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Real time *in situ* composition control of InGaAs lattice matched to InP by an 88-wavelength ellipsometer

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We have compiled the optical constants database for $In_xGa_{1-x}As$ which covers the composition range from 0.51 to 0.55 and the temperature range from 400 to 525 °C. The InP substrate temperature was monitored by diffusive reflectance spectroscopy during the growth of the epitaxial layer. Ellipsometry was used to monitor the $In_xGa_{1-x}As$ composition over the entire temperature and composition range of the database. The composition monitored by ellipsometry is within 0.002 from the high resolution x-ray data with the exception of growth temperature at 400 °C which is 0.005. We have also demonstrated the real time *in situ* feedback control of the $In_xGa_{1-x}As$ composition during epitaxial growth by using ellipsometry. The absolute accuracy of the $In_xGa_{1-x}As$ composition from the controlled experiment is 0.002. We can use this database to grow thick $In_xGa_{1-x}As$ layers grown on the InP substrates and can also use this as an *in situ* tool to fine tune the $In_xGa_{1-x}As$ composition before the growth of the complicated structure. © *1998 American Vacuum Society.* [S0734-211X(98)15503-8]

I. INTRODUCTION

The ideal tool to be used in the molecular beam epitaxial (MBE) growth experiment should be nondestructive, *in situ*, and should be able to monitor the growth of the epitaxial layer with the substrate rotation on. The thickness and composition information should be obtained during the growth to be used in the real time feedback control of either composition or thickness. Various optical techniques like reflectance difference spectroscopy,[1] normal incidence reflectance spectroscopy,[2] and ellipsometry[3,4] were used as *in situ* tools in both MBE and metalorganic chemical vapor deposition (MOCVD) experiments. From the recent development of the multi-wavelength (88-wavelength) ellipsometer, ellipsometry turns out to be the best *in situ* technique to obtain both thickness and composition information during MBE growth.

The ellipsometer has been used not only in the growth thickness control[3] for AlAs/GaAs distributed Bragg reflectors in the MBE chamber, it has also been used in the thickness control of the etching experiment[5–7] of SiO_2 and Si_3N_4 on the Si substrate. This will give the advantage of determining the SiO_2 or Si_3N_4 layer thickness during the etching process in real time and no time consuming sample preparation like cross-section transmission electron microscopy will be needed. We have also demonstrated the strong correlation of *ex situ* ellipsometric data with excimer laser annealed poly-Si thin film crystallinity and grain size.[8] The ellipsometer has also been used to determine the poly-Si crystallinity in real time right after the growth of *a*-Si in the plasma-enhanced chemical vapor deposition (PECVD) chamber.[9] It is important to be able to monitor the quality of the poly-Si thin film which is used in thin film transistors (TFTs). In the mass production environment, it is possible to monitor the poly-Si property right after the excimer laser annealing process, which in turn will effect the device performance of the TFT.

However, the most challenging work in the real time *in situ* control experiment is to control the composition of the ternary layer during the epitaxial growth in the MBE chamber. This has been demonstrated by Aspnes *et al.*[10] in their real time *in situ* composition control of the $In_xGa_{1-x}As$ parabolic quantum well experiment in MOCVD. We are going to demonstrate in this article the real time *in situ* composition control of the $In_xGa_{1-x}As$ layer lattice matched to InP in MBE.

II. EXPERIMENT AND RESULT

The schematic of the DCA 450 MBE chamber used in this experiment is shown in Fig. 1. There are three distinct features in this MBE chamber compared to the traditional MBE system. Substrate temperature was measured by direct recoil spectroscopy (DRS), growth rate and composition were measured by 88-wavelength ellipsometer, and substrate wobbling during rotation was minimized by applying a negative high voltage on the piezo crystal to adjust the length of the three supporting the manipulator. Detailed theory and operation of DRS is not a subject in this article and can be found elsewhere.[11]

