it onto the mask chuck such that its pin is in the center of the mask and the sides are resting on the top of the mask chuck.
  - c. Change the movement increment to 10 µm by pressing on the increment number.
  - d. Lower the wafer chuck (by 10 µm increments) until the reading on the caliper is no longer changing AND keep going until it is at least at 100 µm past that point to make sure.
  - e. Press the "Origin" button on the caliper to return the reading to 0.000 mm.
  - f. Raise the wafer chuck slowly (by 10 μm increments) until the caliper changes to any value besides 0.000 mm.
  - g. Change the movement increment to 3 µm by pressing on the 10 µm number.
  - h. Raise the wafer chuck slowly (by 3 µm increments) until the caliper reads -0.004 mm.
  - i. Record the number in the "Gap" reading in the log book.
  - j. Press the "Zero Gap" button (lower right-hand side of the interface)
  - k. Press the "Run Screen" button (upper left-hand side of the interface) to return to the Run Screen.
- 49. Line up the wafer to the mask by using the micromanipulators at the base of the wafer chuck. Make sure that the mask is in the correct location on the wafer.
- 50. Press the "Contact" button on the interface (upper-center left-hand side of the interface) and wait a few seconds for that to activate.
- 51. Press the "Hard Contact" button on the interface (upper-center right-hand side of the interface) and wait a few seconds for that to activate.
- 52. Press the "Cycle" button on the interface (lower right-hand side of the interface). The system will now move the UV lamp head over the wafer and mask, open the shutter for the specified duration, and return to its original position. It will also lower the wafer chuck back to its loading location and switch the interface to the "Level Screen" where you will see the "Substrate Vacuum" and "Level" buttons.

- 53. Slide the wafer chuck out towards you.
- 54. Press the "Substrate Vacuum" button (upper-center left-hand side of the interface) to release the vacuum from the wafer.
- 55. Grab the wafer from the wafer chuck and take it over the the Hot Plate which should be previously set up for you.
- 56. Place the wafer on the hot plate and start the timer (or press "Start Process" if you are using CEE Coater #1)
- 57. Once the timer has completed, remove the wafer from the hot plate and place it on a cleanroom wipe. This can be located either next to the hot plate or at the Mask Aligner. Leave the wafer on the cleanroom wipe for at least 60 seconds.
- 58. Remove the wafer from the cleanroom wipe and place it back into its slot in the Wafer Box.
- 59. Repeat Steps 36-58 for each wafer in the batch that you are working with.
- 60. Once you have completed processing on all wafers in the batch, push the wafer chuck away from you and back into the system (without a wafer on it).
- 61. Unscrew the 4 spring-loaded screws in the mask chuck.
- 62. Slide the mask chuck out on the rails until it is past the microscope/camera lenses.
- 63. Lift the mask chuck off the rails and flip the mask chuck such that the mask is facing toward the ceiling.
- 64. Place the mask chuck on the rolling cart next to the machine.
- 65. Press the "Main Menu" button (upper left-hand side of the interface).
- 66. Press the "Manual Test" button (center left-hand side of the interface).
- 67. Press the "Mask Vacuum" button (bottom right-hand side of the interface).
- 68. Pull the spring-loaded post (on the mask chuck) away from the mask.
- 69. Remove the mask from the mask chuck and place it back into the mask holder/carrier (mask and tape side facing down). Close the mask holder/carrier when it is in place and return this to the toolbox you brought into the cleanroom with you.
- 70. Flip the mask chuck to its normal orientation, place it back onto the rails it was on, and slide it all the way back to its location.
- 71. Tighten at least 2 of the spring-loaded screws down.
  - a. Note: I usually do either 2 opposite corners or all 4.
- 72. Press your ASU ID against the badge portal that says "Logout". It should flash green and then both the "Login" and "Logout" portals should be red.
- 73. Write the total time you exposed the wafers for (adding up all of the times you used for the batch you just completed) and write the end time into the log book.
- 74. You are now ready for the Developing process.

#### 10.3.3 Photoresist Developing

- Our AZ nLOF 2020 photoresist has a recommended Developer solution of AZ 300 MIF. This
  chemical is available in both the ASU NanoFab and in the ERC 444 cleanroom. If you are doing
  your photoresist processing in the ASU NanoFab, it is recommended to just do this step in the
  NanoFab as well.
- 2. Find a clean working bench at a Base Hood to prepare your work station for this process at.
- 3. Place two cleanroom wipes on the bench, with one near a sink.

- 4. Grab a Pyrex dish from the supply rack (some say "300 MIF" on them and those are preferred to be used) and place it on the cleanroom wipe furthest from the sink.
  - a. Note: The other cleanroom wipe will be used for drying the wafers after the processing.
- 5. Place a timer next to the dish for 300 MIF. (The Stopwatch function will be needed)
  - a. Note: The AZ nLOF 2020 resist typically takes about 30 seconds for complete development.
- 6. Place your Wafer Box containing wafers needing to go through the developing process inside the Hood, next the the dish for 300 MIF.
- 7. Place your wafer tweezers or wand next to the Wafer Box.
- 8. Don the proper PPE required for chemical handling (apron, base gloves, & face shield).
  - a. Note: Technically this is only required when handling the chemicals, so they can be removed once you have put the 300 MIF bottle back in its storage location and have the Hazardous Waste bottle for 300 MIF (and the funnel) ready to go at the fume hood.
- 9. If you are more than a couple steps away from the storage location of the 300 MIF, grab a chemical transport container.
- 10. Grab the 300 MIF bottle from its storage location. (Place this into the chemical transport container if that is required here)
- 11. Bring the bottle of 300 MIF to the Base Hood you will be operating out of.
- 12. Open the cap to the bottle and pour 300 MIF into the dish until the chemical level is at least 1/2" deep. (This should be approximately 100-200 mL, depending on the size of the dish)
- 13. Return the cap to the bottle of 300 MIF.
- 14. Return the 300 MIF bottle to its storage location. (Use the chemical transport container if necessary).
- 15. Make sure there is a Hazardous Waste bottle for 300 MIF at the Base Hood as well. If this is not at the hood already, grab it from its storage location (using the chemical transport container if necessary) and transport it to the hood).
- 16. Make sure there is a funnel at the hood for ease of pouring into the Hazardous Waste bottle when completed.
  - a. Note: If you would like to remove your apron, face shield, and base gloves, you may do so at this point.
- 17. The ASU NanoFab has a sink and a drain attached to it and can be easily used to simply rinse the wafers once they are done developing. If you are in ERC 444 (or at Mayo Clinic), a bath setup will likely be needed for the water rinse step. For this bath setup, you should fill a dish (approximately the same size as the one for the 300 MIF solution) up to the same 1/2" level you filled the 300 MIF dish up to (~100-200 mL depending on the dish). This will also require an additional cleanroom wipe.
- 18. The following steps (19-39) should be repeated for each wafer in the batch that require developing.
- 19. Start the water flow from the sink if you are using the sink for the rinse and not a bath.
- 20. Grab the next wafer to be processed from the Wafer Box in your dominant hand.
- 21. In your other hand, grab the timer and prepare to begin the Stopwatch.
- 22. Simultaneously, begin the Stopwatch as you place the wafer into the 300 MIF solution. Make sure the entire wafer gets submerged into the 300 MIF solution.

- 23. Using the tip of your tweezers (and holding vertically), grab the side of the wafer by the major flat (by pushing against the wafer) and slowly move the wafer up and down in the solution. Be sure to not let the wafer breach the surface of the solution.
  - a. Note: This is performed to agitate and create movement in the solution so that the chemical solution can access all the photoresist on the wafer. This motion can be replaced if there is a stirrer or other similar device to create this movement.
- 24. While the wafer is in the developer solution, you should be able to see the photoresist coming off to form the pattern that you created from the Mask Aligner.
- 25. Once the Stopwatch reaches 27 seconds, pull the wafer from the solution to check if it has completed its processing.
- 26. If the wafer has completed its processing (and you can see the pattern you used at the Mask Aligner), move the wafer to the water stream coming from the sink. If the wafer needs to continue processing, place it back into the solution and repeat Steps 23-26 until completed.
  - a. Note: If using the water bath instead of the sink, place the wafer into the water bath.
- 27. Rinse the front of the wafer (pattern) by running it across the stream of water a few times.
- 28. Rinse the back of the wafer (no pattern, label) by running it across the stream of water a few times.
- 29. Rinse the front of the wafer (pattern) again by running it across the stream of water a few times.
- 30. Place the wafer onto the cleanroom wipe dedicated to drying the wafers.
- 31. Stop the flow of water coming from the sink.
- 32. Grab the Nitrogen air gun and dry the wafer starting in the center.
- 33. Rotate your wrist (NOT your arm/elbow) and push the liquid towards the edges of the wafer until there is no more water on the front of the wafer.
- 34. Dry off your wafer tweezers away from your wafer (so that no liquid splashes onto your dried wafer).
- 35. Pick up your wafer from the cleanroom wipe and place it vertically back on the cleanroom wipe so that you can dry the back side of the wafer.
- 36. Dry the back side of the wafer in the same fashion you dried the front (start in the center and rotate your wrist to push the liquid to the edges of the wafer). The wafer does not have to be exactly vertical, but it just cannot be touching any of the faces (front or back) to the cleanroom wipe.
- 37. Lift the wafer off the cleanroom wipe and pass the Nitrogen air gun across the edge of the wafer (back and forth) across the entire wafer (rotating your wrist to access the rest of the wafer).
  - a. Note: This is hard to put in words, but basically you are just pushing any liquid from the edge of the wafer onto one of the surfaces (front or back) to be dried again.
- 38. Face the front of the wafer towards you and dry off any liquid that got pushed back onto the front of the wafer from the edge dry.
- 39. Place the completed wafer into the Wafer Box for later inspection.
- 40. Repeat Steps 19-39 for each wafer in the batch that you are completing through this stage of the process.
- 41. Once all wafers in the batch have completed their Development process, you can begin the teardown of the station. Start by moving the timer, Wafer Box, and wafer tweezers out of the way and onto another table top.
- 42. Don the PPE for Base chemical handling if you do not have it on anymore (face shield, apron, and base gloves).

- 43. Move the Hazardous Waste bottle for 300 MIF to a location in which you can pour the contents of your dish into it.
- 44. Remove the cap to the waste bottle and place the funnel into the bottle.
- 45. Carefully pour the contents of the dish of 300 MIF into the waste bottle through the funnel.
- 46. Once all the contents have been emptied into the waste bottle, spray/add water to the dish until the water is at least three-quarters as high as the chemical solution was.
- 47. Swirl the water around the dish so that the water can remove any residue 300 MIF solution from the dish.
- 48. Empty the dish into the waste bottle.
- 49. Spray/Add a little bit of water to the dish, swirl it around, and empty it into the waste bottle.
- 50. Spray/Add at least 100 mL of water to the dish, swirl it around, and empty it into the sink (down the drain).
- 51. Repeat Step 50 for rinsing the 300 MIF dish (Completed twice total).
- 52. Using the cleanroom wipe that the 300 MIF dish was sitting on, dry out the dish you just rinsed with water. Place this cleanroom wipe in the "Flammable/Solid Waste" bin.
- 53. Place this dish to the side for returning to its storage location later.
- 54. If you were using a dish for a water bath, swirl the contents around and empty it into the waste bottle for 300 MIF.
- 55. Spray/Add a little more water to the water dish, swirl it around, and empty it into the waste bottle for 300 MIF.
- 56. Spray/Add at least 100 mL of water to the dish, swirl it around, and empty it into the sink (down the drain).
- 57. Repeat Step 56 for rinsing the water dish (Completed twice total).
- 58. Using the cleanroom wipe that the water dish was sitting on, dry out the dish you just rinsed with water. Place this cleanroom wipe in the "Flammable/Solid Waste" bin.
- 59. Place this dish to the side for returning to its storage location later.
- 60. Tap the funnel on the side of the waste bottle a few times to get some last remaining drops off it.
- 61. Screw the cap back onto the waste bottle.
- 62. Move the funnel back to its original location.
- 63. Move the waste bottle to its original location (using the chemical transport container if necessary).
- 64. Remove the PPE you donned for the Developer chemical handling and place it back where you got it from.
- 65. Return the dishes back to their storage locations.
- 66. Make sure the water is not running or dripping from the faucet.
- 67. Using the cleanroom wipe you used for drying the wafers, wipe down the surface area of the hood that you just used for the processing. Grab more cleanroom wipes if necessary. Ensure that the fume hood surface looks as good (if not better) than when you started using it. Place any used cleanroom wipes in the "Flammable/Solid Waste" bin.
- 68. You have completed the Development stage of the process and your wafers are ready for inspection.

#### 10.3.4 Inspection

- 1. There are three methods of inspection we use to measure the quality of photoresist we get after spinning, exposure, and developing.
  - a. The first is a visual inspection using our eyes.
  - b. The second is a visual inspection using a microscope.
  - c. The third is a metric inspection using a measurement tool to get the thickness of the layer.
- 2. As a first inspection, look at the wafer and ensure you have gotten the correct pattern to what you were setting out to get from the photoresist/mask combination. Also inspect for defects like bubbles, dirt, debris, and any voids in the photoresist material. The color combination is also an effective tool to ensure there are not too many striations in the colors, meaning that something didn't quite come out as planned. Luckily, photoresist can always be reworked, so this entire process can be repeated if necessary.
- 3. The second inspection involves using a microscope to inspect any potential defects found as well as to check the correct exposure energy and developing time. Check the two squares used for alignment for checking for the correct exposure energy and development times, based on the connection at the corners. (Bridge versus gap). Using the microscope can also inspect the wafer for any mask issues that you did not already notice.
- 4. The third inspection is where we measure the thickness of the photoresist layer we just deposited onto the wafer. At ASU, we use a Reflectometer in the form of a Filmetrics F40 system (under yellow lights) or a Filmetrics F20 system (under white lights). The following processing steps will follow the technique we require for that process.
  - a. Note: We can program a new material into the Filmetrics Software if needed. A tutorial is being written about how to go about doing that. PI-2611 (soft-baked & cured), Parylene, AZ nLOF 2020, & AZ 15nXT are all present on the F20, soon to be added to the F40.
- 5. Ensure the PC at the F40 station is operational and move the mouse around if the monitor is blank.
- 6. Run a baseline test by grabbing the blank "Baseline" Silicon wafer with your Wafer Tweezers or Wand and placing it underneath the microscope of the system.
- 7. Once the wafer has been placed underneath the microscope, find an edge on the wafer to focus the optics onto.
- 8. Click "Baseline" on the F20 software and then click "OK".
- 9. The software will now ask you to tilt the object at 10°-80° to cancel out the background light it might see. Grab the Container the blank Silicon wafer was sitting on the place it next to the beam spot. Then grab the blank Silicon wafer and set it onto the container such that the flat part is sitting on the base while the curved part is elevated and resting partially on the container. Click "OK" in the software to grab the measurement.
  - a. Note: This is tricky because this system uses a microscope to perform the analysis. Be careful around the optics to not damage them. It may make it easier to drop the stage by a set amount to be able to run this test.
- 10. Grab one of the other wafers in the calibration stack and check the system to make sure it is reading correctly.
- 11. The software is now ready for processing, so start by loading the correct recipe.
  - a. Note: Load "AZ nLOF" from "Photoresists".

- 12. Once the recipe is selected, you will now have to select the correct nominal thickness. Click "Edit Recipe" to change the nominal thickness as well as the measurement type (Angstroms, nanometers, or micrometers).
- 13. The following Steps (14-19) should be repeated for every wafer that needs to be tested for thickness.
- 14. Place the wafer that needs to be inspected onto the microscope such that the center of the wafer is directly underneath the beam of the system. Focus the microscope onto a feature on the surface of the wafer.
- 15. Click the "Measure" button on the software to grab the measurement. Write this measurement into a table in your Cleanroom Notebook as the "Center" location.
- 16. Move the wafer (using the stage manipulator) to the right such that the left middle of the wafer is now underneath the microscope lens.
  - a. Note: For reference, I consider the major flat to be the bottom of the wafer and the opposite side to be the top of the wafer. This makes everything easier to think about in my mind. Orientation is a personal choice and does not matter, as long as it is consistent.
- 17. Click the "Measure" button on the software OR press the "Enter" key on the keyboard. Write this measurement into a table in your Cleanroom Notebook as the "Left Middle" location.
- 18. Move the wafer to the right such that the left side is now underneath the microscope lens.
  - a. Note: You do not want to capture the far edges of the wafer as this is where the edge bead has formed. Make sure this measurement is not on the edge bead section.
- 19. Click the "Measure" button on the software OR press the "Enter" key on the keyboard. Write this measurement into a table in your Cleanroom Notebook as the "Left" location.
- 20. Continue the previous steps to get the "Right Middle", "Right", "Top Middle", "Top", "Bottom Middle", and "Bottom" measurements. Write each measurement into the table in your Cleanroom Notebook.
  - a. Note: These specific locations are important because they ensure consistency and they also cover enough sections of the wafer that any major defects can be determined simply by thickness variance. Gathering 9 points of data also helps in statistical verification as well as getting a surface profile of the wafer every time a process is completed.
- 21. Using your Wafer Tweezers or Wand, scoot the wafer off the base platform and load it back into the Wafer Box it came from.
- 22. Repeat Steps 14-21 for every wafer in the batch that is needing to be tested for thickness.
- 23. Once all wafers are completed, take the data from the Cleanroom Notebook you wrote it on and transfer it to an Excel sheet (or Google Sheet) such that it can be analyzed for averages, standard deviations, and other statistical calculations. Saving all data into a common location can help with product tracing throughout the device's fabrication as well as ensuring that the processing steps remain consistent.
  - a. Note: Examples of the tables used in the Cleanroom Notebook as well as the Excel tables can be provided. This can all be recorded directly into an Excel Sheet or Google Sheet instead of onto a cleanroom notebook. I have to write the measurements onto a cleanroom notebook just because that is the nature of the lab in which the measurement tool is located in and I have not implemented a Google Sheet for this yet.

# 11 Insulation Etching for Electrode Contacts

## **11.1 Equipment Needed**

- RIE Machine (preferred) or Plasma Cleaner (acceptable)
- Quartz ring (>4" diameter) for wafer containment
- Thickness Measurement Tool (Filmetrics F20 Reflectometer or 3D Profilometer)
- Microscope (for inspection)
- Camera (for microscopic imaging)

#### **11.2 Materials Needed**

- Wafer Tweezers or Wand (4")
- Wafer (3" or 4") Silicon w/ Polyimide, Conductive Metal, Parylene, & Photoresist
- Cleanroom Notebook
- Cleanroom Wipes
- Marker for wafer labeling

## **11.3 Etching Process**

#### 11.3.1 Reactive Ion Etching Machine

1.

#### 11.3.2 Inspection

1.

# 12 Photoresist Removal after Electrode Contacts Etching

## **12.1 Equipment Needed**

• Pyrex dishes (>4" diameter) (Qty: 3)

- Fume Hood
- Sonicator OR Nitrogen bubblers
- Timer
- Thickness Measurement Tool (Filmetrics F20 Reflectometer or 3D Profilometer)
- Microscope (for inspection)
- Camera (for microscopic imaging)

- Wafer Tweezers or Wand (4")
- Wafer (3" or 4") Silicon w/ Polyimide, Conductive Metal, Parylene, & Photoresist
- Cleanroom Notebook
- Cleanroom Wipes
- Marker for wafer labeling
- Photoresist Stripper (Pick one or more)
  - AZ 400T Stripper (~100-200 mL per batch)
  - Acetone (~100-200 mL per batch)
  - Accompanying Stripper for Thick Photoresist (~100-200 mL per batch)
- Isopropyl Alcohol (~100-200 mL per batch)
- DI Water (~100-200 mL per batch)
- Waste bottles for all chemicals (~3)

## 12.3 Removal Process

#### 12.3.1 Photoresist Stripper & Cleaning Baths

1.

#### 12.3.2 Inspection

1.

## **13 Electrode Contact Testing**

## **13.1 Equipment Needed**

- Micro Probe Station (at least 4 micromanipulators)
- Multimeter Station OR Data Acquisition Device

- Microscope
- Camera (for microscopic imaging)

- Wafer Tweezers or Wand (4")
- Wafer (3" or 4") Silicon w/ Polyimide, Conductive Metal, & Parylene
- Cleanroom Notebook
- Cleanroom Wipes
- Marker for wafer labeling
- Spare probe tips (Tungsten, Gold, or Stainless Steel likely)
- Calibration samples (Aluminum or Titanium or equivalent to ensure system is within range)

## **13.3 Testing Process**

- 13.3.1 4-Point Probe Testing (Stationary)
  - 1.
- 13.3.2 Probe Station Testing (Micromanipulator)
  - 1.

#### 13.3.3 Inspection

1.

