

Monitoring of the MBE growth processes of CdTe on InSb by laser light scattering

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We have studied by Laser Light Scattering (LLS) the oxides surface desorption of InSb substrates, and the subsequent growth of CdTe layers by molecular beam epitaxy (MBE). LLS measurements allowed us to determine the critical temperature before surface degradation of InSb, which is not evidently noticed by reflection high-energy electron diffraction (RHEED). Surface defects appeared on substrates where this temperature was exceeded, as observed by scanning electron microscopy (SEM) and Atomic Force Microscopy (AFM). During the MBE growth of CdTe on InSb, two features were noticed in LLS measurements. First, a decrease in intensity was observed that can be associated to a change in surface roughness at the initial stages of growth. The second feature is an oscillatory behavior, which can be related to interference. A geometrical model of interference in thin films was used to calculate the layer thickness in real time.

Keywords: MBE, Light Scattering, CdTe/InSb, SEM, AFM

1. Introduction

The *in-situ* determination during molecular beam epitaxy (MBE) of parameters such as roughness, film thickness, growth rate, composition and strain relaxation is very important to fabricate complex, accurate, and defect-free heterostructures. Several techniques like reflection of high-energy electron diffraction (RHEED), mass spectrometry and flux monitoring have been traditionally applied in MBE. However, non-destructive, non-invasive, and low cost probes are needed, such as the optical techniques. Different optical probes have been developed and classified as bulk-oriented, surface oriented, and surface specific, depending on the parameters that can be determined [1]. Laser light scattering (LLS) is classified as a surface-oriented probe. Some of the applications of this technique are: quantitative measurements of haze and other specular defects on silicon surfaces [2], anisotropy of surface scattering correlated with preferential twinning [3], correlation of the LLS intensity signal with the concentration of Ga atoms on the surface of a GaAs substrate [4], detection of defects formed during silicon MBE [5], *in-situ* detection of strained layer relaxation in the InGaAs/GaAs system [6], among others.

In this work, we have applied LLS to the study of the heteroepitaxial growth of CdTe on InSb and GaAs substrates. By using a grazing angle geometry in the experimental arrangement of LLS, we obtained information about surface roughness and epilayer thickness. This information was correlated with RHEED patterns and subsequently with scanning electron microscopy (SEM) and atomic force microscopy (AFM). We demonstrate that LLS is a direct and simple way to determine the critical temperature during substrate preparation, and film thickness in real time during the growth. In particular, we determined the critical temperature for InSb substrates to prevent

surface degradation, and subsequent In droplets formation on the surface. It is also illustrated the use of LLS in *in-situ* monitoring the MBE growth of CdTe on InSb and GaAs substrates.

2. Experimental details

Commercial undoped InSb(111) substrates from MR Semicon Inc. were used in this work. InSb substrates were prepared with the CP4 modified etching solution $\text{HNO}_3:\text{HF}:\text{CH}_3\text{COOH}:\text{DI H}_2\text{O}$ at 2:1:1:10 suggested by W.K. Liu and co-workers [7]. Immediately after etching, the substrates were mounted on molybdenum blocks, and loaded into a Riber 32P MBE system. The base (background) pressure in the MBE chamber was around $\sim 6 \times 10^{-11}$ Torr. A programmed temperature ramp from 100 to 500 °C, at a rate of 9 °C/min, was used to desorb substrate surface oxides. In order to carry out the growths a CdTe cell was heated to 450°C, giving a flux pressure of 1×10^{-6} Torr. CdTe layers were prepared using a substrate temperature of 230°C, and growth rate of ~ 0.6 $\mu\text{m/hr}$.

Figure 1 shows a schematic diagram of the LLS apparatus as used in the MBE reactor. A commercial semiconductor laser of 2mW power was used. The spectral response was measured, and the peak of emission was adjusted to a Gaussian function, obtaining a maximum at 647.4 nm with a width of less than 0.18 nm. The beam was modulated at 167 Hz by a Stanford Research chopper, model SR540. In order to prevent no desired scattered light on the sample edges, an aperture was used to reduce the final size of the beam. The beam was introduced to the UHV growth chamber through a view-port such that the incidence angle was 70° off from the substrate normal (\hat{n} in Fig.1). Scattered laser light was detected with a Si detector installed in a front-upside view port in a position 70° from the sample surface normal, and near to the normal

of the beam plane of incidence (\hat{n}' in Fig.1). The signal was processed with a Stanford Research lock-in amplifier model SR850.