Since there is no optical constant database available for $In_xGa_{1-x}As$ at MBE growth temperature, we have to compile our own database. Five different samples with composition $x = 0.518$, 0.525, 0.532, 0.544, and 0.557 were grown in the MBE chamber with the V/III ratio about 10 from the retractable flux monitoring ion gauge. Each sample was grown at 450 °C with a layer thickness of about 4000 Å. During the growth of each $In_xGa_{1-x}As$ layer, the substrate

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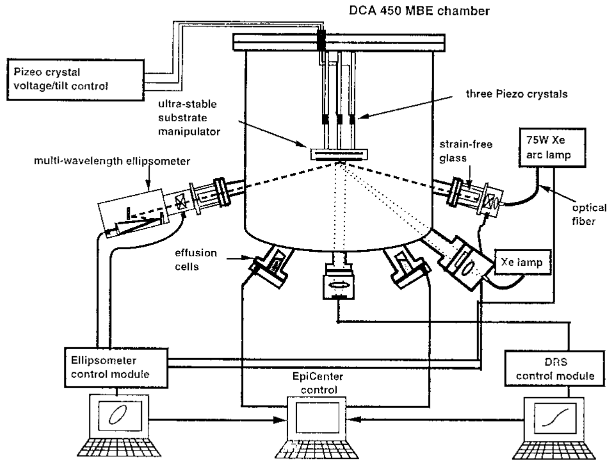

FIG. 1. Implementation of multi-wavelength ellipsometer and diffusive reflectance spectroscopy on a DCA 450 MBE chamber.

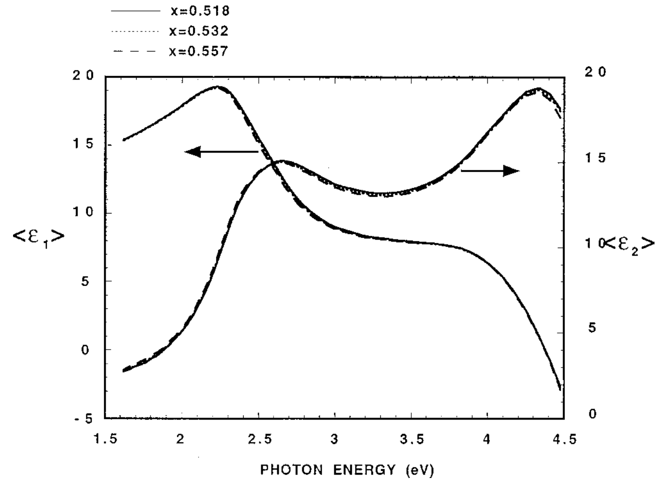

FIG. 2. Pseudodielectric of $In_xGa_{1-x}As$ at 450 °C and composition $x=0.518$, 0.532, and 0.557.

temperature was kept at constant 450 °C DRS temperature by adjusting the substrate eurotherm temperature setpoint. In order to keep a constant substrate temperature during the $In_xGa_{1-x}As$ layer grown on the InP substrate, the substrate thermocouple temperature has to drop about 45 °C for the growth of 4000 Å $In_xGa_{1-x}As$ layer on the InP substrate. This is due to the radiation heating used to heat up the substrate. The $In_xGa_{1-x}As$ layer lattice matched to InP has a smaller band gap than InP. The infrared radiation from the heater plate passing through the InP substrate was absorbed by the grown $In_xGa_{1-x}As$ layer. The absorption of the infrared radiation by the growing $In_xGa_{1-x}As$ causes the substrate temperature to increase during growth of the $In_xGa_{1-x}As$ layer on the InP substrate. The degree of the extra infrared absorption is also dependent on the $In_xGa_{1-x}As$ layer thickness. In order to achieve precise control of the $In_xGa_{1-x}As$ composition, it is important to keep the substrate temperature constant by monitoring substrate temperature through DRS.

