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W. Braun, H. Möller, and Y.-H. Zhang

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Accurate growth rate determination on rotating substrates using electron diffraction dynamics

W. Braun[a)]
Center for Solid State Electronics Research and Department of Electrical Engineering, Arizona State University, Tempe, Arizona 85287-6206

H. Möller
Fraunhofer-Insitut für integrierte Schaltungen-A, D-91058 Erlangen, Germany

Y.-H. Zhang
Center for Solid State Electronics Research and Department of Electrical Engineering, Arizona State University, Tempe, Arizona 85287-6206

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Reflection high-energy electron diffraction oscillation frequencies are determined by measuring the width of the specular spot perpendicular to the surface during substrate rotation. Substrate rotation and data acquisition are phase locked to obtain exact rotation frequencies, allowing the inclusion of satellite peaks in the measurement. The method has a typical accuracy of well below 1% and provides a practical means to measure growth rates on rotating substrates. © *1999 American Institute of Physics.* [S0003-6951(99)02301-3]

Reflection high-energy electron diffraction (RHEED) intensity oscillations are routinely used for growth rate determination in molecular beam epitaxy (MBE). Usually, these oscillations are measured with the substrate held at a fixed position. The accuracy of this approach is limited by the usually strong damping of the oscillations.[1] Since much of this damping is due to flux nonuniformities along the surface area probed by the RHEED beam,[2] measurements during rotation have the potential for greater accuracy. Attempts to measure RHEED oscillations during substrate rotation,[3–5] however, have been hampered by the significant noise levels introduced by mechanical vibrations of the substrate manipulator.[3] Also, the intensity of the specularly reflected spot is a very strong function of the azimuthal angle,[6] requiring detectors with a very high dynamic range. In addition, substrate misorientation and wobble complicate the measurement, since the phase of the intensity oscillations is a strong function of both the azimuthal[7] and the polar angle[8–10] of the incident beam. Previous approaches have therefore relied on very high rotation speeds[4] or spot tracking algorithms combined with numerical filtering.[5]

In this letter, we demonstrate a method that significantly increases the accuracy of the growth rate determination by measuring the full width at half maximum (FWHM) of the specular spot perpendicular to the substrate during sample rotation. Our experiments indicate reliable results for substrate miscut below 0.5°, wobble below 2° and visibility of the specular spot for most of the rotation. The imaging sensor and the substrate rotation are phase locked to the same timebase, allowing us to exactly fix the rotation frequency in the measured data. We can therefore include the positions of satellite peaks in the frequency spectrum to improve the accuracy.

The experiments were performed using a VG V80 MBE chamber equipped with the standard RHEED gun and the standard right angle sample manipulator. The substrate rotation was driven by a specially designed phase-locked motor synchronized to the same timebase as the charge coupled device (CCD) camera that recorded the RHEED signal. The RHEED image processing system[11] was capable of measuring at a rate of 50 Hz, leading to twice the frequency resolution compared to standard CCD-based systems. GaAs growth was monitored at a sample temperature of 560 °C measured by a Pyritte pyrometer. The measurement geometry together with a plot of the raw signal are shown in Fig. 1. The RHEED intensity is measured along a line perpendicular to the substrate surface through the center of the specular spot. During rotation, the specular spot follows a very narrow elliptical trace determined by the added contributions of substrate wobble and sample miscut. Usually, the approximation of this movement by a straight line is good enough for an accurate determination of the spot dimension along the surface normal.