# 14 Photoresist Deposition for Device Removal

## 14.1 Equipment Needed

- Spinner
- Mask Aligner (i-line UV) w/ Z-axis calibration (~66 mJ/cm<sup>2</sup>)
- Fume Hood (for developer)

- Pyrex dish (>4" diameter) (Qty: 2)
- Hot Plate or Oven (110C)
- Nitrogen air gun (for wafer drying)
- Microscope (for inspection)
- Camera (for microscopic imaging)
- Timer
- Thickness Measurement Tool (Filmetrics F20 Reflectometer or 3D Profilometer)
- Refrigerator for storage of AZ nLOF 2020 Photoresist

- Wafer Tweezers or Wand (4")
- Wafer (3" or 4") Silicon w/ Polyimide, Conductive Metal, & Parylene
- Cleanroom Notebook
- Cleanroom Wipes
- Marker for wafer labeling
- Photoresist (Pick one or more)
  - AZ nLOF 2020 Photoresist (~2 mL per wafer)
  - AZ 4330 Photoresist (~2 mL per wafer)
  - Thick Photoresist positive or negative (depends on mask created)
- 5" Soda Lime Glass mask plate (Clear for Mylar films or complete Mask)
- Blue Cleanroom Tape, Low-Tack or Medium-Tack (for Mylar masks)
- Developer Solution (Pick one or more)
  - AZ 300MIF Developer Solution (~100-200 mL per batch)
  - Accompanying Developer Solution for the Thick negative photoresist)
- DI Water (~100-200 mL per batch)
- Waste bottles for all chemicals (~3)

## **14.3 Deposition Process**

#### 14.3.1 Photoresist Spinning

- 1. The process for spinning photoresist is going to depend on the type of spinner system you are using, so these steps will serve as a guideline for what we do here at ASU. Since we typically use the ASU NanoFab for these steps of the process, I will be following the procedure I would use in that cleanroom.
  - a. Note: The mask aligner we have in our cleanroom does not currently have control of the Z-axis nor the Y-axis due to some broken micro-manipulators.
- 2. The ASU NanoFab has 2 Brewer Science CEE Coater systems for spinning use. One of them has a hot plate included and the other does not. I will be walking through the operation of the coater that includes the hot plate (#1) so that the controls are known. If you are using the other coater system (#2), there is a hot plate to be used next to the coater system and just has to be preset to the specified temperature.

- 3. Touch anywhere on the interface screen of the Brewer Science CEE Coater #1 to wake it up from its "Sleep" mode.
- 4. If the software is not running (Windows background), double-click on the CEE Software shortcut in the upper right-hand side of the background.
- 5. Touch the "Log In" button in the upper right-hand side of the software interface.
- 6. Use the password "recipe" for normal use (spinning and hot plate)
  - a. Note: There is another password that can be used for higher functionality, including the ability to run the spinner with the lid up. This is useful for cleaning the coater once you have completed the spinning process. This password is "secret".
- 7. Once the password has been entered and accepted, click on the "Thermal" button to enter the controls of the hot plate.
  - a. Note: The only two button options here are "Spin" or "Thermal"
- 8. Touch the "Load Recipe" button near the top of the interface and select the recipe you would like to use.
  - a. When using the AZ nLOF 2020 resist, we require the use of a 110°C temperature for 60 seconds, so the "110C 60s" recipe works the best for us here.
- 9. Regardless of the current temperature of the hot plate, press the "Start Process" button to begin a cycle of the hot plate.
  - a. Note: This is just to get the hot plate temperature up to where we need it to go.
- 10. A pop-up window will appear if the temperature is outside of the setpoint's range asking if you would like to continue even though the temperature is out of range. Select "Yes" and allow the cycle to run.
- 11. While the temperature of the included hot plate is getting to its setpoint, you can begin to set the spinning system up. Lift the lid to the spinner chamber to allow for the tools to be installed.
  - a. Note: The process for the temperature to get up to setpoint will alarm after 1 minute since it completed the process it was running. Make sure to hit "OK" on the interface once this starts alarming. This may be after you have installed everything or during your installation process, so just be aware you will need to complete this before the annoying alarm destroys your sanity.
- 12. Grab a bowl from the top of the Solvent Hood (to the right of the spinner systems table) and place it inside the spinning chamber.
  - a. Note: There are 2 types of bowls to choose, 1 with a lip and 1 without a lip. If you are only doing 1-2 wafers, the bowl with a lip is best for easy cleaning after spinning. If you are processing more than 2 wafers (normal batch is 4 wafers), use the bowl without a lip so that everything can go down the drain once it reaches the bowl. The bowl without a lip likely requires the use of the "secret" password for the cleaning process.
  - b. Note: Make sure the entire bowl is flush with the chamber and is not sitting on the Stainless Steel rim surrounding the spinning chamber.
- 13. Grab the chuck toolbox and open it up (located underneath the spinning systems).
- 14. Select the corresponding wafer chuck and centering tool based on the size of wafers you are using.
  - a. Note: We typically use a 4" wafer, so the 4" centering tool and the 2.5" wafer chuck (requires screw) are what we typically use.
- 15. Install the wafer chuck with the post aligned with the slot in the wafer chuck. Screw this in using the allen wrench (red handle in the chuck toolbox)

- a. Note: This is hard to describe in words, but you need to make sure the openings in the wafer chuck line up with the things sticking out of the spinner post. Not that this additional description actually helps, but it will make sense when you see the system, hopefully.
- 16. Place the centering tool next to the system for later use.
- 17. Select the "Run" tab on the bottom left-hand side of the interface.
  - a. Note: This step cannot be completed until the Thermal process has completed.
- 18. Select the "Spin" button from the center of the screen.
- 19. Press the "Load Recipe" button on the top left-hand side of the interface and load the recipe needed from the list.
  - a. When using the AZ nLOF 2020 photoresist, we typically use a 3,000 rpm spin speed and a 30 second duration. The "3000 30s KN" recipe includes a 500 rpm (5s) spreading step followed by the 3,000 rpm spin step (30s) and a 500 rpm (5s) cooldown step. This is the preferred recipe. You can save this off as another recipe name if you would like to have one you can always find. Be aware that this is a public system for anyone in the cleanroom to use, so whatever name you pick for a new recipe name would have to be unique and descriptive.
- 20. You can press the "Edit Recipe" button to check that the recipe is correct if you would like.
  - a. Note: Press the "Back" button on the lower right-hand side of the interface to return to the previous screen.
- 21. The system has now been set up and we can begin to process the wafers. Grab 2 cleanroom wipes and place them next to the spinning system.
  - a. Note: 1 of these cleanroom wipes will be used for cleaning the spinning system once you have completed your processing. The other cleanroom wipe will be used for the Mask Aligner system and for cooldown after the PEB (Post-Exposure Bake after the exposure in the Mask Aligner).
- 22. Grab the bottle of photoresist you are using and place it next to the spinning system, on the upper right edge of the cleanroom wipes.
  - a. Note: We store AZ nLOF 2020 in the refrigerator in ERC 320, so you should have placed this bottle inside of the toolbox you are taking into the cleanroom with you. The ASU NanoFab supplies resists such as AZ 3312, AZ 4330, and a couple others. They also supply HMDS if that is required for your processing. Anything else will have to be purchased when we run out and will be stored in our lab space.
- 23. Move the centering tool to the upper left edge of the cleanroom wipes next to the spinning system.
- 24. Grab the Wafer Box containing the wafers you will be processing and place it on the left side of the cleanroom wipes next to the spinning system.
- 25. Ensure the Wafer Tweezers are present next to the spinning system as well.
- 26. Open the bottle of photoresist and set the cap to the side.
- 27. Grab a "Large Pipette" from the bins between the spinning systems.
- 28. Squeeze the bulb of the pipette until you cannot squeeze it further.
- 29. Place the squeezed pipette into the bottle of photoresist and release the bulb once the tip has been fully submerged into the photoresist.
- 30. Open the lid to the spinning bowl.
- 31. Dispense the entirety of the pipette into the bowl in a circular fashion.

- a. Note: We need to create a Solvent-rich environment for the photoresist to spin consistently every time, so this should be distributed throughout the entire bowl as evenly as possible.
- b. Note: This is the first coating of two to ensure a complete environment.
- 32. Empty the entire pipette into the bowl, keep the bulb depressed, place it back into the bottle of photoresist, and release the bulb once the tip has been fully submerged into the photoresist.
- 33. Dispense the entirety of the pipette into the bowl in a circular fashion.
  - a. Note: We need to create a Solvent-rich environment for the photoresist to spin consistently every time, so this should be distributed throughout the entire bowl as evenly as possible.
  - b. Note: This is the second coating of two to ensure a complete environment.
- 34. Empty the entire pipette into the bowl, keep the bulb depressed, place it back into the bottle of photoresist, and release the bulb once the tip has been fully submerged into the photoresist.
- 35. Close the lid to the spinning chamber.
- 36. Steps 37-73 below should be repeated for every wafer in the batch you are trying to process.
- 37. Open the lid to the spinning chamber.
- 38. Grab the next wafer to be processed in your dominant hand.
- 39. Grab the centering tool in the other hand.
- 40. Place the centering tool around the wafer chuck such that its ridge is in contact with the bottom and side of the wafer chuck.
  - a. Note: Do not release the centering tool from its location until the vacuum has been established on the wafer.
- 41. Place the wafer onto the wafer chuck and push it against the centering tool with the edge of your tweezers.
  - a. Note: Maintain the same contact position with the centering tool during this time.
- 42. Using the tweezers hand, Press the "Hold" button on the interface.
- 43. This should illuminate the "Substrate Present" box in blue (upper right-hand side of the interface). Once that is illuminated in blue, you can remove the centering tool from its contact position on the wafer chuck.
- 44. Place the centering tool back on its place on the cleanroom wipes (upper left-hand side).
- 45. Press the "Start Centering" button on the bottom left-hand side of the interface.
- 46. Check to be sure the wafer is completely centered on the wafer chuck. If this is not the case, Press the "Release" button, remove the wafer from the wafer chuck, and repeat Steps 38-46 until the wafer is centered on the wafer chuck.
- 47. Grab the bottle of photoresist in one hand.
- 48. Grab the pipette from the bottle of photoresist in your dominant hand and pull it from the bottle such that the tip is above the photoresist level, but still within the opening of the bottle.
  - a. Note: The photoresist will be dripping from the pipette, so ensure these drops make it back into the bottle of photoresist.
- 49. Once the dripping has slowed to at least a couple seconds between drops, move the pipette from the bottle to the bowl in a swift (but cautious!) movement.
- 50. Once the dripping has slowed to at least a few seconds between drops, move the pipette to the center of the wafer and begin the dispensing process.
  - a. Note: The pipette should be held at approximately a 45° angle from vertical and the tip should be as close to the wafer as possible without touching the wafer or the photoresist.

- 51. To dispense the photoresist evenly and consistently, squeeze the bulb of the pipette with enough pressure that you have a steady and constant flow of photoresist coming out of the tip of the pipette.
- 52. Continue to dispense the photoresist until you have at least a half dollar-sized amount on the wafer or until you have a minimal amount of photoresist remaining in the pipette (and/or start seeing bubbles coming out of the pipette).
- 53. If any bubbles exist in the photoresist that was dispensed, now is the only time you have to get rid of them. Completely depress the bulb of the pipette (emptying any contents into the spinning bowl) and move the pipette to the surface of the photoresist, next to/on top of the bubble. Release the bulb slowly to get the bubble to go up into the pipette tip. Once the bubble has been sucked up into the pipette tip, stop releasing the bulb. Move the pipette from the wafer surface to the spinning bowl and squeeze the bubb completely to expel the bubble from the pipette. Repeat this step until all bubbles have been eliminated from the photoresist puddle.
- 54. Completely depress the bulb to the pipette (emptying any contents inside into the spinning bowl), keep it depressed, move it back to the bottle of photoresist, place the tip into the photoresist liquid, and release the bulb once the tip has gone entirely below the surface of the photoresist.
  - a. Note: DO NOT do this step if there are no more wafers to process. Instead, place the emptied pipette onto the cleanroom wipe for disposal later.
- 55. Place the bottle of photoresist back on its place on the cleanroom wipes (upper right-hand side).
- 56. Close the lid to the spinning chamber.
- 57. Ensure the "Lid Closed" and "Substrate Present" boxes on the interface are both illuminated in blue.
- 58. Press the "Start Process" button on the bottom left-hand side of the interface to begin the spinning process.
- 59. Wait for the process to complete. Once the process is complete, the system will alarm. Press the "OK" button when this alarm goes off.
- 60. After the alarm has been acknowledged, press the "Run" tab on the bottom left-hand side of the interface.
- 61. Select the "Thermal" option.
- 62. Ensure that the temperature is illuminated with a green background, denoting that the temperature is within its setpoint range.
- 63. Open the lid to the spinning chamber.
- 64. Grab the wafer with your Wafer Tweezers in your dominant hand and remove it from the spinning chamber.
- 65. Using your other hand, Press the "Start Process" button on the bottom left-hand side of the interface.
  - a. Note: The hot plate in this system has a 5 second step before it adds vacuum so that the wafer can be placed on the hot plate and adjusted to the center position before it adds a vacuum under the wafer and starts the 60 second timer.
- 66. Open the lid to the Hot Plate chamber with your free hand and place the wafer onto the hot plate such that the 2 standing posts are keeping it centered.
  - a. Note: This lid will not stay up, so you must keep it held with one of your hands while you are placing the wafer onto the hot plate.
- 67. Ensure the wafer achieves a vacuum (gets sucked down onto the hot plate) and close the lid to the Hot Plate chamber.

- a. Note: If there is a previous wafer cooling down on the cleanroom wipe, remove it from the wipe and place it back into the Wafer Box.
- 68. Wait for the timer to get close to expiring (approximately 1-3 seconds remaining) and open the lid to the Hot Plate chamber.
- 69. The wafer should lift up off the hot plate for 5 seconds after the 60 second timer expires, so utilize this time to grab the wafer from the hot plate.
- 70. Place the wafer onto the cleanroom wipe below.
- 71. Close the lid to the Hot Plate chamber.
- 72. The system should alarm once the entire recipe has completed, so hit the "OK" button once this goes off to acknowledge the alarm.
- 73. Keep the wafer on the cleanroom wipe for at least 60 seconds for it to cool down before placing it back into the Wafer Box.
- 74. Repeat Steps 37-73 for all wafers in the batch.
- 75. Once all of the wafers have finished spinning and baking, you then begin the process of returning the system back to how you found it. While the last wafer is cooling down on the cleanroom wipe, you can prepare the system for cleaning. Do this by grabbing the Acetone and Isopropyl Alcohol (IPA) bottles from the General Waste Bin station (or the Solvent Hood).
- 76. Move the pipette from the cleanroom wipe to the Flammable/Solid Waste bin.
  - a. Note: if this still remains full inside the bottle of photoresist, empty its contents into the spinner's bowl completely and then place directly into the Flammable/Solid Waste bin.
- 77. Return the cap to the photoresist bottle and place it back where it came from.
  - a. Note: This should be the toolbox you came in with if it is AZ nLOF 2020, but it could be the dry box in the cleanroom if you were using one of the ASU NanoFab's provided photoresists.
- 78. Ensure the "secret" login information was used so that you have access to the Diagnostics page of the software.
  - a. Note: You can manually clean the spinning bowl and the system, but that just requires some cleanroom wipes and Acetone/IPA combination. I will outline the steps for cleaning when a whole batch was completed.
- 79. Click on the "Diag" tab at the bottom center of the interface.
- 80. Select the "Spin Diagnostics" option.
- 81. Press the spin speed and change it from 250 rpm to 1,000 rpm.
- 82. Press the ramp rate and change it from 1,000 rpm/s to 250 rpm/s.
- 83. Open the lid to the spinning chamber.
- 84. Grab a blank Silicon wafer (not part of a batch) with your dominant hand.
- 85. Grab the centering tool with your other hand.
- 86. Place the centering tool into its contact point with the wafer chuck.
- 87. Place the wafer onto the wafer chuck and push it against the centering tool with the edge of your wafer tweezers.
- 88. Without moving the centering tool from its location, use your tweezer hand to press the "Yes" button under the "Vacuum" section of the interface (center-bottom left-hand side).
- 89. Once you see a checkbox ("Wafer Present?") appear above the button you just pressed, you can remove the centering tool from the spinning chamber.
- 90. Ensure there are no wafers still cooling down on the cleanroom wipe before proceeding to the next step.

- 91. Spray Acetone onto the wafer currently on the chuck until the entire wafer has a coating of Acetone on it.
- 92. Press the "Start" button under the "Spin" settings of the interface to begin spinning.
- 93. Spray Acetone onto the center of the spinning wafer for 5 seconds.
- 94. Continue spraying Acetone onto the center of the spinning wafer and also spray IPA onto the center of the wafer for 5 seconds.
- 95. Stop spraying the Acetone onto the spinning wafer and continue spraying IPA onto the center of the spinning wafer for 5 seconds.
- 96. Stop spraying the IPA onto the spinning wafer and wait until all of the rainbow colored streaks disappear from the wafer.
- 97. Wait another 15 seconds after the rainbow colored streaks disappear.
- 98. Press the "Stop" button until the "Spin" setting of the interface to stop spinning.
- 99. Press the "No" button under the "Vacuum" section of the interface to release the vacuum on the wafer.
- 100. Grab the wafer off the wafer chuck and place it back into the Wafer Box.
- 101. Grab the allen wrench from the chuck toolbox.
- 102. Unscrew the wafer chuck and remove it from the system, placing it back in the chuck toolbox.
  - a. Note: Make sure you keep track of the screw and place it in a spot inside the toolbox that is easy to find.
- 103. Spray Acetone onto one of the cleanroom wipes (preferably the top one since it will need to be disposed of already).
- 104. Wipe the spinner's bowl with the Acetone-soaked cleanroom wipe. Be sure to clean the surface of the bowl, the backside of the bowl, the edges of the bowl, and all surfaces of the bowl.
- 105. Once the bowl has been thoroughly cleaned and looks as good as (or better than) it did when you first grabbed it, place it back above the Solvent Hood.
- 106. Spray more Acetone onto the same cleanroom wipe you were using to clean the bowl and wipe down the spinner chamber, the lid, and the Stainless Steel surfaces of the coating system.
- 107. Once the system has been thoroughly cleaned, dispose of the cleanroom wipe you were using to clean into the Flammable/Solid Waste bin.
- 108. If you had an additional pair of gloves on (I usually do if I'm doing photoresist or if I'll be cleaning a spinner system), take the extra layer of gloves off without getting the other gloves dirty and place the dirty gloves into the Flammable/Solid Waste disposal bin.
- 109. You are now ready for the Exposure stage of the Photoresist Deposition process (4.3.2).
  - a. Note: Be sure to stay in the photolithography bay of the cleanroom since white light would expose the photoresist you just completed and your pattern will not turn out as expected.

#### 14.3.2 Photoresist Exposure

- 1. In the ASU NanoFab, the OAI 808 Mask Aligner is the tool we use for patterning and UV exposure of the photoresists we use.
  - a. Note: We have a HTG Mask Aligner in the ERC 444 cleanroom, but due to the broken Z-axis controls, we cannot use the aligner for anything except gap exposure. The Y-axis controls are also broken, so any alignment besides the first layer (not requiring alignment) is impossible.

- 2. Since we use the AZ nLOF 2020 photoresist and it requires a post-exposure bake to cross-link the polymer chains, set up a hot plate near you for use at 110°C for 60 seconds
  - a. Note: I like to use the built-in hot plate of the Brewer Science CEE Coater #1, which I also like to use for spinning the photoresist on. If this Spinner and Hot Plate were used for the photoresist and have not been changed, you can use the same Thermal program you used previously. If it is currently in use, simply find another hot plate to use and make sure you have a timer (cell phone/smart watch works well) ready to go.
- 3. Place a cleanroom wipe for wafer cooldown after the post-exposure bake. This can be placed either next to the hot plate or next to the Mask Aligner, either one works.
- 4. The OAI 808 Aligner requires training prior to use and uses a badge access to activate the vacuum lines. Scan your ASU ID on the "Login" badge portal and it should flash green lights when you have done so.
- 5. The interface box is located to the right of the machine. This should have been left on the screen that has "Mask Vacuum" in the bottom right-hand corner. If it is not on this screen, press the "Main Menu" button (upper left-hand side of the interface) and select "Manual Test" (middle left option).
- 6. There is a log book for the system on the rolling cart next to the machine. Fill in the information (name, date, start time, resist type, filter or no filter, calibrating gap or not calibrating gap, wafer size, wafer thickness, mask size, mask thickness, frontside alignment).
- 7. You will also need to record the lamp hours from the lamp's power box. This is located underneath the system on the left-hand side. Write this number into the log book.
- 8. The system now has to be prepped for your wafer and mask setup, so check what the current mask chuck and wafer chuck sizes are. If they do not match what you are working with, the following steps will dictate how to install the proper mask chuck and wafer chuck.
  - a. Note: Currently, we are using 4" wafers with a 5" mask plate. Typically, your mask plate is at least 1" larger than the wafer diameter.
- 9. Slide the wafer chuck out towards you.
- 10. Loosen the screws at the front and back of the wafer chuck (2 screws closest to you and 1 screw at the back of the wafer chuck).
- 11. Undo the vacuum line connections (3 of them) at the bottom/back right of the wafer chuck.
  - a. Note: They should read "BALL", "SUB", and "CONT"
- 12. Remove the wafer chuck by lifting it up off the posts.
- 13. Swap the previous wafer chuck with the correct wafer chuck (located on the top of the rack to your right against the wall).
  - a. Note: Be careful of the vacuum lines coming out of the vacuum chuck so that you do not pinch these. I typically will place the previous wafer chuck such that the vacuum lines are going through one of the slots in the wires of the rack and hanging beneath it.
- 14. Place the correct wafer chuck onto the posts the previous one was sitting on.
- 15. Screw the 2 screws closest to you first, but do not tighten completely (only tighten a few turns)
- 16. Screw the 1 screw furthest from you until you feel it contact the wafer chuck.
- 17. Finish screwing the 2 screws closest to you until you feel them contact the wafer chuck.
- 18. Tighten all of the screws, starting with the back one and finishing with the 2 front ones.
- 19. Install all 3 of the vacuum lines into their respective connections. The wafer chuck has now been installed.
  - a. Note: The vacuum lines will have a label ("CONT", "BALL", and "SUB") and the connection ports will have matching labels. Match the vacuum line to the matching

connection port. These can get tangled, so I recommend starting from the back connection port and working your way to the front connection port.