After the growth of each of the $In_xGa_{1-x}As$ layers, the substrate temperature was ramped to 525 °C as indicated by DRS and ellipsometric data were measured by an 88-wavelength ellipsometer. The substrate temperature was ramped down from 525 to 400 °C in 25 °C steps. Ellipsometric data were taken at each temperature step after the substrate temperature is stabilized and a total of six different temperature ellipsometric data were taken from each sample. The layer thickness used in deriving optical constants for each sample was determined by analyzing the *in situ* dynamical data during MBE growth. All the temperature and composition optical constants were passed through a series of iteration loops at J. A. Woollam's Co. to correct for the influence from the change of incident angle. The correction for the incident angle was less than 0.1°. Part of the corrected optical constants are shown in Fig. 2.

As can be seen from Fig. 2, the difference of the 450 °C optical constants at $x=0.518$ and $x=0.557$ is very small. It might not be possible to determine the $In_xGa_{1-x}As$ composition in real time with the absolute precision of 0.005 from ellipsometry. A series of samples were grown to verify the validity of the $In_xGa_{1-x}As$ database that we compiled from static ellipsometry experiments. The first set, short term stability test, of five different $In_xGa_{1-x}As$ composition, $x=0.518$, 0.525, 0.532, 0.544, and 0.557, were grown and monitored by ellipsometry in a period of 2 weeks. Each sample was started at 500 °C growth temperature and ramped down in 50 °C steps and ended at 400 °C growth temperature. The comparison of the $In_xGa_{1-x}As$ composition derived from ellipsometric data and high resolution x-ray data is shown in Fig. 3(a).

The second set, medium term stability test, of four different $In_xGa_{1-x}As$ composition, $x=0.502$, 0.519, 0.535, and 0.546, were grown and monitored by ellipsometry 1 1/2 months after the short term stability test. The first two $In_xGa_{1-x}As$ samples, DCA130 and DCA131, were grown with the same temperature sequence as the short term stability test which started from 500 °C growth temperature and ramped down to 400 °C in 50 °C steps. The last two $In_xGa_{1-x}As$ samples, DCA132 and DCA133, were started from 400 °C growth temperature and ramped up to 500 °C in 50 °C steps. The results of the derived ellipsometric $In_xGa_{1-x}As$ composition compared to high resolution x-ray data are shown in Fig. 3(b).

The third set of the samples, the long term stability test, were grown after the vacuum break in the MBE chamber to reload As and Ga materials. After the bake out of the MBE chamber, three different samples with compositions of $x=0.52$, 0.532, and 0.545 were grown to test the static $In_xGa_{1-x}As$ database. The growth temperature was 450 °C for all three samples and the Ga cell temperature was adjusted manually to achieve the target $In_xGa_{1-x}As$ composition. The $In_xGa_{1-x}As$ compositions obtained from high resolution x-ray data were $x=0.522$, 0.532, and 0.547. The precision of the $In_xGa_{1-x}As$ composition monitored by ellipsometry after bake out of the MBE chamber is still within 0.002 of high resolution x-ray data.