The line profiles plotted as a function of time are shown in the right panel of the figure. The oscillation signal is then obtained by measuring the FWHM of the peak in each trace with a subpixel-accuracy interpolation algorithm. The resulting trace of specular spot FWHM as a function of time is shown in Fig. 2. For low substrate wobble and misorienta-

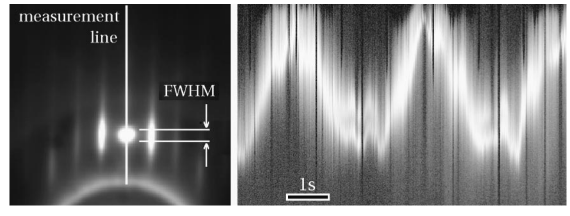

FIG. 1. Measurement geometry and raw signal measured from a rotating substrate. Growth oscillations are measured by determining the FWHM of the specular spot perpendicular to the substrate surface.

a)Present address: Paul-Drude-Institut für Festkörperelektronik, Hausvogteiplatz 5-7, D-10117 Berlin, Germany. Electronic mail: braun@pdi-berlin.de

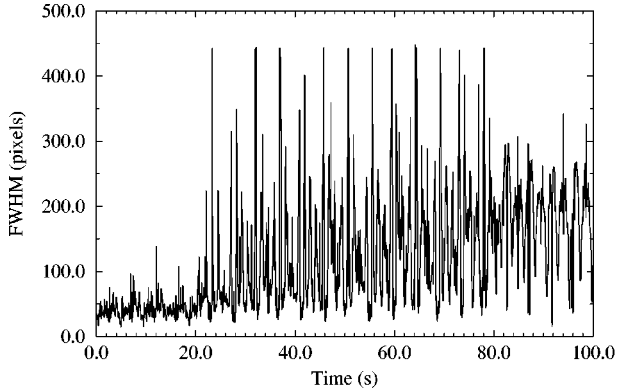

FIG. 2. FWHM of the specular spot perpendicular to the substrate as a function of time in units of CCD pixels. Growth starts at 20 s and terminates at 80 s.

tion, the signal is usually good enough to directly count the oscillations. Additional experiments (not shown) indicate that the signal-to-noise ratio of the FWHM signal perpendicular to the sample surface is much better than both the intensity signal integrated along the line or the FWHM signal parallel to the sample surface. This is mainly due to the much higher transfer width[12] of RHEED along the beam direction compared to perpendicular to the beam. It is therefore advantageous to measure only perpendicular to the surface instead of tracking the spot size in two dimensions.[5] Theoretical treatments of the (01) spot size as a function of step density[13] indicate a strong increase in the FWHM perpendicular to the substrate with increasing step density. Qualitatively similar results may be expected for the specular spot. It is therefore possible that the FWHM of the specular spot perpendicular to the substrate is related to the surface step density in a much more direct way than the intensity of the specular spot.[9]

In contrast to the intensity signal, the FWHM does not change by orders of magnitude in one revolution.[6] The signal shows very little damping. Instead of the signal level dropping at constant noise level as in standard intensity oscillation measurements, the noise level increases at constant signal level. Both properties together with the flat baseline of the signal in Fig. 2 make these measurements ideal candidates for Fourier transform. The transformed signals for three measurements are superimposed in Fig. 3. At a fixed rotation frequency, the growth rate was set at three different values in the measurements. The comparison of the power spectra clearly reveals the structure of the data in frequency space. The rotation frequency peaks at $0.1\overline{6}$ Hz and its harmonics remain unaffected by the change in growth rate. The growth frequency exhibits a set of distinct satellites on both sides at distances of multiples of the rotation frequency. Measuring at 50 Hz sampling frequency, the FWHM of the growth frequency peak is usually very close to the limit set by the frequency–time uncertainty relationship, being approximately equal to the data point spacing in frequency space. For measurement times above 150 s, this results in a FWHM of around 1% of the absolute value for growth frequencies around 1 Hz.

Due to the phase-locked rotation, the rotation frequency values in the power spectrum are *exact*. One can therefore measure the positions of the satellites as well, reducing the