- 20. Loosen the four spring-loaded screws on top of the mask chuck to uninstall it from its location on the stack.
- 21. Undo the vacuum line connection to the left of the mask chuck. (Follow the vacuum line from the mask chuck to find this connection)
- 22. Remove the mask chuck by sliding it off the rails until it is clear of the microscope/camera lenses (alignment scope system) and then lift it off the system once it is clear of the lenses.
- 23. Swap the previous mask chuck with the correct mask chuck (located on the bottom of the rack to your right against the wall).
- 24. Install the vacuum line into the port on the left of the mask chuck. The mask chuck has now been installed.
- 25. Lay the mask chuck upside down (you should see a few posts and a spring system for mask installment) on the rolling cart next to the system.
- 26. The system is now ready for you to install your mask. Place a cleanroom wipe next to your toolbox.
- 27. Grab your 5" clear mask plate from the mask holder/carrier and place it onto the cleanroom wipe.
  - a. Note: If the wrong mask is currently installed, remove the blue tape holding it to the mask plate and place the tape on the corners of the mask plate for later use.
  - b. Note: If the correct mask is currently installed, skip to Step 29.
- 28. Grab the correct mask from your toolbox and install it such that the ink is facing towards you. Secure it to the mask plate with the blue cleanroom tape your removed from the previous mask.
  - a. Note: Typically we use a right-read down mask style, meaning that the ink is on the side that text appears backwards on. This is why I will always include some kind of text in the mask design. (The text should read normally when it is installed into the system and about to come into contact with the wafer)
- 29. Place the mask (with mask and blue cleanroom tape on the side facing towards you) onto the mask chuck and push it against the posts on the corners of the mask chuck.
- 30. There should be a spring-loaded post at one end of the mask chuck. Press this lever/button such that the post comes into contact with the mask plate.
- 31. Press the "Mask Vacuum" button on the interface. Ensure that the vacuum level (gauge on the far left of the machine) is at least to -10 inHg (or lower).
  - a. Note: If the mask vacuum is not high enough, try pressing down on the edges of the mask plate and/or reseating the mask.
- 32. Press the "Level Screen" button (top left-hand side of the interface) to prepare for the next step on the interface.
- 33. Carefully flip the mask chuck such that the mask is on the bottom side and place it onto the rails you first uninstalled it from.
- 34. Slide the mask chuck onto the rails and align the spring-loaded screws with the holes in the structure. Tighten these screws once they are in position.
  - a. Note: Ensure the positioning is correct by checking that the vacuum line is coming out towards you and is on the left side of the mask chuck (closest to the connection port)
- 35. The following steps (36-58) should be repeated for every wafer in the batch that need to get exposed.
- 36. Slide the wafer chuck out towards you.

- 37. Grab the next wafer to be processed from its Wafer Box and place it on the wafer chuck.
- 38. Align the wafer on the wafer chuck by making sure the flat is pushed against the two pins closest to you and that the edge of the wafer is pushed against the pin on the right edge of the wafer.
- 39. Press the "Substrate Vacuum" button (upper-middle left-hand side of the interface). Ensure that the vacuum level (gauge on the far left of the machine is at least to -10 inHg (or lower).
  - a. Note: If the substrate vacuum is not high enough, try pressing down on the edges of the wafer and/or reseating the wafer. Also make sure the wafer is not sitting on top of the pins you pushed it up against.
- 40. Push the wafer chuck away from you and into the system's stack.
- 41. Check the Exposure Time (upper-center right-hand side of the interface) and if it isn't correct, Press the "Main Menu" button (upper left-hand side of the interface).
- 42. Press the "Process Settings" button (upper right-hand side of the interface).
- 43. Press the "Exposure Time" box (upper right-hand side of the interface).
- 44. Change the time to the required exposure time.
  - a. Note: We use 6.6 seconds for our system since it is set to ~10 mW/cm<sup>2</sup> and we require 66 mJ/cm<sup>2</sup> of energy.
- 45. Press the "Main Menu" button (upper left-hand side of the interface).
- 46. Press the "Level Screen" button (center right-hand side of the interface).
- 47. Press the "Level" button (bottom left-hand side of the interface) and wait for the wafer chuck to get up to the height of the mask.
- 48. Calibrate the gap between the wafer chuck and the mask FOR THE FIRST WAFER ONLY. (Skip if not the first wafer)
  - a. Press the "Cal Gap" button (lower left-hand side of the interface).
  - b. Grab the caliper, turn it on, and place it onto the mask chuck such that its pin is in the center of the mask and the sides are resting on the top of the mask chuck.
  - c. Change the movement increment to 10 µm by pressing on the increment number.
  - d. Lower the wafer chuck (by 10 µm increments) until the reading on the caliper is no longer changing AND keep going until it is at least at 100 µm past that point to make sure.
  - e. Press the "Origin" button on the caliper to return the reading to 0.000 mm.
  - f. Raise the wafer chuck slowly (by 10  $\mu m$  increments) until the caliper changes to any value besides 0.000 mm.
  - g. Change the movement increment to 3 µm by pressing on the 10 µm number.
  - h. Raise the wafer chuck slowly (by 3 µm increments) until the caliper reads -0.004 mm.
  - i. Record the number in the "Gap" reading in the log book.
  - j. Press the "Zero Gap" button (lower right-hand side of the interface)
  - k. Press the "Run Screen" button (upper left-hand side of the interface) to return to the Run Screen.
- 49. Line up the wafer to the mask by using the micromanipulators at the base of the wafer chuck. Make sure that the mask is in the correct location on the wafer.
- 50. Press the "Contact" button on the interface (upper-center left-hand side of the interface) and wait a few seconds for that to activate.
- 51. Press the "Hard Contact" button on the interface (upper-center right-hand side of the interface) and wait a few seconds for that to activate.
- 52. Press the "Cycle" button on the interface (lower right-hand side of the interface). The system will now move the UV lamp head over the wafer and mask, open the shutter for the specified

duration, and return to its original position. It will also lower the wafer chuck back to its loading location and switch the interface to the "Level Screen" where you will see the "Substrate Vacuum" and "Level" buttons.

- 53. Slide the wafer chuck out towards you.
- 54. Press the "Substrate Vacuum" button (upper-center left-hand side of the interface) to release the vacuum from the wafer.
- 55. Grab the wafer from the wafer chuck and take it over the the Hot Plate which should be previously set up for you.
- 56. Place the wafer on the hot plate and start the timer (or press "Start Process" if you are using CEE Coater #1)
- 57. Once the timer has completed, remove the wafer from the hot plate and place it on a cleanroom wipe. This can be located either next to the hot plate or at the Mask Aligner. Leave the wafer on the cleanroom wipe for at least 60 seconds.
- 58. Remove the wafer from the cleanroom wipe and place it back into its slot in the Wafer Box.
- 59. Repeat Steps 36-58 for each wafer in the batch that you are working with.
- 60. Once you have completed processing on all wafers in the batch, push the wafer chuck away from you and back into the system (without a wafer on it).
- 61. Unscrew the 4 spring-loaded screws in the mask chuck.
- 62. Slide the mask chuck out on the rails until it is past the microscope/camera lenses.
- 63. Lift the mask chuck off the rails and flip the mask chuck such that the mask is facing toward the ceiling.
- 64. Place the mask chuck on the rolling cart next to the machine.
- 65. Press the "Main Menu" button (upper left-hand side of the interface).
- 66. Press the "Manual Test" button (center left-hand side of the interface).
- 67. Press the "Mask Vacuum" button (bottom right-hand side of the interface).
- 68. Pull the spring-loaded post (on the mask chuck) away from the mask.
- 69. Remove the mask from the mask chuck and place it back into the mask holder/carrier (mask and tape side facing down). Close the mask holder/carrier when it is in place and return this to the toolbox you brought into the cleanroom with you.
- 70. Flip the mask chuck to its normal orientation, place it back onto the rails it was on, and slide it all the way back to its location.
- 71. Tighten at least 2 of the spring-loaded screws down.
  - a. Note: I usually do either 2 opposite corners or all 4.
- 72. Press your ASU ID against the badge portal that says "Logout". It should flash green and then both the "Login" and "Logout" portals should be red.
- 73. Write the total time you exposed the wafers for (adding up all of the times you used for the batch you just completed) and write the end time into the log book.
- 74. You are now ready for the Developing process.

#### 14.3.3 Photoresist Developing

1. Our AZ nLOF 2020 photoresist has a recommended Developer solution of AZ 300 MIF. This chemical is available in both the ASU NanoFab and in the ERC 444 cleanroom. If you are doing
your photoresist processing in the ASU NanoFab, it is recommended to just do this step in the NanoFab as well.

- 2. Find a clean working bench at a Base Hood to prepare your work station for this process at.
- 3. Place two cleanroom wipes on the bench, with one near a sink.
- 4. Grab a Pyrex dish from the supply rack (some say "300 MIF" on them and those are preferred to be used) and place it on the cleanroom wipe furthest from the sink.
  - a. Note: The other cleanroom wipe will be used for drying the wafers after the processing.
- 5. Place a timer next to the dish for 300 MIF. (The Stopwatch function will be needed)
  - a. Note: The AZ nLOF 2020 resist typically takes about 30 seconds for complete development.
- 6. Place your Wafer Box containing wafers needing to go through the developing process inside the Hood, next the the dish for 300 MIF.
- 7. Place your wafer tweezers or wand next to the Wafer Box.
- 8. Don the proper PPE required for chemical handling (apron, base gloves, & face shield).
  - a. Note: Technically this is only required when handling the chemicals, so they can be removed once you have put the 300 MIF bottle back in its storage location and have the Hazardous Waste bottle for 300 MIF (and the funnel) ready to go at the fume hood.
- 9. If you are more than a couple steps away from the storage location of the 300 MIF, grab a chemical transport container.
- 10. Grab the 300 MIF bottle from its storage location. (Place this into the chemical transport container if that is required here)
- 11. Bring the bottle of 300 MIF to the Base Hood you will be operating out of.
- 12. Open the cap to the bottle and pour 300 MIF into the dish until the chemical level is at least 1/2" deep. (This should be approximately 100-200 mL, depending on the size of the dish)
- 13. Return the cap to the bottle of 300 MIF.
- 14. Return the 300 MIF bottle to its storage location. (Use the chemical transport container if necessary).
- 15. Make sure there is a Hazardous Waste bottle for 300 MIF at the Base Hood as well. If this is not at the hood already, grab it from its storage location (using the chemical transport container if necessary) and transport it to the hood).
- 16. Make sure there is a funnel at the hood for ease of pouring into the Hazardous Waste bottle when completed.
  - a. Note: If you would like to remove your apron, face shield, and base gloves, you may do so at this point.
- 17. The ASU NanoFab has a sink and a drain attached to it and can be easily used to simply rinse the wafers once they are done developing. If you are in ERC 444 (or at Mayo Clinic), a bath setup will likely be needed for the water rinse step. For this bath setup, you should fill a dish (approximately the same size as the one for the 300 MIF solution) up to the same 1/2" level you filled the 300 MIF dish up to (~100-200 mL depending on the dish). This will also require an additional cleanroom wipe.
- 18. The following steps (19-39) should be repeated for each wafer in the batch that require developing.
- 19. Start the water flow from the sink if you are using the sink for the rinse and not a bath.
- 20. Grab the next wafer to be processed from the Wafer Box in your dominant hand.
- 21. In your other hand, grab the timer and prepare to begin the Stopwatch.

- 22. Simultaneously, begin the Stopwatch as you place the wafer into the 300 MIF solution. Make sure the entire wafer gets submerged into the 300 MIF solution.
- 23. Using the tip of your tweezers (and holding vertically), grab the side of the wafer by the major flat (by pushing against the wafer) and slowly move the wafer up and down in the solution. Be sure to not let the wafer breach the surface of the solution.
  - a. Note: This is performed to agitate and create movement in the solution so that the chemical solution can access all the photoresist on the wafer. This motion can be replaced if there is a stirrer or other similar device to create this movement.
- 24. While the wafer is in the developer solution, you should be able to see the photoresist coming off to form the pattern that you created from the Mask Aligner.
- 25. Once the Stopwatch reaches 27 seconds, pull the wafer from the solution to check if it has completed its processing.
- 26. If the wafer has completed its processing (and you can see the pattern you used at the Mask Aligner), move the wafer to the water stream coming from the sink. If the wafer needs to continue processing, place it back into the solution and repeat Steps 23-26 until completed.
  - a. Note: If using the water bath instead of the sink, place the wafer into the water bath.
- 27. Rinse the front of the wafer (pattern) by running it across the stream of water a few times.
- 28. Rinse the back of the wafer (no pattern, label) by running it across the stream of water a few times.
- 29. Rinse the front of the wafer (pattern) again by running it across the stream of water a few times.
- 30. Place the wafer onto the cleanroom wipe dedicated to drying the wafers.
- 31. Stop the flow of water coming from the sink.
- 32. Grab the Nitrogen air gun and dry the wafer starting in the center.
- 33. Rotate your wrist (NOT your arm/elbow) and push the liquid towards the edges of the wafer until there is no more water on the front of the wafer.
- 34. Dry off your wafer tweezers away from your wafer (so that no liquid splashes onto your dried wafer).
- 35. Pick up your wafer from the cleanroom wipe and place it vertically back on the cleanroom wipe so that you can dry the back side of the wafer.
- 36. Dry the back side of the wafer in the same fashion you dried the front (start in the center and rotate your wrist to push the liquid to the edges of the wafer). The wafer does not have to be exactly vertical, but it just cannot be touching any of the faces (front or back) to the cleanroom wipe.
- 37. Lift the wafer off the cleanroom wipe and pass the Nitrogen air gun across the edge of the wafer (back and forth) across the entire wafer (rotating your wrist to access the rest of the wafer).
  - a. Note: This is hard to put in words, but basically you are just pushing any liquid from the edge of the wafer onto one of the surfaces (front or back) to be dried again.
- 38. Face the front of the wafer towards you and dry off any liquid that got pushed back onto the front of the wafer from the edge dry.
- 39. Place the completed wafer into the Wafer Box for later inspection.
- 40. Repeat Steps 19-39 for each wafer in the batch that you are completing through this stage of the process.
- 41. Once all wafers in the batch have completed their Development process, you can begin the teardown of the station. Start by moving the timer, Wafer Box, and wafer tweezers out of the way and onto another table top.

- 42. Don the PPE for Base chemical handling if you do not have it on anymore (face shield, apron, and base gloves).
- 43. Move the Hazardous Waste bottle for 300 MIF to a location in which you can pour the contents of your dish into it.
- 44. Remove the cap to the waste bottle and place the funnel into the bottle.
- 45. Carefully pour the contents of the dish of 300 MIF into the waste bottle through the funnel.
- 46. Once all the contents have been emptied into the waste bottle, spray/add water to the dish until the water is at least three-quarters as high as the chemical solution was.
- 47. Swirl the water around the dish so that the water can remove any residue 300 MIF solution from the dish.
- 48. Empty the dish into the waste bottle.
- 49. Spray/Add a little bit of water to the dish, swirl it around, and empty it into the waste bottle.
- 50. Spray/Add at least 100 mL of water to the dish, swirl it around, and empty it into the sink (down the drain).
- 51. Repeat Step 50 for rinsing the 300 MIF dish (Completed twice total).
- 52. Using the cleanroom wipe that the 300 MIF dish was sitting on, dry out the dish you just rinsed with water. Place this cleanroom wipe in the "Flammable/Solid Waste" bin.
- 53. Place this dish to the side for returning to its storage location later.
- 54. If you were using a dish for a water bath, swirl the contents around and empty it into the waste bottle for 300 MIF.
- 55. Spray/Add a little more water to the water dish, swirl it around, and empty it into the waste bottle for 300 MIF.
- 56. Spray/Add at least 100 mL of water to the dish, swirl it around, and empty it into the sink (down the drain).
- 57. Repeat Step 56 for rinsing the water dish (Completed twice total).
- 58. Using the cleanroom wipe that the water dish was sitting on, dry out the dish you just rinsed with water. Place this cleanroom wipe in the "Flammable/Solid Waste" bin.
- 59. Place this dish to the side for returning to its storage location later.
- 60. Tap the funnel on the side of the waste bottle a few times to get some last remaining drops off it.
- 61. Screw the cap back onto the waste bottle.
- 62. Move the funnel back to its original location.
- 63. Move the waste bottle to its original location (using the chemical transport container if necessary).
- 64. Remove the PPE you donned for the Developer chemical handling and place it back where you got it from.
- 65. Return the dishes back to their storage locations.
- 66. Make sure the water is not running or dripping from the faucet.
- 67. Using the cleanroom wipe you used for drying the wafers, wipe down the surface area of the hood that you just used for the processing. Grab more cleanroom wipes if necessary. Ensure that the fume hood surface looks as good (if not better) than when you started using it. Place any used cleanroom wipes in the "Flammable/Solid Waste" bin.
- 68. You have completed the Development stage of the process and your wafers are ready for inspection.

69.

## 14.3.4 Inspection

- 1. There are three methods of inspection we use to measure the quality of photoresist we get after spinning, exposure, and developing.
  - a. The first is a visual inspection using our eyes.
  - b. The second is a visual inspection using a microscope.
  - c. The third is a metric inspection using a measurement tool to get the thickness of the layer.
- 2. As a first inspection, look at the wafer and ensure you have gotten the correct pattern to what you were setting out to get from the photoresist/mask combination. Also inspect for defects like bubbles, dirt, debris, and any voids in the photoresist material. The color combination is also an effective tool to ensure there are not too many striations in the colors, meaning that something didn't quite come out as planned. Luckily, photoresist can always be reworked, so this entire process can be repeated if necessary.
- 3. The second inspection involves using a microscope to inspect any potential defects found as well as to check the correct exposure energy and developing time. Check the two squares used for alignment for checking for the correct exposure energy and development times, based on the connection at the corners. (Bridge versus gap). Using the microscope can also inspect the wafer for any mask issues that you did not already notice.
- 4. The third inspection is where we measure the thickness of the photoresist layer we just deposited onto the wafer. At ASU, we use a Reflectometer in the form of a Filmetrics F40 system (under yellow lights) or a Filmetrics F20 system (under white lights). The following processing steps will follow the technique we require for that process.
  - a. Note: We can program a new material into the Filmetrics Software if needed. A tutorial is being written about how to go about doing that. PI-2611 (soft-baked & cured), Parylene, AZ nLOF 2020, & AZ 15nXT are all present on the F20, soon to be added to the F40.
- 5. Ensure the PC at the F40 station is operational and move the mouse around if the monitor is blank.
- 6. Run a baseline test by grabbing the blank "Baseline" Silicon wafer with your Wafer Tweezers or Wand and placing it underneath the microscope of the system.
- 7. Once the wafer has been placed underneath the microscope, find an edge on the wafer to focus the optics onto.
- 8. Click "Baseline" on the F20 software and then click "OK".
- 9. The software will now ask you to tilt the object at 10°-80° to cancel out the background light it might see. Grab the Container the blank Silicon wafer was sitting on the place it next to the beam spot. Then grab the blank Silicon wafer and set it onto the container such that the flat part is sitting on the base while the curved part is elevated and resting partially on the container. Click "OK" in the software to grab the measurement.
  - a. Note: This is tricky because this system uses a microscope to perform the analysis. Be careful around the optics to not damage them. It may make it easier to drop the stage by a set amount to be able to run this test.
- 10. Grab one of the other wafers in the calibration stack and check the system to make sure it is reading correctly.
- 11. The software is now ready for processing, so start by loading the correct recipe.

- a. Note: Load "AZ nLOF" from "Photoresists".
- 12. Once the recipe is selected, you will now have to select the correct nominal thickness. Click "Edit Recipe" to change the nominal thickness as well as the measurement type (Angstroms, nanometers, or micrometers).
- 13. The following Steps (14-19) should be repeated for every wafer that needs to be tested for thickness.
- 14. Place the wafer that needs to be inspected onto the microscope such that the center of the wafer is directly underneath the beam of the system. Focus the microscope onto a feature on the surface of the wafer.
- 15. Click the "Measure" button on the software to grab the measurement. Write this measurement into a table in your Cleanroom Notebook as the "Center" location.
- 16. Move the wafer (using the stage manipulator) to the right such that the left middle of the wafer is now underneath the microscope lens.
  - a. Note: For reference, I consider the major flat to be the bottom of the wafer and the opposite side to be the top of the wafer. This makes everything easier to think about in my mind. Orientation is a personal choice and does not matter, as long as it is consistent.
- 17. Click the "Measure" button on the software OR press the "Enter" key on the keyboard. Write this measurement into a table in your Cleanroom Notebook as the "Left Middle" location.
- 18. Move the wafer to the right such that the left side is now underneath the microscope lens.
  - a. Note: You do not want to capture the far edges of the wafer as this is where the edge bead has formed. Make sure this measurement is not on the edge bead section.
- 19. Click the "Measure" button on the software OR press the "Enter" key on the keyboard. Write this measurement into a table in your Cleanroom Notebook as the "Left" location.
- Continue the previous steps to get the "Right Middle", "Right", "Top Middle", "Top", "Bottom Middle", and "Bottom" measurements. Write each measurement into the table in your Cleanroom Notebook.
  - a. Note: These specific locations are important because they ensure consistency and they also cover enough sections of the wafer that any major defects can be determined simply by thickness variance. Gathering 9 points of data also helps in statistical verification as well as getting a surface profile of the wafer every time a process is completed.
- 21. Using your Wafer Tweezers or Wand, scoot the wafer off the base platform and load it back into the Wafer Box it came from.
- 22. Repeat Steps 14-21 for every wafer in the batch that is needing to be tested for thickness.
- 23. Once all wafers are completed, take the data from the Cleanroom Notebook you wrote it on and transfer it to an Excel sheet (or Google Sheet) such that it can be analyzed for averages, standard deviations, and other statistical calculations. Saving all data into a common location can help with product tracing throughout the device's fabrication as well as ensuring that the processing steps remain consistent.
  - a. Note: Examples of the tables used in the Cleanroom Notebook as well as the Excel tables can be provided. This can all be recorded directly into an Excel Sheet or Google Sheet instead of onto a cleanroom notebook. I have to write the measurements onto a cleanroom notebook just because that is the nature of the lab in which the measurement tool is located in and I have not implemented a Google Sheet for this yet.