We have also demonstrated the real time *in situ* closed

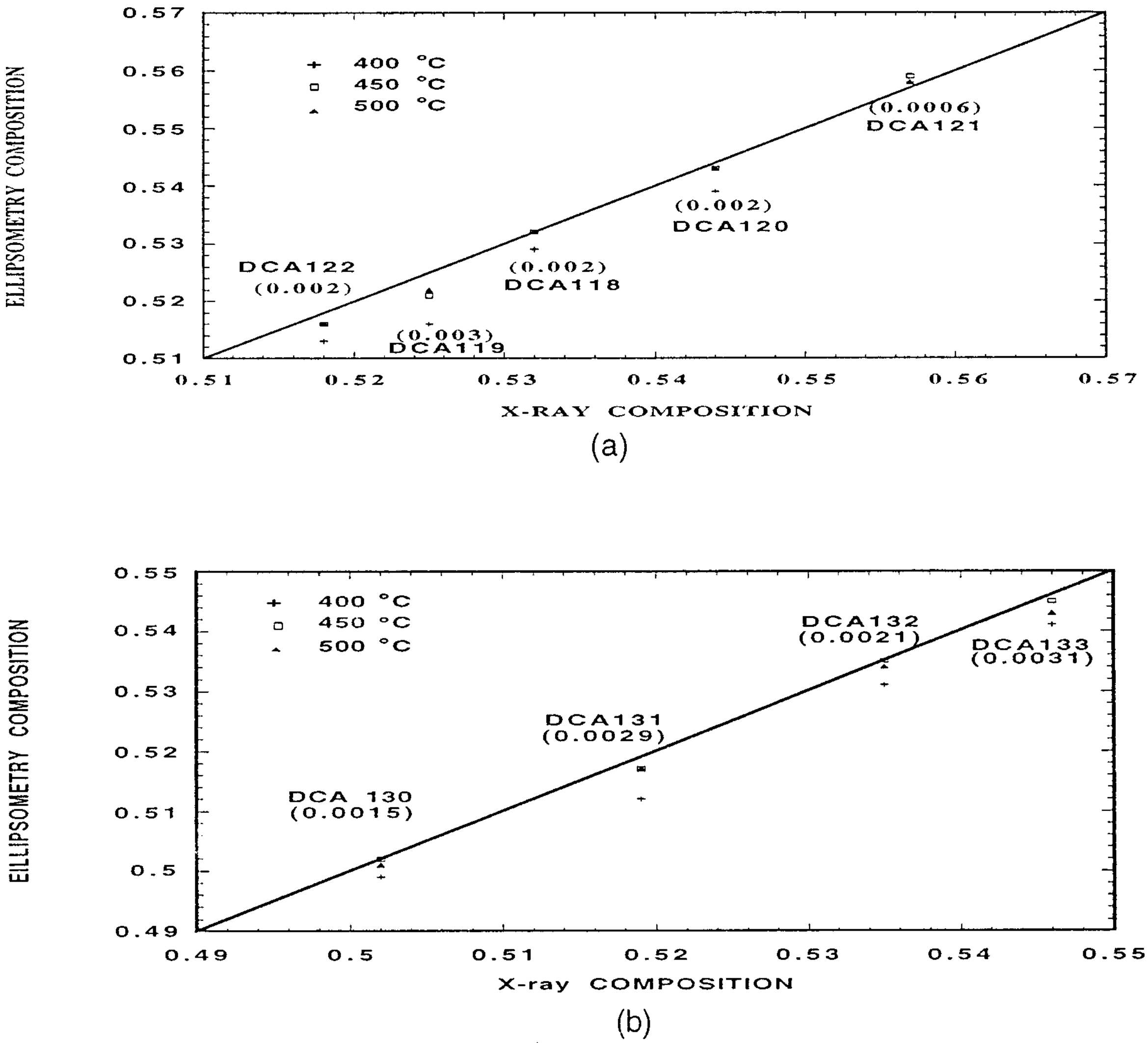

FIG. 3. Comparison of the $In_xGa_{1-x}As$ composition between high resolution x-ray data and ellipsometry data. The number in parentheses is the standard deviation of the difference of composition between ellipsometry and x-ray data for each sample at three different growth temperatures: (a) the result from the short term stability test and (b) the result from the medium term stability test.

loop feedback control of $In_xGa_{1-x}As/InP$. As shown in Fig. 1, the substrate temperature from DRS and composition data from ellipsometry were sent to the control computer with the control program called EPICENTER, which was developed at Arizona State University. EPICENTER analyzed the input data from the ellipsometer and DRS and proper action was taken to maintain a constant substrate temperature and $In_xGa_{1-x}As$ composition. A simple algorithm to control substrate and Ga cell temperature was used in this experiment to achieve both substrate temperature control and $In_xGa_{1-x}As$ composition control. Substrate temperature control was achieved by ramping the substrate temperature down 6 °C in 6 s when both the In and Ga shutter were open to grow the $In_xGa_{1-x}As$ layer. The substrate temperature was updated at EpiCenter by DRS about every 2 s. The EPICENTER checked the substrate temperature every 30 s. If the substrate is not within 0.5 °C of the desired value, the substrate eurotherm setpoint was set to the proper temperature according to the difference of the DRS temperature and the desired growth temperature. No action will be taken again at the substrate temperature control within 30 s of each substrate temperature change.