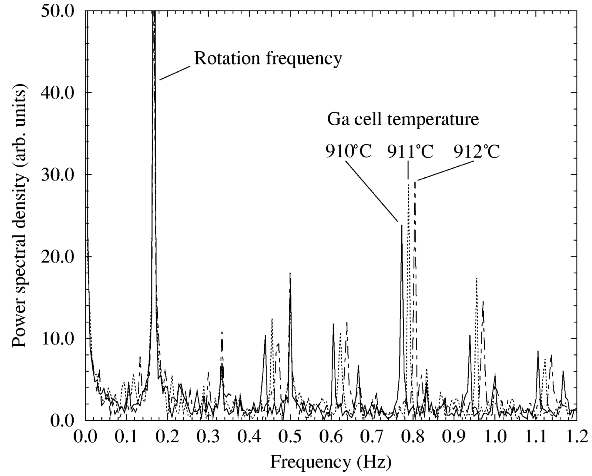

FIG. 3. Frequency spectra of FWHM measurements at three different growth rates, but identical rotation frequency. The measurement time was 180 s.

error as $\sqrt{n}$ with the number n of satellite positions measured. At least four satellites are usually discernible, resulting in an improvement by more than a factor of 2 and a final accuracy of below 0.5%. For the data in Fig. 3, this resolution corresponds to about 0.2° in the Ga cell temperature, which is getting close to the absolute temperature resolution of MBE systems and which is certainly good enough to calibrate even sensitive structures such as vertical cavity surface emitting lasers.

Usually, the rotation frequency is an adjustable parameter in MBE, the case of the growth frequency being an exact multiple of the rotation frequency can therefore be avoided. If the frequency spectrum is ambiguous, a change in the rotation frequency by $\frac{2}{3}$ clearly separates the peaks. The method works best with the rotation frequency and the growth frequency being within the same order of magnitude. If the rotation frequency is too low, the effects of growth rate nonuniformity across the wafer broaden the peak. The spectra of three different measurements are shown in Fig. 4. The solid line represents the Fourier spectrum of the specular spot intensity signal without rotation, the dashed line is the result from the integrated intensity signal along the measure-

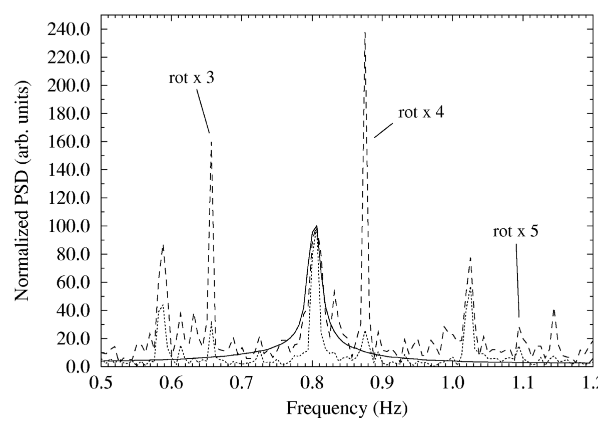

FIG. 4. Comparison of the growth frequency peaks (normalized) for three different measurement methods: (solid line) specular spot intensity, no rotation, (dashed line) integrated intensity along the line perpendicular to the substrate through the specular spot during rotation, (dotted line) FWHM along the line perpendicular to the substrate through the specular spot during rotation.

ment line perpendicular to the substrate and the dotted line is the FWHM signal. The latter two were obtained from the same measurement using 0.25 Hz rotation. In all three cases, the growth interval was 160 s long.

The FWHM of the frequency peak for the static intensity measurement is 0.027 Hz. With rotation, the width decreases to 0.021 Hz for the intensity and 0.013 Hz for the FWHM signal. The measurement based on the FWHM perpendicular to the sample surface is clearly superior, both in accuracy and also in the ratio of the growth rate peak intensity to the rotation peak intensity. The larger width for the nonrotating measurement can be explained by the strong damping of intensity oscillations.[1] Much of this damping is due to growth rate nonuniformities that are strongly reduced with rotation. While it is well known that the phase of the intensity oscillations of the specular spot strongly varies with azimuthal[7] as well as polar angle,[8–10] which would lead to a broadening of the peak, the behavior of the specular spot FWHM oscillation phase as a function of diffraction conditions is not well known. The strong difference of the peak widths in Fig. 4 seems to imply that the FWHM of the specular spot perpendicular to the substrate is much less sensitive to these variations. Further studies are needed to clarify this point.

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