# **15 Substrate Etching for Device Removal**

# **15.1 Equipment Needed**

- RIE Machine (preferred) or Plasma Cleaner (acceptable)
- Quartz ring (>4" diameter) for wafer containment
- Thickness Measurement Tool (Filmetrics F20 Reflectometer or 3D Profilometer)
- Microscope (for inspection)
- Camera (for microscopic imaging)

# **15.2 Materials Needed**

- Wafer Tweezers or Wand (4")
- Wafer (3" or 4") -Silicon w/ Polyimide, Conductive Metal, Parylene, & Photoresist
- Cleanroom Notebook
- Cleanroom Wipes
- Marker for wafer labeling

# **15.3 Etching Process**

## 15.3.1 Reactive Ion Etching Machine

1.

## 15.3.2 Inspection

1.

# 16 Photoresist Removal after Device Removal Etching

# **16.1 Equipment Needed**

- Pyrex dishes (>4" diameter) (Qty: 3)
- Fume Hood
- Sonicator OR Nitrogen bubblers
- Timer
- Thickness Measurement Tool (Filmetrics F20 Reflectometer or 3D Profilometer)
- Microscope (for inspection)
- Camera (for microscopic imaging)

## **16.2 Materials Needed**

- Wafer Tweezers or Wand (4")
- Wafer (3" or 4") Silicon w/ Polyimide, Conductive Metal, Parylene, & Photoresist
- Cleanroom Notebook
- Cleanroom Wipes
- Marker for wafer labeling
- Photoresist Stripper (Pick one or more)
  - AZ 400T Stripper (~100-200 mL per batch)
  - Acetone (~100-200 mL per batch)
  - Accompanying Stripper for thick negative photoresist (~100-200 mL per batch)
- Isopropyl Alcohol (~100-200 mL per batch)
- DI Water (~100-200 mL per batch)
- Waste bottles for all chemicals (~3)

# 16.3 Removal Process

## 16.3.1 Photoresist Stripper & Cleaning Baths

1.

## 16.3.2 Inspection

1.

# **17 Device Removal**

# **17.1 Equipment Needed**

•

# **17.2 Materials Needed**

- Wafer Tweezers or Wand (4")
- Wafer (3" or 4") Silicon w/ Polyimide, Conductive Metal, & Parylene
- Cleanroom Notebook
- Cleanroom Wipes
- Marker for wafer labeling

# **17.3 Removal Process**

1.

## APPENDIX B ELECTRODE EVALUATION

# ASU Wafer Shipment Detailed Evaluation

## M<sup>3</sup> Electrode Arrays

#### Received: December 2018

Evaluated by Jon Garich

- 128-channel arrays per wafer: 1
  - Macros: 4 per array
  - Mesos: 16 per array
  - Micros: 108 per array
- 32-channel arrays per wafer: 6
  - Macros: 1 per array
  - Mesos: 4 per array
  - Micros: 27 per array
- 4" wafers received: 20
  - o 128-channel arrays: 20
  - o 32-channel arrays: 120

## **Evaluation Parameters:**

The arrays will be inspected for any defects in the metal layer such as shorts, opens, debris underneath, scratches/tears, missing details, trace defects (blobs or holes), or other potential causes for electrical concern. The insulation layer will also be inspected for defects such as holes/openings, bubbles, debris, tears, or other potential causes for mechanical concern.

Together, the defects should not amount to more than 20% of the available electrodes or traces in order to be considered "good" arrays. For the 128-channel arrays, this means fewer than 26 defects throughout the entire array. For the 32-channel arrays, this means fewer than 7 defects throughout the entire array.

Arrays with damage to meso or macro electrodes will be marked as such, but will not be excluded from consideration unless additional defects push the array over the 20% defect allowance.

Arrays with insulation remaining on any part of the array (electrodes or connector pads) will be marked as such and excluded from consideration. Arrays that would be otherwise be considered quality arrays could potentially receive a patterned oxide plasma etch to completely remove the insulation remaining.

Arrays with perfect metal layers, perfect insulation layers, or both will be marked as such to provide a good idea of how many "perfect" layers were produced.

## Contents

Fabrication Information	
Array Results & Yields	9
128-Channel Arrays	9
Yields	9
Results by Array	9
32-Channel Arrays	
Yields	10
Results by Array	10
Microscope Evaluation	
B01-001	12
128-Channel Array #1	12
32-Channel Array #1 (Top of Top Section)	13
32-Channel Array #2 (Middle of Top Section)	
32-Channel Array #3 (Bottom of Top Section)	
32-Channel Array #4 (Top of Bottom Section)	
32-Channel Array #5 (Middle of Bottom Section)	15
32-Channel Array #6 (Bottom of Bottom Section)	
B01-002	
128-Channel Array #2	17
32-Channel Array #7 (Top of Top Section)	
32-Channel Array #8 (Middle of Top Section)	
32-Channel Array #9 (Bottom of Top Section)	20
32-Channel Array #10 (Top of Bottom Section)	20

	32-Channel Array #11 (Middle of Bottom Section)	. 20
	32-Channel Array #12 (Bottom of Bottom Section)	. 21
В	01-003	. 22
	128-Channel Array #3	. 22
	32-Channel Array #13 (Top of Top Section)	. 23
	32-Channel Array #14 (Middle of Top Section)	. 24
	32-Channel Array #15 (Bottom of Top Section)	. 24
	32-Channel Array #16 (Top of Bottom Section)	. 24
	32-Channel Array #17 (Middle of Bottom Section)	. 25
	32-Channel Array #18 (Bottom of Bottom Section)	. 25
В	01-004	. 25
	128-Channel Array #4	. 25
	32-Channel Array #19 (Top of Top Section)	. 26
	32-Channel Array #20 (Middle of Top Section)	. 26
	32-Channel Array #21 (Bottom of Top Section)	. 27
	32-Channel Array #22 (Top of Bottom Section)	. 27
	32-Channel Array #23 (Middle of Bottom Section)	. 27
	32-Channel Array #24 (Bottom of Bottom Section)	. 27
В	02-005	. 28
	128-Channel Array #5	. 28
	32-Channel Array #25 (Top of Top Section)	. 28
	32-Channel Array #26 (Middle of Top Section)	. 29
	32-Channel Array #27 (Bottom of Top Section)	. 29
	32-Channel Array #28 (Top of Bottom Section)	. 29
	32-Channel Array #29 (Middle of Bottom Section)	. 30
	32-Channel Array #30 (Bottom of Bottom Section)	. 30
В	02-006	. 32
	128-Channel Array #6	. 32
	32-Channel Array #31 (Top of Top Section)	. 33
	32-Channel Array #32 (Middle of Top Section)	. 33

32-Channel Array #33 (Bottom of Top Section)	
32-Channel Array #34 (Top of Bottom Section)	
32-Channel Array #35 (Middle of Bottom Section)	
32-Channel Array #36 (Bottom of Bottom Section)	
B02-007	
128-Channel Array #7	
32-Channel Array #37 (Top of Top Section)	
32-Channel Array #38 (Middle of Top Section)	
32-Channel Array #39 (Bottom of Top Section)	
32-Channel Array #40 (Top of Bottom Section)	
32-Channel Array #41 (Middle of Bottom Section)	
32-Channel Array #42 (Bottom of Bottom Section)	
B02-008	
128-Channel Array #8	
32-Channel Array #43 (Top of Top Section)	
32-Channel Array #44 (Middle of Top Section)	
32-Channel Array #45 (Bottom of Top Section)	
32-Channel Array #46 (Top of Bottom Section)	
32-Channel Array #47 (Middle of Bottom Section)	
32-Channel Array #48 (Bottom of Bottom Section)	
B03-009	
128-Channel Array #9	
32-Channel Array #49 (Top of Top Section)	
32-Channel Array #50 (Middle of Top Section)	
32-Channel Array #51 (Bottom of Top Section)	
32-Channel Array #52 (Top of Bottom Section)	
32-Channel Array #53 (Middle of Bottom Section)	
32-Channel Array #54 (Bottom of Bottom Section)	
B03-010	50
128-Channel Array #10	50

	32-Channel Array #55 (Top of Top Section)	. 50
	32-Channel Array #56 (Middle of Top Section)	. 50
	32-Channel Array #57 (Bottom of Top Section)	. 51
	32-Channel Array #58 (Top of Bottom Section)	. 51
	32-Channel Array #59 (Middle of Bottom Section)	. 51
	32-Channel Array #60 (Bottom of Bottom Section)	. 52
В	03-011	. 52
	128-Channel Array #11	. 52
	32-Channel Array #61 (Top of Top Section)	. 53
	32-Channel Array #62 (Middle of Top Section)	. 53
	32-Channel Array #63 (Bottom of Top Section)	. 54
	32-Channel Array #64 (Top of Bottom Section)	. 54
	32-Channel Array #65 (Middle of Bottom Section)	. 54
	32-Channel Array #66 (Bottom of Bottom Section)	. 54
В	03-012	. 55
	128-Channel Array #12	. 55
	32-Channel Array #67 (Top of Top Section)	. 56
	32-Channel Array #68 (Middle of Top Section)	. 56
	32-Channel Array #69 (Bottom of Top Section)	. 56
	32-Channel Array #70 (Top of Bottom Section)	. 57
	32-Channel Array #71 (Middle of Bottom Section)	. 57
	32-Channel Array #72 (Bottom of Bottom Section)	. 57
В	04-013	. 57
	128-Channel Array #13	. 57
	32-Channel Array #73 (Top of Top Section)	. 57
	32-Channel Array #74 (Middle of Top Section)	. 57
	32-Channel Array #75 (Bottom of Top Section)	. 57
	32-Channel Array #75 (Bottom of Top Section)	. 57 . 58
	<ul> <li>32-Channel Array #75 (Bottom of Top Section)</li> <li>32-Channel Array #76 (Top of Bottom Section)</li> <li>32-Channel Array #77 (Middle of Bottom Section)</li> </ul>	. 57 . 58 . 58

B	04-014	. 58
	128-Channel Array #14	. 58
	32-Channel Array #79 (Top of Top Section)	. 58
	32-Channel Array #80 (Middle of Top Section)	. 58
	32-Channel Array #81 (Bottom of Top Section)	. 59
	32-Channel Array #82 (Top of Bottom Section)	. 59
	32-Channel Array #83 (Middle of Bottom Section)	. 59
	32-Channel Array #84 (Bottom of Bottom Section)	. 59
В	04-015	. 59
	128-Channel Array #15	. 59
	32-Channel Array #85 (Top of Top Section)	. 59
	32-Channel Array #86 (Middle of Top Section)	. 60
	32-Channel Array #87 (Bottom of Top Section)	. 60
	32-Channel Array #88 (Top of Bottom Section)	. 60
	32-Channel Array #89 (Middle of Bottom Section)	. 60
	32-Channel Array #90 (Bottom of Bottom Section)	. 60
В	04-016	. 60
	128-Channel Array #16	. 60
	32-Channel Array #91 (Top of Top Section)	. 61
	32-Channel Array #92 (Middle of Top Section)	. 61
	32-Channel Array #93 (Bottom of Top Section)	. 61
	32-Channel Array #94 (Top of Bottom Section)	. 61
	32-Channel Array #95 (Middle of Bottom Section)	. 61
	32-Channel Array #96 (Bottom of Bottom Section)	. 61
В	05-017	. 62
	128-Channel Array #17	. 62
	32-Channel Array #97 (Top of Top Section)	. 62
	32-Channel Array #98 (Middle of Top Section)	. 62
	32-Channel Array #99 (Bottom of Top Section)	. 62
	32-Channel Array #100 (Top of Bottom Section)	. 62

	32-Channel Array #101 (Middle of Bottom Section)	62
	32-Channel Array #102 (Bottom of Bottom Section)	62
B	05-018	. 63
	128-Channel Array #18	. 63
	32-Channel Array #103 (Top of Top Section)	. 63
	32-Channel Array #104 (Middle of Top Section)	. 63
	32-Channel Array #105 (Bottom of Top Section)	. 63
	32-Channel Array #106 (Top of Bottom Section)	. 63
	32-Channel Array #107 (Middle of Bottom Section)	64
	32-Channel Array #108 (Bottom of Bottom Section)	64
B	05-019	. 64
	128-Channel Array #19	. 64
	32-Channel Array #109 (Top of Top Section)	. 64
	32-Channel Array #110 (Middle of Top Section)	. 64
	32-Channel Array #111 (Bottom of Top Section)	65
	32-Channel Array #112 (Top of Bottom Section)	65
	32-Channel Array #113 (Middle of Bottom Section)	65
	32-Channel Array #114 (Bottom of Bottom Section)	65
B	05-020	65
	128-Channel Array #20	65
	32-Channel Array #115 (Top of Top Section)	65
	32-Channel Array #116 (Middle of Top Section)	. 66
	32-Channel Array #117 (Bottom of Top Section)	. 66
	32-Channel Array #118 (Top of Bottom Section)	. 66
	32-Channel Array #119 (Middle of Bottom Section)	66
	32-Channel Array #120 (Bottom of Bottom Section)	66
Cor	clusions	. 66

## **Fabrication Information**

Table/list of material thicknesses and conductivity measurements, recorded at ASU.

Thickness measurements were collected with a reflectometry system inside the ASU NanoFab cleanroom (Filmetrics F20 and/or F40 systems) at nine measurement points throughout the wafers (left, left-center, center, right-center, right, top, top-center, bottom-center, and bottom, where the bottom is the location of the major flat). Insulation thickness measurements in the center of the wafers were moved to the metal test pads on the perimeter of the wafer (designed for four-point probe testing) to give a more accurate measurement of the thickness without having to take the variability of the substrate thickness into account.

Four-point probe measurements were taken with a four-point probe measurement station inside the ASU NanoFab cleanroom (Signatone probe stand with a Keithley 2000 multimeter). The nine measurement locations for four-point probe testing were different from thickness measurements because the array structures internal to the wafers were not testable with the large probe of the station, so four designated test pads on the outer perimeter of the wafer were designed for this testing purpose.

	Average	Average	Average	Average	Average	Average
	Substrate	Insulation	Total Array	Four-Point	Sheet	Resistivity
	Thickness	Thickness	Thickness	Probe	Resistance	(μΩ*cm)
	(µm)	(µm)	(µm)	Resistance	(Ω/sq)	
				(Ω)		
B01-001	9.752	9.575	19.327	1.463	6.630	69.62
B01-002	9.726	9.408	19.134	1.349	6.114	64.19
B01-003	9.668	9.484	19.152	1.414	6.409	67.30
B01-004	9.751	9.606	19.357	1.399	6.338	66.55
B02-005	9.734	9.530	19.264	1.425	6.457	67.80
B02-006	9.686	9.611	19.298	1.428	6.471	67.95
B02-007	9.820	9.424	19.245	1.303	5.903	61.98
B02-008	9.773	9.437	19.210	1.264	5.729	60.16
B03-009	9.818	9.569	19.387	1.437	6.511	68.37
B03-010	9.728	9.427	19.155	1.376	6.236	65.48
B03-011	9.734	9.567	19.302	1.241	5.624	59.05
B03-012	9.755	9.588	19.343	1.359	6.160	64.69
B04-013	9.738	9.479	19.217	1.327	6.013	63.14
B04-014	9.741	9.579	19.320	1.416	6.418	67.39
B04-015	9.784	9.455	19.240	1.311	5.941	62.38
B04-016	9.789	9.502	19.291	1.242	5.627	59.09
B05-017	9.719	9.476	19.195	1.393	6.314	66.29
B05-018	9.766	9.490	19.256	1.401	6.351	66.68
B05-019	9.738	9.617	19.355	1.379	6.250	65.63
B05-020	9.732	9.680	19.413	1.322	5.990	62.89

Note: Conversion to sheet resistance from the four-point probe resistance measurement uses a conversion factor of 4.532, which is calculated based on the distance between the probe tips, the probe material, and the system resistance. Sheet resistance is a measurement in  $\Omega$ /square and can be translated to resistivity when multiplied by the metal thickness in centimeters. The

resulting resistivity numbers for thin-film metal layers are typically quite small, so it is beneficial to convert the measurement from  $\Omega$ -cm to  $\mu\Omega$ -cm (or a respective conversion based on the resulting data).

## **Array Results & Yields**

Below are tables of all arrays from the received wafers and the conclusion of the evaluation. This conclusion includes a designation of a good array or the reason in which it was determined to be a bad array.

## **128-Channel Arrays**

Yields Total: 13 / 20 [65% yield]

#### **Good Arrays**

Meso/Macro Defects: 5 / 13 [38.5% of good arrays]

Perfect Metal: 4 / 13 [30.7% of good arrays]

Perfect Insulation: 3 / 13 [23.1% of good arrays]

Perfect Arrays: 1 / 13 [7.7% of good arrays]

#### **Bad Arrays**

Insulation Remaining: 3 / 7 [42.9% of bad arrays] but would otherwise be considered Good

#### **Results by Array**

Wafer ID	Array #	Good/Bad	Reason	Notes
B01-001	1	Good		Possibly etched polyimide
B01-002	2	Bad	Too many defects	Defect in meso; Laser hole created 21 open channels
B01-003	3	Good		
B01-004	4	Good		Perfect metal
B02-005	5	Good		Defect in macro & mesos
B02-006	6	Reworkable	Insulation Remaining	1 other defect
B02-007	7	Good		Defect in meso
B02-008	8	Good		Debris throughout wafer
B03-009	9	Good		Perfect metal; Defect in meso
B03-010	10	Good		Perfect array
B03-011	11	Bad	Too many defects	15 metal or insulation defects
B03-012	12	Bad	Too many defects	23 metal defects (shorts/opens)
B04-013	13	Good		Possible etched polyimide; Perfect metal
B04-014	14	Reworkable	Insulation Remaining	2 other defects
B04-015	15	Good		Perfect insulation
B04-016	16	Reworkable	Insulation Remaining	1 other defect

B05-017	17	Good		Perfect insulation
B05-018	18	Bad	Too many defects	11 open connections & 2 insulation defects
B05-019	19	Good		Defect in macro & mesos; Debris throughout wafer
B05-020	20	Good		Defect in macro & meso

## **32-Channel Arrays**

**Yields** Total: 94 / 120 [78.3% yield]

#### Good Arrays

Meso/Macro Defects: 24 / 94 [25.5% of good arrays]

Perfect Metal: 59 / 94 [62.8% of good arrays]

Perfect Insulation: 59 / 94 [62.8% of good arrays]

Perfect Arrays: 35 / 94 [37.2% of good arrays]

#### **Bad Arrays**

Insulation Remaining: 6 / 26 [23.1% of bad arrays] but would otherwise be considered Good

## **Results by Array**

Wafer ID	Array #	Good/Bad	Reason	Notes
	1	Reworkable	Insulation Remaining (Electrodes)	0 other defects; Possibly etched polyimide
	2	Good		Perfect metal; Possibly etched polyimide
D01 001	3	Good		Perfect metal; Possibly etched polyimide
B01-001	4	Good		Perfect metal; Possibly etched polyimide
	5	Bad	Too many defects	10 defects; Possibly etched polyimide
	6	Good		Defect in macro; Possibly etched polyimide
	7	Bad	Too many defects	18+ open connections
	8	Bad	Too many defects	8 open connections
B01 000	9	Good		Perfect array
B01-002	10	Good		Perfect metal
	11	Bad	Too many defects	7 insulation defects
	12	Bad	Opens in macro & meso	3 open connections (including the macro and a meso)
	13	Reworkable	Insulation Remaining (Electrodes)	0 other defects
	14	Good		Perfect array
<b>DO1 000</b>	15	Good		Perfect array
B01-005	16	Good		Perfect metal; Defect in meso
	17	Good		Perfect array
	18	Good		Perfect array
	19	Bad	Insulation Tears	Insulation Tear (Connector & Electrodes); Insulation Remaining; 2 other defects
	20	Good		Perfect array
B01-004	21	Good		Perfect array
	22	Good		Perfect array
	23	Good		Perfect metal; Defect in macro
	24	Good		Perfect array
	25	Good		Perfect array
	26	Bad	Opens in macro	3 open connections (including the macro)
D02.005	27	Good		Perfect metal
B02-005	28	Good		Perfect array
	29	Good		Perfect metal
1	30	Good		Perfect metal; Defect in mesos
	31	Bad	Too many defects	Insulation remaining; Trace defects affecting all channels
<b>DO3 000</b>	32	Good		Perfect array
R02-006	33	Good		Perfect array
	34	Good		Perfect array