A similar algorithm was used in the Ga cell temperature control. The $In_xGa_{1-x}As$ composition was controlled by using a fixed In cell temperature and ramped the Ga cell temperature to achieve the target $In_xGa_{1-x}As$ composition. Details of the ellipsometric model used in the real time *in situ* composition control will be discussed in a later publication. The $In_xGa_{1-x}As$ composition value sent to EpiCenter was neglected for the first 8 min of growth of the $In_xGa_{1-x}As$ layer to allow a stable composition value from the ellipsometry. After 8 min, the Ga cell temperature was ramped up or down according to the difference in ellipsometric determined composition and the desired composition. For each $+0.01$ change in $In_xGa_{1-x}As$ composition, the Ga cell was changed by -1 °C. The composition value at EpiCenter was updated by the 88-wavelength ellipsometer every 9 s and the Ga cell temperature was set to the proper temperature according to

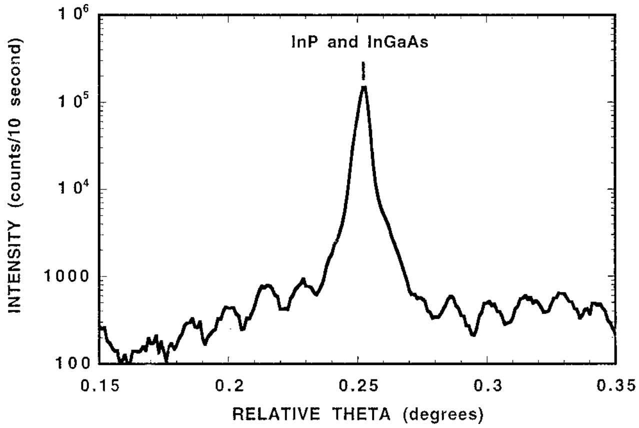

FIG. 4. High resolution x-ray data for sample DCA 150. The $In_xGa_{1-x}As$ layer composition is lattice matched to InP.

the difference in ellipsometer return composition and the desired composition. The Ga cell temperature was not changed within 30 s from the previous Ga cell temperature change or the ellipsometry determined $In_xGa_{1-x}As$ composition is within 0.002 of the desired composition.

The temperature control algorithm used in the substrate and Ga cell temperature is very simple and effective. Two real time *in situ* $In_xGa_{1-x}As$ composition controlled samples, $x=0.54$ and 0.532, were used to demonstrate the absolute precision of the InGaAs composition control. The high resolution x-ray data indicated the $In_xGa_{1-x}As$ was $x=0.542$ and 0.532. The x-ray data for $x=0.532$ is shown in Fig. 4 and the analyzed $In_xGa_{1-x}As$ composition from ellipsometric data and Ga cell temperature is shown in Fig. 5.

III. DISCUSSION

We have compiled a database for $In_xGa_{1-x}As$ lattice matched to InP. Although the database was compiled from the $In_xGa_{1-x}As$ layer after the growth, it is still valid to use