	35	Good		Perfect array
	36	Good		Perfect array
	37	Good		Perfect insulation: Defect in macro & meso
	38	Good		Perfect array
	20	Ded	Tee menu defects	10 metal and insulation defeate
B02-007	39	Bdu	Too many defects	10 metal and insulation defects
	40	Good		Perfect metal
	41	Good		Perfect metal; Defect in macro * meso
	42	Bad	Too many defects	19 metal defects (opens and shorts)
	43	Good		Perfect metal; Debris throughout array
	44	Good		Debris throughout array
	45	Good		Perfect metal: Debris throughout array
B02-008	15	Good		Defect in mese: Debris throughout array
	40	Good		Defect milleso, Debris throughout array
	47	GOOU		Perfect metal; Debris throughout array
	48	Good		Perfect metal; Debris throughout array
	49	Bad	Too many defects	16 metal defects from hair (opens and shorts)
	50	Good		Perfect array
<b>DOD 000</b>	51	Bad	Too many defects	11 metal and insulation defects
B03-009	52	Bad	Too many defects	9 insulation defects
	53	Good		Perfect array
	54	Good		Perfect array
	54	Good		Perfect insulation
	55	Good		Perfect Insulation
	56	Good		Perfect insulation
B03-010	57	Good		Perfect array
505 010	58	Good		Perfect insulation; Defect in meso
	59	Good		Perfect array
	60	Good		Perfect array
	61	Good		Defect in meso
	62	Good		Perfect array
	63	Good	1	Perfect metal
B03-011	03	Guud		Perfect Inicial
	64	Good		Perfect array
	65	Good		Perfect array
	66	Good		Perfect metal; Defect in meso
	67	Good		Hair under metal
	68	Good		Perfect insulation
	69	Good		Perfect array
B03-012	70	Good		Perfect insulation: Defect in meso
	71	Good		Perfect array
	71	Good		Perfect metal
	72	Guu	landation Trans (Comparison)	Perfect filetal
	73	Bdu	insulation rear (connector)	2 Other derects; Possibly etched polyimide
	74	Good		Perfect metal; Possibly etched polyimide
B04-013	75	Good		Perfect metal; Possibly etched polyimide
	76	Good		Defect in meso; Possibly etched polyimide
	77	Good		Perfect metal; Possibly etched polyimide
	78	Bad	Too many defects	8 metal defects; Possibly etched polyimide
	79	Reworkable	Insulation Remaining (Connector)	2 other defects
	80	Good		Perfect insulation
	81	Good		Perfect array
B04-014	82	Good		Perfect insulation: Defect in meso
	02	Good		Berfect array
	03	Good		Perfect inculation: Defect in mass
	64	Good		Perfect insulation; Defect in meso
	85	Good		Perfect insulation; Defect in meso
	86	Good		Perfect insulation; Defect in meso
B04-015	87	Good		Perfect array
501 015	88	Bad	Too many defects	Possible trace defects in all traces (scratches)
	89	Good		Perfect insulation
	90	Bad	Too many defects	19 open or shorted connections
	91	Bad	Too many defects	6 defects (including the macro)
	92	Good	·	Perfect insulation
	93	Good		Perfect array
B04-016	04	Good		Perfect insulation: Defect in more
	54	Good		Perfect insulation, Delect in meso
	95	GOOU		Periect array
	96	Bad	Too many defects	20 metal defects; insulation Remaining
	97	Good		Perfect insulation; Defect in macro
	98	Good		Perfect insulation
POE 017	99	Good		Perfect array
803-017	100	Good		Perfect insulation; Defect in meso
	101	Good		Perfect metal; Defect in meso
	102	Reworkable	Insulation Remaining (Electrodes)	4 other defects
	103	Good		Perfect insulation
	105	Reworkabla	Insulation Remaining (Electrodes)	2 other defects
1	111/1	INC WUINDUE	moulation nemaning (Electrodes)	2 Other derects
	104	Dougo de la la	Inculation Domaining (Contract)	O other defects
B05-018	104	Reworkable	Insulation Remaining (Connector)	0 other defects
B05-018	104 105 106	Reworkable Good	Insulation Remaining (Connector)	0 other defects Perfect insulation; Defect in meso
B05-018	104 105 106 107	Reworkable Good Bad	Insulation Remaining (Connector) Too many defects	0 other defects Perfect insulation; Defect in meso 18 metal defects
B05-018	104 105 106 107 108	Reworkable Good Bad Bad	Insulation Remaining (Connector) Too many defects Too many defects	0 other defects Perfect insulation; Defect in meso 18 metal defects 24 metal defects; Insulation Remaining
B05-018	104 105 106 107 108 109	Reworkable Good Bad Bad Bad	Insulation Remaining (Connector) Too many defects Too many defects Too many defects Too many defects	0 other defects Perfect insulation; Defect in meso 18 metal defects 24 metal defects; Insulation Remaining 8 metal defects; Debris throughout array
B05-018	104 105 106 107 108 109 110	Reworkable Good Bad Bad Bad Bad	Insulation Remaining (Connector) Too many defects	0 other defects Perfect insulation; Defect in meso 18 metal defects 24 metal defects; Insulation Remaining 8 metal defects; Debris throughout array 13 metal and insulation defects; Debris throughout array
B05-018 B05-019	104 105 106 107 108 109 110 111	Reworkable Good Bad Bad Bad Bad Good	Insulation Remaining (Connector) Too many defects	0 other defects Perfect insulation; Defect in meso 18 metal defects 24 metal defects; Insulation Remaining 8 metal defects; Debris throughout array 13 metal and insulation defects; Debris throughout array Perfect metal: Debris throughout array
B05-018 B05-019	104 105 106 107 108 109 110 111 111	Reworkable Good Bad Bad Bad Good Good	Insulation Remaining (Connector) Too many defects	0 other defects Perfect insulation; Defect in meso 18 metal defects 24 metal defects; Insulation Remaining 8 metal defects; Debris throughout array 13 metal and insulation defects; Debris throughout array Perfect metal; Debris throughout array Defect in meso; Debris throughout array

	113	Good	Perfect metal; Debris throughout array
	114	Good	Debris throughout array
	115	Good	Perfect insulation
	116	Good	Perfect insulation; Defect in macro & meso
POE 020	117	Good	Perfect insulation
B05-020	118	Good	Perfect insulation; Defect in meso
	119	Good	Perfect array
	120	Good	Perfect insulation

## **Microscope Evaluation**

## B01-001

#### 128-Channel Array #1

- Consideration: <u>Good Array</u>, but possibly etched polyimide [7 defects]
- Metal Defects: Shorts near BL connector (u8-u11)
- Insulation Defects: Bubble at BR connector (19NC, GND, u31, u29); Possible etched polyimide



128-1 – (Left) Shorts in connector of BL section across u8-u11; (Right) Bubble at connector pads of BR section across u29-u31.

## 32-Channel Array #1 (Top of Top Section)

- Consideration: <u>Reworkable Array (Insulation Remaining)</u>, possibly etched polyimide [0 other defects]
- Metal Defects: N/A
- Insulation Defects: Insulation remains on electrodes (m1, m16, M17, m32); Possible etched polyimide



32-1 – Insulation remaining on electrodes.

#### 32-Channel Array #2 (Middle of Top Section)

- Consideration: Good Array, but possibly etched polyimide [0 defects]
- Metal Defects: N/A
- Insulation Defects: Possible etched polyimide

#### 32-Channel Array #3 (Bottom of Top Section)

- Consideration: Good Array, but possibly etched polyimide [3 defects]
- Metal Defects: N/A
- Insulation Defects: Bubble near connector (u10-u12); Possible etched polyimide



32-3 – Bubble near connector section, across u10-u12.

#### 32-Channel Array #4 (Top of Bottom Section)

• Consideration: Good Array, but possibly etched polyimide [4 defects]

- Metal Defects: N/A
- Insulation Defects: Bubble/Hole near connector (u25-u28); Possible etched polyimide



32-4 – Bubble/Hole near connector section across u25-u28.

## 32-Channel Array #5 (Middle of Bottom Section)

- Consideration: **Bad Array**, but possibly etched polyimide [10 defects]
- Metal Defects: Opens in tail (GND, m1, u2-u7)
- Insulation Defects: Bubble in tail (u6-u10); Possible etched polyimide



32-5 – (Left) Opens in tail section across m1 and u2-u7; (Right) Bubble in tail section across u6u10.

## 32-Channel Array #6 (Bottom of Bottom Section)

- Consideration: <u>Good Array</u>, but possibly etched polyimide [1 defect \*macro]
- Metal Defects: Tear in electrode (M17)
- Insulation Defects: Possible etched polyimide



32-6 – Tear in metal at macroelectrode (M17).

## **B01-002**

128-Channel Array #2

- Consideration: Bad Array (Accidental hole in tail from laser) [2 prior defects, 23 current defects - \*mesos]
- Metal Defects: Open in BL tail (m32); Hole in TR tail from laser (m1, u2-u10); Hole in BR tail from laser (u18-u24, m17)
- Insulation Defects: Bubble near TL electrodes (m2)



128-2 – (Left) Open in tail of BL section at m32; (Right) Bubble near electrodes of TL section at m2.

## 32-Channel Array #7 (Top of Top Section)

- Consideration: Bad Array [18+ defects]
- Metal Defects: Opens in tail (u3-u6, u8-u12, u14-u15, m16, M17, m18, u19, u30-u31, m32, 39NC, GND [x3])
- Insulation Defects: N/A



32-7 – (Left) Opens in tail across u3-u6, u8-u10, u12, u14, u15, m16, M17, m18, u19, u30, u31, and m32; (Right) Opens in tail across u11, u15, and m16.

## 32-Channel Array #8 (Middle of Top Section)

- Consideration: Bad Array [8 defects]
- Metal Defects: Opens in tail (u20-u26, u28)
- Insulation Defects: N/A



32-8 – Opens in tail across u20-u26 and u28.

## 32-Channel Array #9 (Bottom of Top Section)

- Consideration: Good Array [0 defects]
- Metal Defects: N/A
- Insulation Defects: N/A

#### 32-Channel Array #10 (Top of Bottom Section)

- Consideration: Good Array [5 defects]
- Metal Defects: N/A
- Insulation Defects: Bubble near electrodes (u9-u12)



32-10 – Bubble near electrodes across u9-u12.

## 32-Channel Array #11 (Middle of Bottom Section)

• Consideration: Bad Array [7 defects]

- Metal Defects: N/A
- Insulation Defects: Bubbles/Holes in tail (u19-u25)



32-11 – (Left) Bubbles/Holes in tail across u19-u25; (Right) Bubbles/Holes in tail across u21-u23.

## 32-Channel Array #12 (Bottom of Bottom Section)

- Consideration: Bad Array [3 defects \*macro/meso]
- Metal Defects: Opens in tail (M17, m18, u19, GND, 39NC)
- Insulation Defects: N/A



32-12 – Opens in tail across M17, m18, and u19.

## **B01-003**

## 128-Channel Array #3

- Consideration: Good Array [14 defects \*macro/meso]
- Metal Defects: Dimples/Bubbles in TR macro (M17)
- Insulation Defects: Bubble at BR electrodes (M1, m2, u3-u13)



128-3 – (Left) Dimples/Bubbles in metal at electrodes of TR section on M17; (Right) Bubble at electrodes of BR section across M1, m2, u3-u8, and u9-u13.

## 32-Channel Array #13 (Top of Top Section)

- Consideration: Reworkable Array (Insulation Remaining) [0 other defects]
- Metal Defects: N/A
- Insulation Defects: Insulation remains on electrodes (m16, M17, m32)



32-13 – Insulation remaining on electrodes.

#### 32-Channel Array #14 (Middle of Top Section)

- Consideration: Good Array [0 defects]
- Metal Defects: N/A
- Insulation Defects: N/A

#### 32-Channel Array #15 (Bottom of Top Section)

- Consideration: Good Array [0 defects]
- Metal Defects: N/A
- Insulation Defects: N/A

## 32-Channel Array #16 (Top of Bottom Section)

- Consideration: Good Array [4 defects \*meso]
- Metal Defects: N/A
- Insulation Defects: Bubble in tail (m1, u2-u4)



32-16 – Bubble in tail across m1 and u2-u4.

## 32-Channel Array #17 (Middle of Bottom Section)

- Consideration: Good Array [0 defects]
- Metal Defects: N/A
- Insulation Defects: N/A

## 32-Channel Array #18 (Bottom of Bottom Section)

- Consideration: Good Array [0 defects]
- Metal Defects: N/A
- Insulation Defects: N/A

## **B01-004**

#### **128-Channel Array #4**

- Consideration: Good Array [2 defects]
- Metal Defects: N/A
- Insulation Defects: Hole in BL tail (u23-u24)



128-4 – Hole in tail of BL section across u23 and u24.

## 32-Channel Array #19 (Top of Top Section)

- Consideration: Bad Array (Insulation Tears) [2 other defects]
- Metal Defects: Opens in tail (u29-u30)
- Insulation Defects: Insulation tear near connector; Insulation tear near electrodes



32-19 – (Left) Opens in tail across u29 and u30; (Center) Tear in insulation near connectors; (Right) Insulation remaining on electrodes and tear in insulation near electrodes.

#### 32-Channel Array #20 (Middle of Top Section)

- Consideration: Good Array [0 defects]
- Metal Defects: N/A
• Insulation Defects: N/A

### 32-Channel Array #21 (Bottom of Top Section)

- Consideration: Good Array [0 defects]
- Metal Defects: N/A
- Insulation Defects: N/A

#### 32-Channel Array #22 (Top of Bottom Section)

- Consideration: Good Array [0 defects]
- Metal Defects: N/A
- Insulation Defects: N/A

### 32-Channel Array #23 (Middle of Bottom Section)

- Consideration: Good Array [1 defect \*macro]
- Metal Defects: N/A
- Insulation Defects: Bubble/Hole in tail (M17)



32-23 – Bubble/Hole in tail across M17.

32-Channel Array #24 (Bottom of Bottom Section)

- Consideration: Good Array [0 defects]
- Metal Defects: N/A
- Insulation Defects: N/A

## **B02-005**

#### **128-Channel Array #5**

- Consideration: Good Array [4 defects \*macro/meso]
- Metal Defects: Tears in TR electrodes (m16, M17, m32)
- Insulation Defects: Spot in insulation on BR electrode (m32)



128-5 – (Left) Tears in electrodes of TR section across m16, M17, and m32; (Right) Spot near electrodes of the BR section on m32.

#### 32-Channel Array #25 (Top of Top Section)

- Consideration: Good Array [0 defects]
- Metal Defects: N/A
- Insulation Defects: N/A

## 32-Channel Array #26 (Middle of Top Section)

- Consideration: Bad Array [3 defects \*macro]
- Metal Defects: Opens near electrodes (M17, u25-u26, GND, 39NC)
- Insulation Defects: N/A



32-26 – Opens near electrodes across M17, u25, and u26.

## 32-Channel Array #27 (Bottom of Top Section)

- Consideration: Good Array [4 defects]
- Metal Defects: N/A
- Insulation Defects: Bubble/Hole at connector (u2-u5)



32-27 – Bubble/Hole at connector across u2-u5.

### 32-Channel Array #28 (Top of Bottom Section)

- Consideration: Good Array [0 defects]
- Metal Defects: N/A
- Insulation Defects: N/A

## 32-Channel Array #29 (Middle of Bottom Section)

- Consideration: Good Array [1 defect]
- Metal Defects: N/A
- Insulation Defects: Bubble/Hole at electrodes (u2)



32-29 – Bubble/Hole at electrodes on u2.

## 32-Channel Array #30 (Bottom of Bottom Section)

- Consideration: Good Array [2 defects \*mesos]
- Metal Defects: N/A
- Insulation Defects: Bubble/Spot in tail (m32); Spot at electrodes (m1)



32-30 – (Left) Bubble/Spot in tail on m32; (Right) Spot at electrodes on m1.

# **B02-006**

## 128-Channel Array #6

- Consideration: Bad Array (Insulation Remaining) [1 other defect \*macro]
- Metal Defects: N/A
- Insulation Defects: Hole at TR connector (M17)



128-6 – (Left) Hole at connectors of TR section on M17; (Right) Insulation remaining on connectors.

## 32-Channel Array #31 (Top of Top Section)

- Consideration: Bad Array (Insulation Remaining) [32 other defects]
- Metal Defects: Trace defects in tail (m1, u2-u15, m16)
- Insulation Defects: Bubble/Hole at connectors (u15, m16); Insulation remains at connectors



32-31 – (Left) Open in tail (GND) and trace defects throughout m1, u2-u15, m16, m18, u19-u31, m32; (Right) Insulation remaining on some connectors as well as a Bubble/Hole near connectors across u15 and m16.

### 32-Channel Array #32 (Middle of Top Section)

- Consideration: Good Array [0 defects]
- Metal Defects: N/A
- Insulation Defects: N/A

#### 32-Channel Array #33 (Bottom of Top Section)

- Consideration: Good Array [0 defects]
- Metal Defects: N/A
- Insulation Defects: N/A

#### 32-Channel Array #34 (Top of Bottom Section)

- Consideration: Good Array [0 defects]
- Metal Defects: N/A
- Insulation Defects: N/A

### 32-Channel Array #35 (Middle of Bottom Section)

- Consideration: Good Array [0 defects]
- Metal Defects: N/A
- Insulation Defects: N/A

### 32-Channel Array #36 (Bottom of Bottom Section)

- Consideration: Good Array [0 defects]
- Metal Defects: N/A
- Insulation Defects: N/A

### **B02-007**

#### 128-Channel Array #7

- Consideration: Good Array [8 defects \*meso]
- Metal Defects: Opens at TL connector (u24); Opens at TR electrodes (u4)

Insulation Defects: Bubble at TR connector (u9-u10); Bubble at BR connector (m32, u29-u31)





128-7 – (Top Left) Open at connectors of the TL section at u24; (Top Right) Open at electrodes of the TR section at u4; (Bottom Left) Bubble/Hole near connector of the TR section across u9-u10; (Bottom Right) Bubble/Hole near connector of the BR section across u29-u31 and m32.

## 32-Channel Array #37 (Top of Top Section)

- Consideration: Good Array [6 defects \*macro/meso]
- Metal Defects: Opens at connectors (u8, u31); Opens in tail (M17, u25-u26, m32)
- Insulation Defects: N/A



32-37 – (Left) Opens at connectors at u8 and u31; (Center) Opens in tail at M17, u25, and u26; (Right) Open in tail at m32.

### 32-Channel Array #38 (Middle of Top Section)

- Consideration: Good Array [0 defects]
- Metal Defects: N/A
- Insulation Defects: N/A

### 32-Channel Array #39 (Bottom of Top Section)

- Consideration: Bad Array [10 defects \*mesos]
- Metal Defects: Opens in tail (u13-u15, m16); Debris in tail (u28-u31, m32)
- Insulation Defects: Spot near electrodes (u28-u30)



32-39 – (Left) Opens in tail across u13-u15 and m16 as well as Debris across u28-u31 and m32; (Right) Spot near electrodes across u28-u30.

# 32-Channel Array #40 (Top of Bottom Section)

- Consideration: Good Array [2 defects]
- Metal Defects: N/A
- Insulation Defects: Spot/Hole in tail (u5-u6)



32-40 – Spot/Hole in tail across u5-u6.

### 32-Channel Array #41 (Middle of Bottom Section)

- Consideration: Good Array [3 defects \*macro/meso]
- Metal Defects: N/A
- Insulation Defects: Spot near connector (M17, m18, u19)



32-41 – Spot near connector across M17, m18, and u19.

### 32-Channel Array #42 (Bottom of Bottom Section)

- Consideration: Bad Array [19 defects \*macro/meso]
- Metal Defects: Opens in tail (M17, m18); Open in tail (u23); Opens in tail (u3, u27, u28); Shorts in tail (u12-u15, m16); Shorts in tail (u19-u26); Open in tail (m18)

• Insulation Defects: N/A





32-42 – (Top Left) Opens in tail across M17 and m18; (Top Right) Open in tail at u23; (Bottom Left) Opens in tail across u3, u27, and u28 as well as Shorts in tail across u12-u15 and m16 as well as Shorts in tail across u19-u26; (Bottom Right) Open in tail at m18.

## **B02-008**

#### 128-Channel Array #8

- Consideration: Good Array [8 defects]
- Metal Defects: Open in TR tail (u20)

• Insulation Defects: Spots near TR electrodes (u13-u15, u25-u27); Spot near BR connector (u21)



128-8 – (Left) Open in tail of TR section at u20; (Center) Spots in electrodes of TR section across u13-u15 and u25-u27; (Right) Spot at connector of BR section at u21.

### 32-Channel Array #43 (Top of Top Section)

- Consideration: Good Array [0 defects]
- Metal Defects: N/A
- Insulation Defects: N/A

### 32-Channel Array #44 (Middle of Top Section)

- Consideration: Good Array [2 defects]
- Metal Defects: Open in tail (u28); Short in tail (u28-u29)
- Insulation Defects: N/A



32-44 – Open in tail at u28 as well as a Short in tail between u28 and u29.

## 32-Channel Array #45 (Bottom of Top Section)

- Consideration: Good Array [5 defects]
- Metal Defects: N/A
- Insulation Defects: Bubble/Hole in tail (u2-u6)



32-45 – Bubble/Hole in tail across u2-u6.

## 32-Channel Array #46 (Top of Bottom Section)

- Consideration: Good Array [1 defect \*meso]
- Metal Defects: Possible short in tail (m16, GND)
- Insulation Defects: N/A

	and the second se
And the second diversity of th	
	the second s
	and the second
	and the second
	and the second
And the second	
	the second s

32-46 – Possible short in tail across m16 and GND.

## 32-Channel Array #47 (Middle of Bottom Section)

- Consideration: Good Array [0 defects]
- Metal Defects: N/A
- Insulation Defects: N/A

## 32-Channel Array #48 (Bottom of Bottom Section)

- Consideration: Good Array [0 defects]
- Metal Defects: N/A
- Insulation Defects: N/A

# B03-009

### 128-Channel Array #9

- Consideration: Good Array [10 defects \*meso]
- Metal Defects: N/A
- Insulation Defects: Bubbles/Holes in BL tail (u30-u31); Bubbles/Holes in TR tail (m16, u14-u15); Bubble/Hole in BR tail (u25-u26); Bubble/Hole in BR tail (u27-u29)





128-9 – (Top Left) Bubbles/Holes in tail of BL section across u30 and u31; (Top Right)
Bubble/Hole in tail of TR section across m16, u15, and u14; (Bottom Left) Bubble/Hole in tail of BR section across u25-u26; (Bottom Right) Bubble/Hole in tail of BR section across u27-u29.

### 32-Channel Array #49 (Top of Top Section)

- Consideration: Bad Array [16 defects \*macro/meso]
- Metal Defects: Shorts in tail from hair (M17, m18, u19, u22-u26); Opens in tail from hair (M17, m18, u19-u21, u26-u30)
- Insulation Defects: N/A



32-49 – Metal defects from a hair in the tail include Shorts across M17, m18, and u19, Shorts across u22-u26, and Opens across M17, m18, u19-u21, and u26-u30.

## 32-Channel Array #50 (Middle of Top Section)

- Consideration: Good Array [0 defects]
- Metal Defects: N/A
- Insulation Defects: N/A

### 32-Channel Array #51 (Bottom of Top Section)

- Consideration: Bad Array [11 defects \*macro/meso]
- Metal Defects: Opens in tail (u24-u27)
- Insulation Defects: Bubbles/Holes in tail (M17, m18, u19-u21, u24-u25)



32-51 – (Left) Opens in tail across u24-u27; (Right) Bubbles/Holes in tail across M17, m18, u19u21, and u24-u25.

### 32-Channel Array #52 (Top of Bottom Section)

• Consideration: Bad Array [9 defects - \*meso]

- Metal Defects: N/A
- Insulation Defects: Bubbles/Holes in tail (m32, u29-u31); Spot in tail (u4-u5); Bubbles/Holes in tail (u21-u23)



32-52 – (Left) Bubbles/Holes in tail across m32 and u29-u31; (Center) Spot in tail across u4-u5; (Right) Bubbles/Holes in tail across u21-u23.

## 32-Channel Array #53 (Middle of Bottom Section)

- Consideration: Good Array [0 defects]
- Metal Defects: N/A
- Insulation Defects: N/A

### 32-Channel Array #54 (Bottom of Bottom Section)

- Consideration: Good Array [0 defects]
- Metal Defects: N/A
- Insulation Defects: N/A

# B03-010

### 128-Channel Array #10

- Consideration: Good Array [0 defects]
- Metal Defects: N/A
- Insulation Defects: N/A

### 32-Channel Array #55 (Top of Top Section)

- Consideration: Good Array [1 defect]
- Metal Defects: Metal defect at connector (u5)
- Insulation Defects: N/A



32-55 – Metal defect at connector of u5 (possible short across u4-u5).

### 32-Channel Array #56 (Middle of Top Section)

- Consideration: Good Array [2 defects]
- Metal Defects: Short in tail (u28-u29)
- Insulation Defects: N/A



32-56 – Short in tail across u28-u29.

## 32-Channel Array #57 (Bottom of Top Section)

- Consideration: Good Array [0 defects]
- Metal Defects: N/A
- Insulation Defects: N/A

### 32-Channel Array #58 (Top of Bottom Section)

- Consideration: Good Array [1 defect \*meso]
- Metal Defects: Possible short in tail (m16-GND)
- Insulation Defects: N/A



32-58 – Possible short in tail across m16 and GND.

## 32-Channel Array #59 (Middle of Bottom Section)

- Consideration: Good Array [0 defects]
- Metal Defects: N/A

• Insulation Defects: N/A

### 32-Channel Array #60 (Bottom of Bottom Section)

- Consideration: Good Array [0 defects]
- Metal Defects: N/A
- Insulation Defects: N/A

# B03-011

## 128-Channel Array #11

- Consideration: Bad Array [15 defects]
- Metal Defects: Metal defects in TL tail from hair (u25-u29); Short near TR connector (u2-u3)
- Insulation Defects: Holes in TL tail (u21-u24); Spots in BL tail (u8); Hole in BR tail (u11-u12); Spot in BR tail (u12-u13)



128-11 – (Top Left) Metal defects in tail of TL section from hair across u25-u29; (Top Center) Short near connector of TR section across u2-u3; (Top Right) Holes in tail of TL section across

u21-u24; (Bottom Left) Spots in tail of BL section on u8; (Bottom Right) Hole across u11-u12 as well as a Spot across u12-u13 in tail of BR section.

## 32-Channel Array #61 (Top of Top Section)

- Consideration: Good Array [4 defects]
- Metal Defects: Metal defect at connector (u5)
- Insulation Defects: Holes at connector (m16, u14-u15)



32-61 – Defects at connector include a metal defect across u5 and holes across m16 and u14-

u15.

### 32-Channel Array #62 (Middle of Top Section)

- Consideration: Good Array [0 defects]
- Metal Defects: N/A
- Insulation Defects: N/A

### 32-Channel Array #63 (Bottom of Top Section)

- Consideration: Good Array [1 defect]
- Metal Defects: N/A
- Insulation Defects: Hole at connector (u31)



32-63 – Hole at connector on u31.

### 32-Channel Array #64 (Top of Bottom Section)

- Consideration: Good Array [0 defects]
- Metal Defects: N/A
- Insulation Defects: N/A

#### 32-Channel Array #65 (Middle of Bottom Section)

- Consideration: Good Array [0 defects]
- Metal Defects: N/A
- Insulation Defects: N/A

#### 32-Channel Array #66 (Bottom of Bottom Section)

- Consideration: Good Array [5 defects \*meso]
- Metal Defects: N/A
- Insulation Defects: Hole in tail (m1, u2-u5)



32-66 – Hole in tail across m1 and u2-u5.

## **B03-012**

## 128-Channel Array #12

- Consideration: Bad Array [23 defects]
- Metal Defects: Metal defect from debris at TL connector (m17); Short at TR electrodes (u12-u15, m16, M17, m18, u19-u31, m32, GND); Opens at TR electrodes (M17, m18, u19, u21-u27, u29)
- Insulation Defects: Spot on BL electrodes (u4)



128-12 – (Left) Metal defect from debris at connector of TL section (m17); (Center) Defects at electrodes of TR section include Shorts across u12-u15, m16, M17, m18, u19-u31, m32, and GND as well as Opens across M17, m18, u19, u21-u27, and u29; (Right) Spot at electrodes of BL section on u4.

### 32-Channel Array #67 (Top of Top Section)

- Consideration: Good Array [6 defects]
- Metal Defects: Metal defect at connector (u5); Metal defect from hair (u12-u13); Trace defect at electrodes (u28)
- Insulation Defects: Hole in tail (u19-u20)

### 32-Channel Array #68 (Middle of Top Section)

- Consideration: Good Array [2 defects]
- Metal Defects: Short in tail (u28-u29)
- Insulation Defects: N/A

### 32-Channel Array #69 (Bottom of Top Section)

- Consideration: Good Array [0 defects]
- Metal Defects: N/A
- Insulation Defects: N/A

### 32-Channel Array #70 (Top of Bottom Section)

- Consideration: Good Array [1 defect]
- Metal Defects: Possible short in tail (m16-GND)
- Insulation Defects: N/A

### 32-Channel Array #71 (Middle of Bottom Section)

- Consideration: Good Array [1 defect]
- Metal Defects: Open in tail (u24)
- Insulation Defects: N/A

## 32-Channel Array #72 (Bottom of Bottom Section)

- Consideration: Good Array [0 defects]
- Metal Defects: N/A
- Insulation Defects: N/A

## **B04-013**

### 128-Channel Array #13

- Consideration: Good Array, but possibly etched polyimide [0 defects]
- Metal Defects: N/A
- Insulation Defects: Possible etched polyimide

### 32-Channel Array #73 (Top of Top Section)

- Consideration: Bad Array (Insulation Tear), possibly etched polyimide [2 other defects]
- Metal Defects: Short at connector (u4-u5)
- Insulation Defects: Insulation ear near connector; Possible etched polyimide

### 32-Channel Array #74 (Middle of Top Section)

- Consideration: Good Array, but possibly etched polyimide [0 defects]
- Metal Defects: N/A
- Insulation Defects: Possible etched polyimide

### 32-Channel Array #75 (Bottom of Top Section)

- Consideration: Good Array, but possibly etched polyimide [0 defects]
- Metal Defects: N/A

• Insulation Defects: Possible etched polyimide

### 32-Channel Array #76 (Top of Bottom Section)

- Consideration: Good Array, but possibly etched polyimide [1 defect \*meso]
- Metal Defects: Possible short in tail (m16-GND)
- Insulation Defects: Possible etched polyimide

#### 32-Channel Array #77 (Middle of Bottom Section)

- Consideration: Good Array, but possibly etched polyimide [0 defects]
- Metal Defects: N/A
- Insulation Defects: Possible etched polyimide

### 32-Channel Array #78 (Bottom of Bottom Section)

- Consideration: Bad Array, possibly etched polyimide [8 defects \*macro/meso]
- Metal Defects: Short in tail (m32, u31); Opens in tail (m18, u19); Shorts in tail (GND, M17, m18, u19-u20)
- Insulation Defects: Possible etched polyimide

## **B04-014**

#### **128-Channel Array #14**

- Consideration: Reworkable Array (Insulation Remaining) [2 other defects]
- Metal Defects: Metal defects in TL tail (u7); Metal defect at TR connector (u22)
- Insulation Defects: Insulation remains on connectors

#### 32-Channel Array #79 (Top of Top Section)

- Consideration: Reworkable Array (Insulation Remaining) [2 other defects]
- Metal Defects: Short at connector (u4-u5)
- Insulation Defects: Insulation remains on connectors

#### 32-Channel Array #80 (Middle of Top Section)

- Consideration: Good Array [1 defect]
- Metal Defects: Metal defect in tail (u31)
- Insulation Defects: N/A

### 32-Channel Array #81 (Bottom of Top Section)

- Consideration: Good Array [0 defects]
- Metal Defects: N/A
- Insulation Defects: N/A

### 32-Channel Array #82 (Top of Bottom Section)

- Consideration: Good Array [1 defect \*meso]
- Metal Defects: Possible short in tail (m16-GND)
- Insulation Defects: N/A

### 32-Channel Array #83 (Middle of Bottom Section)

- Consideration: Good Array [0 defects]
- Metal Defects: N/A
- Insulation Defects: N/A

### 32-Channel Array #84 (Bottom of Bottom Section)

- Consideration: Good Array [2 defects \*meso]
- Metal Defects: Short in tail (m32, u31)
- Insulation Defects: N/A

# **B04-015**

#### **128-Channel Array #15**

- Consideration: Good Array [6 defects]
- Metal Defects: Metal defects in TL tail (m17); Trace defects in BL tail (M17, m18, u19); Trace defects in TR connectors (u11, u22)
- Insulation Defects: N/A

### 32-Channel Array #85 (Top of Top Section)

- Consideration: Good Array [4 defects \*meso]
- Metal Defects: Short at connector (u4-u5); Opens at connector (m1, u2)
- Insulation Defects: N/A

### 32-Channel Array #86 (Middle of Top Section)

- Consideration: Good Array [3 defects \*meso]
- Metal Defects: Trace defect from debris (m16); Trace defect in tail (u29-u30)
- Insulation Defects: N/A

### 32-Channel Array #87 (Bottom of Top Section)

- Consideration: Good Array [0 defects]
- Metal Defects: N/A
- Insulation Defects: N/A

### 32-Channel Array #88 (Top of Bottom Section)

- Consideration: Bad Array [32 possible defects]
- Metal Defects: Possible trace defects in tail (ALL); Possible short in tail (m16-GND)
- Insulation Defects: N/A

### 32-Channel Array #89 (Middle of Bottom Section)

- Consideration: Good Array [2 defects]
- Metal Defects: Trace defect in tail (u20); Trace defect in tail (u6)
- Insulation Defects: N/A

### 32-Channel Array #90 (Bottom of Bottom Section)

- Consideration: Bad Array [19 defects]
- Metal Defects: Trace defects at connector (M17, m18, u19); Short in tail (m32, u31); Opens in tail (m32, u30); Trace defects in tail (u19-u20, u24-u25); Opens in tail (u27, u30); Trace defect in tail (u23); Opens in tail (u9, u30); Trace defects in tail (u2, u14, u25); Opens at electrodes (u5, M17, u19, u21, u23, u25-u31, m32); Trace defects at electrodes (u22, u24)
- Insulation Defects: N/A

## **B04-016**

#### 128-Channel Array #16

- Consideration: Reworkable Array (Insulation Remaining) [1 other defect]
- Metal Defects: Metal defect at TR connector (u22)

• Insulation Defects: Insulation remains on connectors

### 32-Channel Array #91 (Top of Top Section)

- Consideration: Bad Array [6 defects \*macro]
- Metal Defects: Metal defect at connector (u4); Trace defect in tail (M17)
- Insulation Defects: Hole at connector (u23-u24); Spot in tail (u28-u29)

### 32-Channel Array #92 (Middle of Top Section)

- Consideration: Good Array [2 defects]
- Metal Defects: Trace defect at connector (u8); trace defect from debris in tail (u24)
- Insulation Defects: N/A

### 32-Channel Array #93 (Bottom of Top Section)

- Consideration: Good Array [0 defects]
- Metal Defects: N/A
- Insulation Defects: N/A

### 32-Channel Array #94 (Top of Bottom Section)

- Consideration: Good Array [1 defect \*meso]
- Metal Defects: Possible short in tail (m16-GND)
- Insulation Defects: N/A

#### 32-Channel Array #95 (Middle of Bottom Section)

- Consideration: Good Array [0 defects]
- Metal Defects: N/A
- Insulation Defects: N/A

#### 32-Channel Array #96 (Bottom of Bottom Section)

- Consideration: Bad Array [20 defects]
- Metal Defects: Short in tail (m32, u31); Opens in tail (u4-u6); Trace defects in tail (u7-u8, u10-u11, u13-u14, m16, m18, u21-u22, u24, u27-u28, u30, m32); Shorts in tail (u8-u9)
- Insulation Defects: Insulation remains on electrodes;

# **B05-017**

### **128-Channel Array #17**

- Consideration: Good Array [1 defect]
- Metal Defects: Metal defect at TR connector (u27)
- Insulation Defects: N/A

#### 32-Channel Array #97 (Top of Top Section)

- Consideration: Good Array [3 defects \*macro]
- Metal Defects: Short at connector (u4-u5); Tear in electrode (M17)
- Insulation Defects: N/A

### 32-Channel Array #98 (Middle of Top Section)

- Consideration: Good Array [2 defects]
- Metal Defects: Short in tail (u28-u29)
- Insulation Defects: N/A

### 32-Channel Array #99 (Bottom of Top Section)

- Consideration: Good Array [0 defects]
- Metal Defects: N/A
- Insulation Defects: N/A

#### 32-Channel Array #100 (Top of Bottom Section)

- Consideration: Good Array [1 defect \*meso]
- Metal Defects: Possible short in tail (m16-GND)
- Insulation Defects: N/A

#### 32-Channel Array #101 (Middle of Bottom Section)

- Consideration: Good Array [1 defect \*meso]
- Metal Defects: N/A
- Insulation Defects: Bubble/Hole at connector (m1)

#### 32-Channel Array #102 (Bottom of Bottom Section)

- Consideration: Reworkable Array (Insulation Remaining) [4 other defects]
- Metal Defects: Metal defect in tail (u24)
• Insulation Defects: Spot in tail (u3-u5); Insulation remains on all electrodes

## B05-018

#### **128-Channel Array #18**

- Consideration: Bad Array [13 defects \*mesos]
- Metal Defects: Trace defects/Possible opens near TR connector (m1, u2, u7-u9); Metal defect at TR connector (u27); Trace defects/Possible opens at TR connector (m1); Trace defects/Possible opens near BR connector (m1, u2-u5)
- Insulation Defects: Bubble/Hole in BR tail (u25-u26)

## 32-Channel Array #103 (Top of Top Section)

- Consideration: Good Array [2 defects]
- Metal Defects: Short at connector (u4-u5)
- Insulation Defects: N/A

#### 32-Channel Array #104 (Middle of Top Section)

- Consideration: Reworkable Array (Insulation Remaining) [2 other defects]
- Metal Defects: Short in tail (u28-u29)
- Insulation Defects: Insulation remains on electrodes

#### 32-Channel Array #105 (Bottom of Top Section)

- Consideration: Reworkable Array (Insulation Remaining) [0 other defects]
- Metal Defects: N/A
- Insulation Defects: Insulation remaining on connector

## **32-Channel Array #106 (Top of Bottom Section)**

- Consideration: Good Array [1 defect \*meso]
- Metal Defects: Possible short in tail (m16-GND)
- Insulation Defects: N/A

## 32-Channel Array #107 (Middle of Bottom Section)

- Consideration: Bad Array [18 defects \*macro/mesos]
- Metal Defects: Trace defects/Possible opens at connector (M17, m18, u19-u24); Trace defects/Possible opens (m1, u2, u4-u10); Trace defect in tail (u11)
- Insulation Defects: N/A

## 32-Channel Array #108 (Bottom of Bottom Section)

- Consideration: Bad Array [24 defects]
- Metal Defects: Metal defect in tail (u24); Trace defects/Possible opens in tail (u4-u5, u7u15, m16, u21-u31, m32); Trace defects/Possible opens in tail (m16, u11-u15)
- Insulation Defects: Insulation remains on all electrodes

## B05-019

## 128-Channel Array #19

- Consideration: Good Array [10 defects \*macro/mesos]
- Metal Defects: Trace defects in TL tail (u9-u10); Tear in TR electrode (M17); Trace defects/Possible opens in TR tail (m16, u15); Metal defects near TR connector (m32, u29-u31); Open at connector (u7)
- Insulation Defects: Debris throughout array

## 32-Channel Array #109 (Top of Top Section)

- Consideration: Bad Array [8 defects]
- Metal Defects: Short at connector (u4-u5); Trace defect in tail (u2); Trace defects in tail (u24-u25); Trace defects in tail (u28-u30)
- Insulation Defects: Debris throughout array

## 32-Channel Array #110 (Middle of Top Section)

- Consideration: Bad Array [13 defects \*macro/meso]
- Metal Defects: Trace defects in tail (m32, u8-u10, u28-u31); Short in tail (u28-u29)
- Insulation Defects: Hole in tail (M17); Hole at electrodes (u2-u5); Debris throughout array

## 32-Channel Array #111 (Bottom of Top Section)

- Consideration: Good Array [0 defects]
- Metal Defects: N/A
- Insulation Defects: Debris throughout array

#### 32-Channel Array #112 (Top of Bottom Section)

- Consideration: Good Array [1 defect \*meso]
- Metal Defects: Possible short in tail (m16-GND)
- Insulation Defects: Debris throughout array

#### 32-Channel Array #113 (Middle of Bottom Section)

- Consideration: Good Array [0 defects]
- Metal Defects: N/A
- Insulation Defects: Debris throughout array

#### 32-Channel Array #114 (Bottom of Bottom Section)

- Consideration: Good Array [1 defect]
- Metal Defects: Metal defect in tail (u24)
- Insulation Defects: Debris throughout array

## B05-020

#### **128-Channel Array #20**

- Consideration: Good Array [10 defects \*macro/meso]
- Metal Defects: Trace defect in BL tail (u28); Short at TR electrodes (u28-u29); Metal defect at TR connector (u27)
- Insulation Defects: Hole near BL connector (m16, u13-u15); Holes at BL electrodes (M17, u12-u13)

#### 32-Channel Array #115 (Top of Top Section)

- Consideration: Good Array [3 defects]
- Metal Defects: Short at connector (u4-u5); Trace defect in tail (u23)
- Insulation Defects: N/A

## 32-Channel Array #116 (Middle of Top Section)

- Consideration: Good Array [4 defects \*macro/meso]
- Metal Defects: Short at connector (M17, m18); Trace defects at connector (M17, m18, u19); Trace defect at connector (u13)
- Insulation Defects: N/A

## 32-Channel Array #117 (Bottom of Top Section)

- Consideration: Good Array [1 defect]
- Metal Defects: Trace defect in tail (u26)
- Insulation Defects: N/A

#### 32-Channel Array #118 (Top of Bottom Section)

- Consideration: Good Array [1 defect \*meso]
- Metal Defects: Possible short in tail (m16-GND)
- Insulation Defects: N/A

#### 32-Channel Array #119 (Middle of Bottom Section)

- Consideration: Good Array [0 defects]
- Metal Defects: N/A
- Insulation Defects: N/A

#### 32-Channel Array #120 (Bottom of Bottom Section)

- Consideration: Good Array [3 defects]
- Metal Defects: Metal defect in tail (u24); Opens near electrodes (u30-u31)
- Insulation Defects: N/A

# Conclusions

# **Application of PEEK Backings**

## Work Instruction

#### Document written by Jon Garich

Images used were screenshots from an instructional video filmed for the purposes of sharing process information with Arizona State University.

Polyether ether ketone (PEEK) was purchased as a 12" x 12" x 0.005" sheet (127  $\mu m$  thick) from McMaster-Carr.