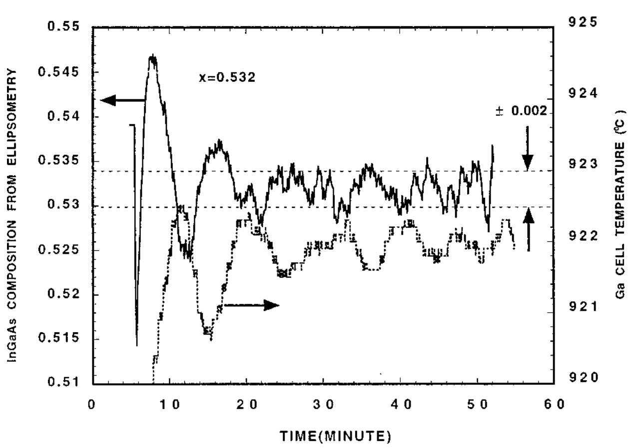

FIG. 5. Solid line is the time plot of the ellipsometry determined $In_xGa_{1-x}As$ composition after the shutter was opened. The dash line is the time plot of the Ga cell temperature controlled by the EPICENTER program to achieve $x=0.532$ for $In_xGa_{1-x}As$ composition. The Ga cell temperature is not changed if the $In_xGa_{1-x}As$ composition is within ± 0.002 of the target value which is 0.532 for sample DCA 150.

it to monitor the $In_xGa_{1-x}As$ composition during the growth by using a proper ellipsometric model. The short term, medium term test of the $In_xGa_{1-x}As$ composition determined from ellipsometry was consistent with high resolution x-ray data as shown in Figs. 2 and 3. It is clear that the $In_xGa_{1-x}As$ composition from 500 and 450 °C growth temperature can be *in situ* measured by ellipsometry with the absolute precision of 0.002. However, the $In_xGa_{1-x}As$ composition at 400 °C growth temperature always shows a larger deviation, 0.005, from the high resolution x-ray data. As indicated in the second set of the experiment, part of the sample was grown with the reverse growth temperature sequence. If the problem is coming from the inability of the substrate temperature determination from DRS with the presence of the thick $In_xGa_{1-x}As$ layers, we will expect to have a larger deviation of ellipsometric composition compared to high resolution x-ray data at 500 °C growth temperature from samples DCA132 and DCA133. We observed from the medium term stability test that the growth temperature sequence does not affect the precision of the ellipsometric determined $In_xGa_{1-x}As$ composition at 500 and 400 °C growth temperatures. It is clear from the medium term stability test that the substrate temperature from DRS is not the reason for lager deviation at 400 °C as shown in Fig. 3.

One possible explanation for the larger deviation of composition at 400 °C is the strong anisotropy in the broadening parameters of the E_1, $E_1+\Delta_1$ transition at 400 °C, or lower growth temperature observed by Philips *et al.*[1] from their *in situ* RDS experiment. Since the database was collected in the static condition after the growth of the $In_xGa_{1-x}As$ layer, the surface structure at 400 °C probably doesn't show the same strong anisotropy effect as in the dynamical condition during growth. The difference of the dielectric response between the dynamical and static condition causes the ellipsometer measured $In_xGa_{1-x}As$ composition to drift away from the real value. The ellipsometric model used in the real time data analysis just cannot take care of the problem in the database and the composition determined by the ellipsometer was as accurate as the higher growth temperature.

We have demonstrated from the short term, medium term, and long term stability the absolute accuracy of $In_xGa_{1-x}As$ composition determined from the ellipsometry to be better than 0.002 by using the static database. We have also demonstrated that we can use this database to do real time *in situ* feedback control of the $In_xGa_{1-x}As$ composition. The absolute precision of the composition is within 0.002 of the target value as shown in Fig. 5. The Ga cell temperature was oscillating in the controlled samples as shown in Fig. 5. This is caused by the time constant to get a stable Ga flux out of the Ga cell and time delay from the $In_xGa_{1-x}As$ composition value determined by signal averaging of ellipsometric data. We can apply a proportional integral differential (PID) type of algorithm to solve this problem. The other drawback is the inability of the ellipsometry to determine the $In_xGa_{1-x}As$ composition in the first 8 min of the thin film growth which is about 500 Å thick.

We have demonstrated the absolute accuracy in determining the $In_xGa_{1-x}As$ composition lattice matched to InP by using our database to be better than 0.002 from the *in situ* ellipsometry experiment. This can also apply to other ternary systems as soon as the database is available. This makes ellipsometry a useful tool in MBE and MOCVD growth for both composition control and thickness control.

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