# Contents

Step 1 – Preparation	2
Step 2 – Application	3
Option 1 – Glue Dot on PEEK + Toothpick Spread	4
Option 2 – Glue Dot on PEEK (Without Manual Spread)	9
Option 3 – Toothpick Transfer from Glue Tray to PEEK	LO
Step 3 – Drying	1
Step 4 – Trimming	12

# **Step 1 – Preparation**

- 1. Find a table with about 1 square foot of working space available next to/near a microscope
- 2. Lay the following items out on the table:
  - a. Tweezers (auto-closing or standard)
  - b. Cyanoacrylate (super glue)
  - c. Kimwipes (or equivalent technical wipe with little or no fiber residue)
  - d. Thin film (electrode arrays in this example)
  - e. Platform (~1" height and ~6" width/length)
- 3. Some optional items might be necessary, depending on the application method you choose:
  - a. Toothpick
  - b. Glue "tray" (similar to a paint tray, but can be just a strip of plastic or a concave bowl)
- 4. It helps to lay the thin films out by the platform prior to beginning the following steps, as seen in the screenshot below:



# **Step 2 – Application**

There are three methods for cyanoacrylate dispensing onto the PEEK sections and all three will be highlighted in this document. Screenshots from the supplemental videos on this process are included in Option 1. Options 2 and 3 are just variations to Option 1, but technical difficulties prevented these videos from being saved, so no screenshots will be included in these alternate methods.

## **Option 1 – Glue Dot on PEEK + Toothpick Spread**

a.

a.

This method of application utilizes a toothpick to spread out a drop of cyanoacrylate on the PEEK section prior to film placement. The steps below, with screenshots from the supplemental video for visual aid, will walk through the application steps.

1. Place a piece of PEEK (~0.5-1" width) onto your platform next to a film.



2. Apply a drop of cyanoacrylate onto the center of the PEEK section, near the edge of the platform



3. Using a toothpick, spread out the drop of cyanoacrylate to at least the approximate width of the film's connector section.



a.4. Using tweezers, grab the film (de-insulated metal side up) from about 0.5" back from the connector section.



- 5. With the Kimwipe in your other hand, carefully place the film onto the spread-out drop of cyanoacrylate on the PEEK section apply pressure with the Kimwipe immediately.
  - a. NOTE: The tweezers can be released once the Kimwipe has pressure on the film.



6. Press down on the film with the Kimwipe to soak up excess cyanoacrylate.

b.



7. Blot the corners and edges of the film with fingers from your other hand. Then lift the Kimwipe off the film and blot spots remaining on the film.

a.



8. Remove the Kimwipe from the film and ensure the PEEK is hanging over the edge of the platform to prevent cyanoacrylate from drying under the PEEK. Leave the cyanoacrylate to dry.



## **Option 2 – Glue Dot on PEEK (Without Manual Spread)**

This method of application simples uses a drop of cyanoacrylate on the PEEK section prior to film placement. All process steps from Option 1 remain the same, excluding Step 3. The steps are repeated below, but without visual aid from screenshots.

- 1. Place a piece of PEEK (~0.5-1" width) onto your platform next to a film.
- 2. Apply a drop of cyanoacrylate onto the center of the PEEK section, near the edge of the platform
- 3. Using tweezers, grab the film (de-insulated metal side up) from about 0.5" back from the connector section.
- 4. With the Kimwipe in your other hand, carefully place the film onto the spread-out drop of cyanoacrylate on the PEEK section apply pressure with the Kimwipe immediately.
  - a. NOTE: The tweezers can be released once the Kimwipe has pressure on the film.
- 5. Press down on the film with the Kimwipe to soak up excess cyanoacrylate.
- 6. Blot the corners and edges of the film with fingers from your other hand. Then lift the Kimwipe off the film and blot spots remaining on the film.
- 7. Remove the Kimwipe from the film and ensure the PEEK is hanging over the edge of the platform to prevent cyanoacrylate from drying under the PEEK. Leave the cyanoacrylate to dry.

## **Option 3 – Toothpick Transfer from Glue Tray to PEEK**

This method of application utilizes a toothpick to transfer and spread out cyanoacrylate from a tray to the PEEK section prior to film placement. Using this technique requires more components, but allows for the cyanoacrylate dispersion to be uniform and the quantity dispensed is more precise. Some of the process steps (steps 1 and 4-8) from Option 1 are still used here, but the dispensing steps (steps 2 and 3) are different. No screenshots were used for visual aid in this section.

- 1. Place a piece of PEEK (~0.5-1" width) onto your platform next to a film.
- 2. If needed, apply drops of cyanoacrylate onto the tray.
  - a. NOTE: A couple drops of cyanoacrylate on the tray should be enough for 2-3 arrays, depending on how successful the transfer is.
- 3. Using a toothpick, collect cyanoacrylate from the tray.
- 4. Apply the cyanoacrylate on the toothpick to the PEEK section, near the edge of the platform, and spread it out until there is no more cyanoacrylate coming from the toothpick.
- 5. Repeat Steps 3-4 until there is a collection of cyanoacrylate at least as wide as the film's connector section and thick enough that the cyanoacrylate is fully covering the PEEK below.
- 6. Using tweezers, grab the film (de-insulated metal side up) from about 0.5" back from the connector section.
- 7. With the Kimwipe in your other hand, carefully place the film onto the spread-out drop of cyanoacrylate on the PEEK section apply pressure with the Kimwipe immediately.
  - a. NOTE: The tweezers can be released once the Kimwipe has pressure on the film.
- 8. Press down on the film with the Kimwipe to soak up excess cyanoacrylate.
- 9. Blot the corners and edges of the film with fingers from your other hand. Then lift the Kimwipe off the film and blot spots remaining on the film.
- 10. Remove the Kimwipe from the film and ensure the PEEK is hanging over the edge of the platform to prevent cyanoacrylate from drying under the PEEK. Leave the cyanoacrylate to dry.

# **Step 3 – Drying**

- Allow enough time after the PEEK is applied to the thin film in order for the glue to dry.
  a. Minimum: Approx. 1 hour
- 2. If there was excess glue under the PEEK during application, make sure the PEEK is not stuck to the platform by nudging the edges every so often before proceeding to the following step.
  - a. Suggested: Approx. every 20 minutes

# **Step 4 – Trimming**

a.

After the cyanoacrylate has dried completely, it is time to cut the excess PEEK off the films. This process will require the microscope, fine scissors, and tweezers for proper handling.

1. Turn on the light for the microscope and place the scissors near the working area of the microscope.



- 2. Grad the film you wish to cut out with tweezers on the PEEK.
  - a. NOTE: You may need to hold the PEEK with gloved hands for a stronger grip.
- 3. Place the scissors onto the side of the connector and begin cutting the excess PEEK as closely to the line of the film as possible.
  - a. <u>NOTE: Orient the scissors such that the scissor arm above the film is on the outside</u>



- 4. Cut the excess PEEK from the side of the connector, following the line of the film as closely as possible, until just past the edge of the film.
  - a. <u>NOTE: Orient the scissors such that the scissor arm above the film is on the outside</u>



5. Rotate the film 90 degrees and cut out the excess PEEK outside of the film.

b.



- 6. Cut the excess PEEK from the edge of the film, following the line of the film as closely as possible.
  - a. NOTE: Orient the scissors such that the scissor arm above the film is on the outside

a.





a.

8. Rotate the film 90 degrees and place the scissors onto the side of the connector and begin cutting the excess PEEK as closely to the line of the film as possible.



a. <u>NOTE: Orient the scissors such that the scissor arm above the film is on the</u> <u>outside</u>

- 9. Cut the excess PEEK from the side of the connector, following the line of the film as closely as possible, until just past the edge of the film.
  - a. <u>NOTE: Orient the scissors such that the scissor arm above the film is on the</u> <u>outside</u>



10. The film now has a backing of PEEK successfully adhered to it. This should be a secure adhesion, but if the backing falls off after repeated use, repeat these steps to replace the PEEK backing.

# EIS Results for ASU M<sup>3</sup> Electrode Arrays

A look at the electrochemical performance of electrode arrays with the  $M^3$  electrode combination fabricated at Arizona State University

**Evaluated by Jon Garich** Testing occurred between 1/28/2019 and 2/11/2019 and model fitting occurred on 2/18/2019.

# Contents

Array Design 3	,
Fabrication Details	,
Wafer Shipment Details 3	,
Testing Selections	•
EIS Recording Setup	,
EIS Results – Individual Arrays	į
128 - #4	i
Magnitude	į
Phase7	•
128 - #7	•
Magnitude	•
Phase 8	,
128 - #9	,
Magnitude	ł
Phase	ł
128 - #15	ł
Magnitude 10	,
Phase 10	,
128 - #17	•
Magnitude 11	•
Phase	
EIS Results – Averages 12	
Averages – Each Array13	,
Magnitude 13	,
Phase	,
Averages – All Arrays14	•
Magnitude	Ļ

Phase	
Model Fitting Results	
Comparison Table	
Discussion	
Conclusions	

## **Array Design**

Arrays were designed with the  $M^3$  concept, featuring a clinical macroelectrode (4 mm diameter) encompassing four intermediate mesoelectrodes (1 mm diameter), which encompassed seven spatially distinct microelectrodes (40  $\mu$ m diameter). Traces internal to the macroelectrodes had a width of 10  $\mu$ m and opened up to widths of 50  $\mu$ m in the tail. Each wafer was designed with a single 128-channel array and six 32-channel arrays with additional features for precise alignment (with scales of 10, 5, 3, and 1  $\mu$ m) and test pads for resistivity measurement.

## **Fabrication Details**

Fabrication occurred at Arizona State University (ASU) using the ASU NanoFab (Class 100 cleanroom) for standard photolithography, metal deposition, plasma etching, and characterization as well as the Soft Lithography Lab (Class 1,000 cleanroom) for polyimide deposition and lift-off processing. Metal deposition occurs in batches of four wafers per run, so the labeling is by batch number and overall wafer number, e.g., B2-007 would be the third wafer in the second metal batch.

## Wafer Shipment Details

A total of 20 wafers were fabricated and shipped to Mayo Clinic's Rochester campus, totaling 20 of the 128-channel arrays and 120 of the 32-channel arrays. Previous documentation reviewed the yield of this shipment, based purely on a visual microscopic inspection, resulting in 13 acceptable 128-channel arrays and 94 acceptable 32-channel arrays, with a handful of arrays capable of being reworked for inclusion. [Link to document on Google Drive] [Link to document on DoE's L:\drive]

## **Testing Selections**

Out of the 13 acceptable 128-channel arrays, one array from each metal batch was selected for electrochemical impedance spectroscopy (EIS), totaling five arrays. A single macroelectrode, two mesoelectrodes, and two microelectrodes per connector of each array tested were chosen for EIS analysis, equating to four macroelectrodes, eight mesoelectrodes, and eight microelectrodes per array; in other words, 20 electrodes were tested in total per array. A section of polyether ether ketone (PEEK) at a thickness of 127  $\mu$ m was added to the connector sections of each array tested to ensure reliable contact between the metal contacts on the thin-film arrays (20  $\mu$ m thickness) with the pins on the ZIF connectors attached to the electrode interface board (EIB), which has a ~150  $\mu$ m insertion slot tolerance.

# **EIS Recording Setup**

- Recording Device:
  - Gamry Instruments Reference 600+ Potentiostat (REF600P-31099)
- Electrochemical Setup:
  - Three-Cell Model:

- Floating Ground (GND)
  - GND line of Faraday cage
  - Reference Electrode (RE)
    - Ag/AgCl rod suspended in a 3 M solution of NaCl
- Counter Electrode (CE)
  - Pt wire (0.4 mm diameter wire) coil with a geometric surface area (GSA) of 1.25 mm<sup>2</sup> and a coiled length of 10 cm
- Working Electrode/Working Sense (WE/WS)
  - Electrode undergoing testing from the respective array
    - Microelectrode GSA: 3,769.911  $\mu$ m<sup>2</sup> = 0.004 mm<sup>2</sup>
    - $\circ$  Mesoelectrode GSA: 1,118,299.471  $\mu$ m<sup>2</sup> = 1.118 mm<sup>2</sup>
    - Macroelectrode GSA: 11,805,656.713 μm<sup>2</sup> = 11.806 mm<sup>2</sup>

- Solution:
  - 1xPBS at room temperature (RT)
- Connection to Test Electrodes:
  - 39-pin ZIF connector to attach the thin-film arrays to the 32-channel electrode interface board (EIB)
  - Custom 32-channel EIB with 32 AWG wires soldered to each pin that corresponds to a recording channel
- Recording Setup:
  - Software: Gamry Instruments Framework
  - Frequency Sweep: 0.1 Hz 1 MHz
  - o Point Per Decade: 5
  - AC Voltage: 10 mV (rms)
  - DC Voltage: 0 V vs. Eoc
  - Area: 1 cm<sup>2</sup> (ignored in impedance calculation)
  - Initial Delay: 100 s for stable (0 mV/s) signal
  - $\circ \quad \text{Estimated Impedance: 1 } M\Omega$

## **EIS Results - Individual Arrays**

Below are the EIS results of each tested array with all electrodes from the testing.

## **128 - #4**

Below are the results from the 128-#4 array, including the following electrodes:

M1, M49, M65, M113, m16, m17, m33, m64, m80, m81, m97, m128, u7, u25, u40, u58, u71, u89, u104, and u122

#### Magnitude







Below are the results from the 128-#7 array, including the following electrodes:

M1, M49, M65, M113, m16, m32, m33, m64, m80, m81, m112, m128, u7, u26, u40, u58, u71, u89, u103, and u122



128 - #7 [Magnitude]





Below are the results from the 128-#9 array, including the following electrodes:

M1, M49, M65\*, M113, m16, m17, m33, m64, m80, m96, m97, m128, u7, u25, u40, u58, u71, u89, u104, and u122

\* Electrode removed from calculations





-80 - 10<sup>0</sup> 10<sup>2</sup> 10<sup>4</sup> 10<sup>6</sup> Frequency (Hz)

Note: M65 has been removed from average calculations due to the lack of a stable opencircuit potential and subsequent lack of a contact dot from the ZIF connector.

Below are the results from the 128-#15 array, including the following electrodes:

M1, M49, M65, m16, m17, m33, m64, m80, m81, m97, m128, u7, u25, u40, u58, u71, u89, u104, and u122



Magnitude





Note: M113 has been removed from average calculations due to the lack of a stable opencircuit potential and subsequent lack of a contact dot from the ZIF connector.

Below are the results from the 128-#17 array, including the following electrodes:

M1, M49\*, M65, M113, m16, m32, m33, m64, m80, m96, m97, m128, u7, u25, u40, u58, u71, u89, u104, and u122

\* Electrode removed from calculations

## Magnitude





Note: M49 has been removed from average calculations due to the lack of a stable opencircuit potential and subsequent lack of a contact dot from the ZIF connector.

## **EIS Results - Averages**

Below are the EIS results of the averages or each array by electrode size as well as the EIS results of the averages across all arrays by electrode size.

## **Averages – Each Array**

## Magnitude



Phase



## **Averages – All Arrays**

## Magnitude



Phase



## **Model Fitting Results**

Electrodes are not a purely resistive or purely capacitive entity, so analyzing the complex impedance data does not provide the complete performance picture. Additional information about the electrodes can be gathered by fitting the complex impedance data to an equivalent electrical circuit model, such as the Simplified Randle's cell shown below, to estimate theoretical values of individual electrochemical components the electrode represents. These components represent the solution resistance, R<sub>s</sub>, the double layer capacitance, C<sub>DL</sub>, and the polarization resistance, R<sub>P</sub>. Each component from the model corresponds to part of the electrode and recording setup that can complete our analysis. Additional background into the definition of these equivalent circuit variables can be found in the <u>Basics of Electrochemical Impedance</u> <u>Spectroscopy</u> by Gamry Instruments.



Simplified Randle's cell (source: Gamry Instruments)

For our testing, a constant-phase element (CPE) model was used to account for realworld variability in the capacitance variable of the equivalent circuit model shown above. After recording the complex impedance across the desired frequency range using this CPE model, a conversion is needed to convert the constant-phase element model into the capacitive element desired. This conversion takes the constant-phase element value ( $Y_0$ , given in  $S^*s^{\alpha}$ ) along with the exponent value ( $\alpha$ ) and parallel resistance value ( $R_p$  in this case) to provide a capacitance in Farads. The Gamry Instruments Echem Analyst software package also includes a "Goodness of Fit" variable to help in determining how well the model used fits to the data acquired. Unlike an  $R^2$  value from typical statistics, this measured value depicts the distance of the residuals from the acquired data values. In other words, a lower "Goodness of Fit" number means the model was able to match closely to the data.

Prior to completing the electrochemical impedance spectroscopy and before fitting the resulting data to an equivalent circuit model, we can make assumptions about our electrodes solely from the design. Based on the geometric surface area (GSA) component driving the electrochemical performance of an electrode, we would assume the electrodes with a larger GSA would result in a higher double-layer capacitance and a lower resistance while electrodes with a smaller GSA would have a lower double-layer capacitance and a higher resistance. Three electrode sizes would mean we should have three different levels of capacitances and resistances. The table below shows the comparison between the average of each of the electrode sizes from each array tested as well as a combined average of each electrode size across all arrays tested.
Array/Set	Electrodes	<b>R</b> <sub>s</sub> (kΩ)	R <sub>P</sub> (GΩ)	C <sub>DL</sub> (μF)	Fit* (1E-3)
128-#4	micro	5.30	236500	0.235	11.6
	Meso	1.98	3080	18.0	0.831
	MACRO	1.67	0.01	8.31	6.74
128-#7	micro	7.26	4.02	0.008	13.7
	Meso	1.97	0.14	0.658	0.707
	MACRO	1.50	0.02	4.93	0.455
128-#9	micro	4.58	10.16	0.008	17.6
	Meso	1.90	21340	46.7	1.16
	MACRO	1.46	2147	391	0.908
128-#15	micro	4.54	13.59	0.009	18.4
	Meso	2.00	26280	43.7	0.909
	MACRO	1.54	1059	295	2.11
128-#17	micro	5.30	12.09	0.005	11.2
	Meso	2.04	9072	23.2	0.940
	MACRO	1.63	773.7	215	0.472
ALL	micro	5.36	21.71	0.010	13.4
	Meso	1.98	11200	21.3	0.781
	MACRO	1.57	455.7	93.7	1.41
* A lower fit score equates to a better model fit (less residuals)					

## **Comparison Table**

## **Discussion**

Across all arrays including the combined average, we see higher solution resistance ( $R_s$ ) in the microelectrodes while it is lower in the macroelectrodes, in comparison to the mesoelectrodes. Double-layer capacitance ( $C_{DL}$ ) is on average higher in the macroelectrodes and lower in the microelectrodes, in comparison to the mesoelectrodes, but there is certainly variance seen between the individual arrays. Unfortunately, the values obtained for the polarization resistance ( $R_P$ ) have too much standard deviation, some on the scale of 10<sup>7</sup> G $\Omega$  (10<sup>16</sup>  $\Omega$ ) or more, which means it is difficult to say much about this variable. However, array 128-#7 is free from significant standard deviation and aligns with the results seen from the  $R_s$  variable, meaning the microelectrodes carry a higher resistance and the macroelectrodes carry a lower resistance, in comparison to the mesoelectrodes.

## **Conclusions**

Data from the complex impedance graphs show that the electrodes fit into distinct sections based on their GSA. The macroelectrodes have the lowest impedance values with an upper corner frequency in the range of 100-1,000 Hz. Mesoelectrodes have intermediate impedance values with an upper corner frequency in the range of 1,000-10,000 Hz. Higher impedance values are found in microelectrodes with an upper corner frequency in the range of 100,000-10,000 Hz. These results follow the expected results predicted based on the GSA of the respective electrodes. Unfortunately, the lower corner frequencies were not detected from the frequency range selected and it is possible the upper corner frequency was not included in the range for the microelectrodes.

Fitting the complex impedance results to a CPE model shows that we also have alignment with expectations in the majority of variables. Due to the large deviation seen in the polarization resistance variable across the board, no conclusive information can be determined about the exact equivalent circuit variables from this model alone. However, the double-layer capacitance and solution resistance variables provide enough stability to estimate the electrochemical performance of the electrodes to a limited degree of certainty.

Since the frequency range selected did not include the lower corner frequency for any of the electrode sizes tested, it is possible that this accounted for the large deviation seen in the polarization resistance variables. Unfortunately, testing this theory is not practical due to the time required for each cycle of the lower frequencies without a more complex testing system. Previous tests down to 100  $\mu$ Hz did not reveal a lower corner frequency for microelectrodes of the same size, so it is likely that 10  $\mu$ Hz or even 1  $\mu$ Hz would be required. Just to clarify the time restriction, a single cycle at 10  $\mu$ Hz takes 100,000 seconds = 1 day, 3 hours, 46 minutes, and 40 seconds while a single cycle at 1  $\mu$ Hz takes 1,000,000 seconds = 11 days, 13 hours, 46 minutes, and 40 seconds. Maintaining a consistent and stable electrochemical environment for that long in a benchtop test would require a system that periodically/constantly exchanges the solution (PBS in our case) to prevent the salt concentration in the environment from changing over time as the electrodes are being tested.

# Phantom Electrophysiology Results

A look at the benchtop performance of simulated physiological performance of electrode arrays with the M<sup>3</sup> electrode combination fabricated at Arizona State University.

### Evaluated by Jon Garich

Testing occurred on 2/15/2019 and data analysis occurred between 3/11/2019 and 3/26/2019.

## **Contents**

Array Design 2
Fabrication Details
Wafer Shipment Details 2
Prior Characterization 2
Testing Selections
Phantom Electrophysiology Setup 4
System Test Setup 5
Notch Filter Design
Signal-to-Noise Ratio Calculations
Results – Averages
Array 128-#3
Macros
Mesos 12
Micros 15
Array 128-#4 19
Macros19
Mesos 22
Micros
Results – Electrode Comparison 28
Array 128-#3 28
Array 128-#4 29
Conclusions

## **Array Design**

Arrays were designed with the  $M^3$  concept, featuring a clinical macroelectrode (4 mm diameter) encompassing four intermediate mesoelectrodes (1 mm diameter), which encompassed seven spatially distinct microelectrodes (40  $\mu$ m diameter). Traces internal to the macroelectrodes had a width of 10  $\mu$ m and opened up to widths of 50  $\mu$ m in the tail. Each wafer was designed with a single 128-channel array and six 32-channel arrays with additional features for precise alignment (with scales of 10, 5, 3, and 1  $\mu$ m) and test pads for resistivity measurement.

## **Fabrication Details**

Fabrication occurred at Arizona State University (ASU) using the ASU NanoFab (Class 100 cleanroom) for standard photolithography, metal deposition, plasma etching, and characterization as well as the Soft Lithography Lab (Class 1,000 cleanroom) for polyimide deposition and lift-off processing. Metal deposition occurs in batches of four wafers per run, so the labeling is by batch number and overall wafer number, e.g., B2-007 would be the third wafer in the second metal batch.

# **Wafer Shipment Details**

A total of 20 wafers were fabricated and shipped to Mayo Clinic's Rochester campus, totaling 20 of the 128-channel arrays and 120 of the 32-channel arrays. Previous documentation reviewed the yield of this shipment, based purely on a visual microscopic inspection, resulting in 13 acceptable 128-channel arrays and 94 acceptable 32-channel arrays, with a handful of arrays capable of being reworked for inclusion. [Link to Visual Inspection results document on Google Drive] [Link to Visual Inspection results document on DoE's L:\ drive]

# **Prior Characterization**

Out of the 13 acceptable 128-channel arrays, one array from each metal batch was selected for electrochemical impedance spectroscopy (EIS), totaling five arrays. A single macroelectrode, two mesoelectrodes, and two microelectrodes per connector of each array tested were chosen for EIS analysis, equating to four macroelectrodes, eight mesoelectrodes, and eight microelectrodes per array; in other words, 20 electrodes were tested in total per array. A section of polyether ether ketone (PEEK) at a thickness of 127  $\mu$ m was added to the connector sections of each array tested to ensure reliable contact between the metal contacts on the thin-film arrays (20  $\mu$ m thickness) with the pins on the ZIF connectors attached to the electrode interface board (EIB), which has a ~150  $\mu$ m insertion slot tolerance. [Link to EIS results document on Google Drive] [Link to EIS results document on DoE's L:\ drive]

# **Testing Selections**

Two arrays were selected for phantom electrophysiology (ephys) testing. One array, 128-#3 (from B1-003), was not included in the prior EIS testing and should be free from possible experimental bias. The other array, 128-#4 (from B1-004), was part of the prior EIS testing and should be confirmed as functional. Both arrays tested were connected to a 64-channel EIB (made by Neuralynx) and half of each array was tested at a time, but the data for both was stored in the same continuously sampled channel (CSC) file. Array 128-#3 was recorded first, beginning with electrodes M65-m128 and ending with electrodes M1-m64. Array 128-#4 was recorded second, beginning with electrodes M1-m64 and ending with electrodes M65-m128. Approximately 10 seconds of data (~600 timestamp segments) were selected for each of the data segments for consistent calculations and any missing samples or dropped packets were excluded from the windows of data selected. Data was recorded in proprietary Neuralynx measurements and required conversion to a common and translatable format, so it was multiplied by the conversion factor (found in Neuralynx documentation) to make it a measurement in volts and then converted to millivolts since the recorded signal should be in the same range.

# Phantom Electrophysiology Setup

- Recording Device:
  - Neuralynx Wireless Cube (DC battery operated)
- Electrochemical Setup:
  - o 2-Cell Model:
    - Stimulation Source:
      - AD-Tech 1x8 strip of platinum/iridium electrodes on silicone
        - Contact #1 was used as the positive source terminal
        - Contact #8 was used as the negative source terminal
    - Reference Electrode:
      - AD-Tech 1x8 strip of platinum iridium electrodes on silicone
        - Contact #1 was used as the positive reference terminal
        - Contact #8 was used as the negative reference terminal
    - Working Electrode:
      - Electrode undergoing testing from the respective array (GSA = geometric surface area)
        - Microelectrode GSA: 3,769.911  $\mu$ m<sup>2</sup> = 0.004 mm<sup>2</sup>
        - Mesoelectrode GSA: 1,118,299.471 μm<sup>2</sup> = 1.118 mm<sup>2</sup>
        - Macroelectrode GSA: 11,805,656.713  $\mu$ m<sup>2</sup> = 11.806 mm<sup>2</sup>

- Solution:
  - 1x sterile 0.9% saline at room temperature (RT)
- Connection to Test Electrodes:
  - Two 39-pin ZIF connectors to attach the thin-film arrays to the 64-channel electrode interface board (EIB)
  - o 64-channel EIB was custom designed at Neuralynx for our thin-film application
- Recording Setup:
  - o Software: Neuralynx Cheetah (Electrophysiology Recording Software)
  - Signal Viewer: Hantek DSO4202C Digital Oscilloscope
  - Signal Generator: NTI Audio Minirator MR2
    - Amplitude: 700 mV peak-to-peak
      - Frequency: 10 Hz

## **System Test Setup**

Using a one-by-eight strip of platinum/iridium electrodes encased in silicone (AD-Tech), a signal was induced across two of the electrodes to create the simulated, or "phantom", electrophysiological signal as part of the bench top analysis performed on the thin-film electrode arrays fabricated at Arizona State University (ASU). The signal induced was a 10 Hz AC waveform with 700 mV peak-to-peak amplitude across a 7 cm electrode-to-electrode distance (see Figure 1B below), approximately 3-4 cm<sup>3</sup> away from the ASU electrode array in sterile saline solution. Generation of the induced signal occurred through an NTI Audio Minirator MR2 and the input and output signals were viewed on a Hantek DSO4202C Digital Oscilloscope. Another one-by-eight strip of AD-Tech electrodes were used as the reference electrodes in the electrophysiological signal with a 3 cm electrode-to-electrode separation distance (see Figure 1C below). Both the recording array and reference strip were connected to the electrode interface board (EIB), which is then connected to the Neuralynx Wireless Cube (see Figure 1A below). A detectable waveform from the recording array was seen on the Cheetah recording software, which had a 1 mV peak-to-peak viewing window for all 64 channels of recording.



Fig. 1 – (A) System setup with highlighted components including the stimulation strip [red], reference strip [blue], recording array [orange], waveform generator [purple], Neuralynx Cube [green], and Cheetah recording software [black]; (B) AD-Tech 1x8 strip inducing the simulated signal using a positive terminal at electrode 1 [red] and a ground terminal at electrode 8 [white] with the recording array [orange] and reference strip [blue] highlighted; (C) AD-Tech 1x8 strip referencing the background signal in solution across a positive terminal at electrode 4 [blue] and a ground terminal at electrode 7 [white] with the recording array [orange] and stimulation strip [red] highlighted.

## **Notch Filter Design**

Unfortunately, no data was recorded without any stimulation present, so there is no background signal we can use to calculate the noise value for signal-to-noise (SNR) evaluation. However, since we know the frequency of the induced signal, we can closely approximate the noise signal by filtering out the induced signal and leaving the remaining data as noise. Since this is a low-frequency signal at 10 Hz, a 10<sup>th</sup> order Chebyshev II Bandstop (notch) IIR filter from 9-11 Hz should be sufficient to isolate the noise from the induced signal. This filter was applied to the recorded data such that both the recorded signal and the filtered signal could be analyzed. Signal averages were taken both before and after filtering to ensure consistent data averaging. Both the original recorded data and the filtered noise data were converted to the frequency domain and the power spectrum density was analyzed to detect the induced signal in the original recorded data and confirm the successful filtering in the noise data.

## Signal-to-Noise Ratio Calculations

With both the original recorded data and the filtered noise data, the signal-to-noise ratio (SNR) can now be calculated for each electrode and across averaged data sets. Typically, this is done with unfiltered noise data, but this will be completed in a future comparison between the AD-Tech strips and the thin-film electrode arrays fabricated at ASU. Instead, the noise can be closely approximated from the filtered data isolating the noise from the recorded data. The peak-to-peak amplitude of the recorded signal and the root mean square (RMS) value of the filtered noise data are necessary variables to compute the SNR, as shown in the equation below.

$$SNR = \frac{V_{pp}}{2 * V_{rms}}$$

 $V_{pp}$  is the peak-to-peak amplitude of the recorded signal (including inherent noise) and  $V_{rms}$  is the RMS value of the filtered noise signal. This calculation should yield a ratio value and the greater this value, the greater the signal amplitude is compared to the amplitude of the noise.

## **Results – Averages**

Data gathered was averaged at separate points of the analysis: prior to processing, after filtering, and after signal-to-noise calculation. Presented here are only the averaged values for each electrode size of each array, not the complete index of results from individual electrodes. Signal-to-noise ratio (SNR) measurements were recorded in ratio form and presented as bar graphs with numeric labels on averaged results.

## Array 128-#3





*Fig. 2* – Average of recorded data from all macroelectrodes prior to any further analysis, approximately 1 second of data is shown to show detail on the 10 Hz signal. Time is shown in microseconds.



Fig. 3 – Power spectrum density (PSD) of average recorded data from all macroelectrodes prior to any filtering or calculations, shown from 0-1 kHz. A Blackman window was used to control frequency spread.



*Fig.* **4** – Filtered data isolating the recorded noise with data from all macroelectrodes averaged after filtering. Approximately 1 second of data is shown to highlight possible remnants of the 10 Hz signal. Time is shown in microseconds.



Fig. 5 – Power spectrum density (PSD) of the filtered data isolating the recorded noise with data from all macroelectrodes averaged after filtering. Frequencies are shown from 0-1 kHz. A Blackman window was used for both plots to control frequency spread.



Fig. 6 – Signal-to-noise ratio (SNR) of each of the macroelectrodes from the array (128-#3) presented in numerical order.



Fig. 7 – SNR averages from array 128-#3 with data from all macroelectrodes averaged either after filtering (Post-Filter) or after calculating the SNR for each (Combined Average).

The lower-amplitude signal on M49 and M65 significantly affected the averaging for this array, which can be seen by the noisy signal in Figure 2, likely exemplified by the 30 Hz spike seen in Figures 3 & 5. This array did have a poor trim of the PEEK backing, so it is possible these results were caused by poor connection to the recording system.



Fig. 8 – Average of recorded data from all mesoelectrodes prior to any further analysis, approximately 1 second of data is shown to show detail on the 10 Hz signal. Time is shown in microseconds.



Fig. 9 – Power spectrum density (PSD) of average recorded data from all mesoelectrodes prior to any filtering or calculations, shown from 0-1 kHz. A Blackman window was used to control frequency spread.



*Fig.* **10** – Filtered data isolating the recorded noise with data from all mesoelectrodes averaged after filtering. Approximately 1 second of data is shown to highlight possible remnants of the 10 Hz signal. Time is shown in microseconds.



Fig. 11 – Power spectrum density (PSD) of the filtered data isolating the recorded noise with data from all mesoelectrodes averaged after filtering. Frequencies are shown from 0-1 kHz. A Blackman window was used for both plots to control frequency spread.



Fig. 12 – Signal-to-noise ratio (SNR) of each of the mesoelectrodes from the array (128-#3) presented in numerical order.



Fig. 13 – SNR averages from array 128-#3 with data from all mesoelectrodes averaged either after filtering (Post-Filter) or after calculating the SNR for each (Combined Average).

The lower-amplitude signal on m64, m66, m80, m81, and m96 affected the averaging for this array, but there were more quality signals than poor signals. This array did have a poor trim of the PEEK backing, so it is possible these results were caused by poor connection to the recording system.



Fig. 14 – Average of recorded data from all microelectrodes prior to any further analysis, approximately 1 second of data is shown to show detail on the 10 Hz signal. Time is shown in microseconds.



Fig. 15 – Power spectrum density (PSD) of average recorded data from all microelectrodes prior to any filtering or calculations, shown from 0-1 kHz. A Blackman window was used to control frequency spread.



*Fig.* 16 – Filtered data isolating the recorded noise with data from all microelectrodes averaged after filtering. Approximately 1 second of data is shown to highlight possible remnants of the 10 Hz signal. Time is shown in microseconds.



Fig. 17 – Power spectrum density (PSD) of the filtered data isolating the recorded noise with data from all microelectrodes averaged after filtering. Frequencies are shown from 0-1 kHz. A Blackman window was used for both plots to control frequency spread.



Fig. 18 - Signal-to-noise ratio (SNR) of each of the microelectrodes from the array (128-#3) presented in numerical order.



Fig. 19 – SNR averages from array 128-#3 with data from all microelectrodes averaged either after filtering (Post-Filter) or after calculating the SNR for each (Combined Average).

The lower-amplitude signal on a number of microelectrodes (listed below) partially affected the averaging for this array, but there were more quality signals than poor signals. This array did have a poor trim of the PEEK backing, so it is possible these results were caused by poor connection to the recording system.

u4, u51, u53, u67, u68, u69, u70, u71, u72, u73, u74, u75, u76, u77, u78, u79, u82, u83, u84, u85, u86, u87, u88, u89, u90, u91, u92, u93, u94, u95, u115, u116, u118, and u119

Array 128-#4





Fig. 20 – Average of recorded data from all macroelectrodes prior to any further analysis, approximately 1 second of data is shown to show detail on the 10 Hz signal. Time is shown in microseconds.



Fig. 21 – Power spectrum density (PSD) of average recorded data from all macroelectrodes prior to any filtering or calculations, shown from 0-1 kHz. A Blackman window was used to control frequency spread.



Fig. 22 – Filtered data isolating the recorded noise with data from all macroelectrodes averaged after filtering. Approximately 1 second of data is shown to highlight possible remnants of the 10 Hz signal. Time is shown in microseconds.



Fig. 23 – Power spectrum density (PSD) of the filtered data isolating the recorded noise with data from all macroelectrodes averaged after filtering. Frequencies are shown from 0-1 kHz. A Blackman window was used for both plots to control frequency spread.



Fig. 24 – Signal-to-noise ratio (SNR) of each of the macroelectrodes from the array (128-#4) presented in numerical order.



Fig. 25 – SNR averages from array 128-#4 with data from all macroelectrodes averaged either after filtering (Post-Filter) or after calculating the SNR for each (Combined Average).

The macroelectrodes from array 128-#4 were very consistent. This array underwent EIS testing prior to this, so we were able to confirm proper connections throughout the majority of the array.



Fig. 26 – Average of recorded data from all mesoelectrodes prior to any further analysis, approximately 1 second of data is shown to show detail on the 10 Hz signal. Time is shown in microseconds.



Fig. 27 – Power spectrum density (PSD) of average recorded data from all mesoelectrodes prior to any filtering or calculations, shown from 0-1 kHz. A Blackman window was used to control frequency spread.



*Fig.* 28 – Filtered data isolating the recorded noise with data from all mesoelectrodes averaged after filtering. Approximately 1 second of data is shown to highlight possible remnants of the 10 Hz signal. Time is shown in microseconds.



Fig. 29 – Power spectrum density (PSD) of the filtered data isolating the recorded noise with data from all mesoelectrodes averaged after filtering. Frequencies are shown from 0-1 kHz. A Blackman window was used for both plots to control frequency spread.



Fig. 30 – Signal-to-noise ratio (SNR) of each of the mesoelectrodes from the array (128-#4) presented in numerical order.



Fig. 31 – SNR averages from array 128-#4 with data from all mesoelectrodes averaged either after filtering (Post-Filter) or after calculating the SNR for each (Combined Average).

The mesoelectrodes from array 128-#4 were very consistent. This array underwent EIS testing prior to this, so we were able to confirm proper connections throughout the majority of the array.

**Micros** 



Fig. 32 – Average of recorded data from all microelectrodes prior to any further analysis, approximately 1 second of data is shown to show detail on the 10 Hz signal. Time is shown in microseconds.



Fig. 33 – Power spectrum density (PSD) of average recorded data from all microelectrodes prior to any filtering or calculations, shown from 0-1 kHz. A Blackman window was used to control frequency spread.



*Fig.* 34 – Filtered data isolating the recorded noise with data from all microelectrodes averaged after filtering. Approximately 1 second of data is shown to highlight possible remnants of the 10 Hz signal. Time is shown in microseconds.



Fig. 35 – Power spectrum density (PSD) of the filtered data isolating the recorded noise with data from all microelectrodes averaged after filtering. Frequencies are shown from 0-1 kHz. A Blackman window was used for both plots to control frequency spread.



Fig. 36 – Signal-to-noise ratio (SNR) of each of the microelectrodes from the array (128-#4) presented in numerical order.



Fig. 37 –SNR averages from array 128-#4 with data from all microelectrodes averaged either after filtering (Post-Filter) or after calculating the SNR for each (Combined Average).

The microelectrodes from array 128-#4 were consistent with a few lower-amplitude channels (listed below. This array underwent EIS testing prior to this, so we were able to confirm proper connections throughout the majority of the array.

• u70, u98, u99, u104, u107, u108, and u110

## **Results – Electrode Comparison**

A design that incorporates multiple electrode sizes, especially in the encompassing manner accomplished in these electrode arrays, requires an individual analysis of each electrode size without combining all electrodes into a single average. This, however, allows for the unique ability to compare electrodes of different sizes on the same array, eliminating certain test conditions required of separate arrays. Below are the signal-to-noise ratio (SNR) results of the comparison between electrode sizes from the 128-channel electrode arrays fabricated at ASU.



#### Array 128-#3

Fig. 38 – Comparison between the average signal-to-noise ratios (SNR) from each of the electrode sizes from array 128-#3 with data averaged after calculating the SNR for each electrode.

As seen in the previous individual analysis, multiple electrodes from this array had low SNR values, due to lower-amplitude recorded signals. Since two out of the four macroelectrodes (50%) had a significantly lower-amplitude than the others, this average SNR value was significantly reduced compared to the consistency of the other two macroelectrodes. Five out of the sixteen mesoelectrodes (31.25%) also recorded lower-amplitude signals, but the presence of eleven mesoelectrodes with consistent amplitudes allowed the average SNR to be only slightly reduced. Similarly, 34 out of 108 microelectrodes (31.5%) recorded lower-amplitude signals, which reduced the average SNR a little more than was seen on the average SNR from the mesoelectrodes from this array.

As was noted in the individual results above, it is possible these seemingly erroneous results were caused by poor connection to the recording system due to the poor trim of the PEEK backing.



## Array 128-#4

Fig. 39 – Comparison between the average signal-to-noise ratios (SNR) from each of the electrode sizes from array 128-#4 with data averaged after calculating the SNR for each electrode.

The resulting average SNRs from this array (128-#4) were much more consistent than the previous array (128-#3) as very few channels were noted with lower-amplitude signals. The macroelectrodes maintained the highest average SNR out of the three electrode sizes while the microelectrodes had the lowest average SNR out of the three electrode sizes. It is noteworthy that this array did undergo EIS testing prior to phantom electrophysiology testing, which helped confirm correct connections on the majority of the array.

## **Conclusions**

Coming into this test, the expectation was that larger electrodes should record the highest amplitude signal due to the larger surface area, meaning more ions can collect there creating a higher potential at the surface. It could also be expected, in some respect, that the smaller electrodes might have more sensitivity due to the smaller surface area, meaning fewer ions can collect there and any change would be more significant. Unfortunately, this is not a good test for measuring the sensitivity of the electrodes, but it would show the signal collection abilities as well as the inherent noise in the recorded signal since a known signal from a waveform generator was used.

Array 128-#4 provided a clear look at consistent SNR values across all electrodes. As the expectation predicted, the macroelectrodes recorded the highest signal-to-noise ratio (SNR) out of all three electrode sizes while the microelectrodes recording the lowest SNR out of all three electrode sizes. However, the massive difference in the geometric surface area (four orders of magnitude), the microelectrodes would be expected to have a bigger difference in signal obtained than the macroelectrodes, but the difference is only about 4.1% of the average SNR from the macroelectrodes (0.82 in ratio difference). While this result did not match expectations, a higher SNR value from a smaller electrode is always beneficial.

Array 128-#3, on the other hand, did not record as many consistent SNR values from all electrodes as array 128-#4 provided. Instead, a drastic reduction in average SNR was found in the macroelectrodes, which recorded the lowest SNR out of the three electrode sizes. The mesoelectrodes actually recorded the highest average SNR value, but there were still electrodes with lower-amplitude recorded data. While the microelectrodes also had lower-amplitude recorded data on many of the channels, the average SNR was only about 4.6% lower than the average SNR from the mesoelectrodes. Typically, the value of the average SNR from the macroelectrodes would be used for comparison to the microelectrodes, but the drastic differences in SNR for two of the macroelectrodes prevent that from being a reliable source for comparison. Instead, we find the average SNR for the macroelectrodes to be about 23.6% lower than the average SNR from the mesoelectrodes. Again, it should be noted that the poor trim on the PEEK backing and therefore the possible poor connections may have been the sole driving factor in this inconsistent data. It should also be mentioned that the 30 Hz spike seen in the power spectrum density plots, which was amplified in the macroelectrodes from array 128-#3, was slightly present in all recorded electrodes without any direct correlation to AC noise (60 Hz) or rhythmic oscillation from our input signal, which would be seen at 20 Hz with higher power than it would at 30 Hz.

From these results, the expectations can be confirmed, provided that all channels have a good connection to the electrode interface board. It is recommended to perform a test to confirm connectivity prior to implanting the electrode arrays in an animal model. Whether the connectivity confirmation test is EIS or phantom electrophysiology likely depends on the time available and the detailed analysis expected as EIS testing takes approximately a day per array to record and less than a day to analyze all results while phantom electrophysiology can be recorded in a few minutes per array but analysis might take a day or more depending on how many arrays were tested and how many electrodes are being used to calculate averages.