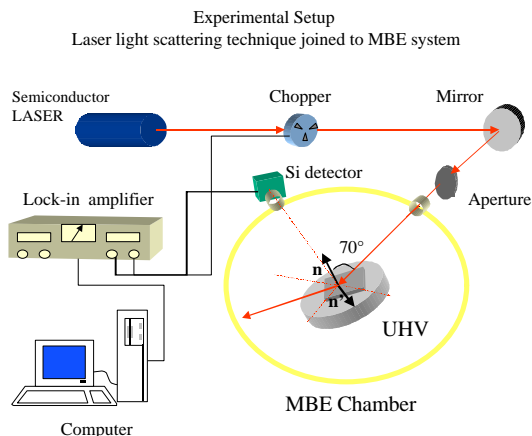


Fig. 1. Laser Light Scattering (LLS) apparatus joined to the MBE chamber for *in-situ* and real time measurements.

RHEED patterns were observed during growth, and digitized using a CCD camera connected to a PC computer. After the growth, the samples were analyzed by scanning electron microscopy (SEM) and in air by atomic force microscopy (AFM).

3. Results and discussion

First we will discuss the desorption of surface oxides on InSb substrates. A careful monitoring of the desorption of these oxides is very important because the critical behavior produced by the large difference in vapor pressures of In and Sb atoms may easily lead to a surface stoichiometry loss. Figure 2 displays a comparison between two different desorbed InSb samples. In Fig. 2(a), we observed a minimum in the LSS signal at about 420 °C, but the temperature ramp was continued and finally stopped at 435 °C, then the temperature was maintained there for 12 minutes. During this time the LLS signal continued increasing reaching a value 80% larger than that at the minimum at 420°C. The LLS signal remained constant, when the temperature was later reduced. The increase in the LLS signal should be caused by an increase in the scattering centers associated to a surface roughening. This LLS behavior indicates an irreversible surface degradation when substrate temperature reaches a value higher than 420 °C.

Moreover, CdTe layers grown on InSb substrates prepared under these conditions presented a very rough surface. Figure 3(a) shows a SEM image of the surface of a ~6000 Å thick CdTe layer grown on the substrate prepared as in Fig. 2(a). Large surface defects ~1µm wide, attributed to an irregular growth on regions where defects were created during the desorption process are clearly observed. The inset in this figure is an AFM image of the surface, an analysis of this surface yields an rms value of 60.3 Å. In contrast, for the sample in Fig. 2(b), the temperature ramp

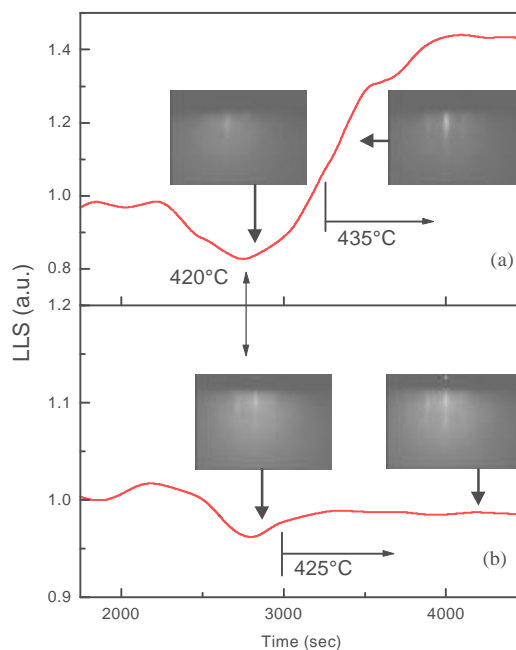


Fig. 2 LLS signal normalized to initial value, around critical temperature with RHEED insets for: (a) sample A, (b) sample B.

was stopped near the minimum of the LLS signal. The LLS signal had a small increase but stabilized at a value only 10% larger than that at the minimum. The substrate temperature was maintained there for 12 min, and then it was decreased to room temperature, the LLS signal remained constant during this process. Figure 3(b) shows a SEM image and the corresponding AFM image of the surface of a ~6000 Å thick CdTe layer grown on this substrate. The surface of this sample is smooth with an rms value of 10.6 Å, thus indicating the improvement in the substrate desorption process by avoiding the increase in the LLS signal intensity. It is important to note that the InSb surface degradation is not clearly detected by RHEED. This can be observed in the insets in Fig. 2(a), showing RHEED patterns at the LLS minimum and at 435 °C, the RHEED patterns look very similar making difficult the control of the desorption process. However, since LLS is very sensitive to large-scale surface roughness the desorption process can be easily controlled using this technique, as shown in Fig. 2(b).

Now we will show that from LLS measurements we can also obtain information of the growth process. For comparison purposes we present the result of the CdTe growth on GaAs and InSb substrates. Figure 4(a), shows the behavior of the LLS signal during the CdTe growth on a GaAs substrate. In this figure we observe that interference oscillations dominate the LLS signal as a function of time. A similar oscillatory behavior has been obtained using light of 1.5µm of wavelength, the produced interference was between light scattered from the surface and from the backside of the wafer [8]. In our experimental apparatus the incidence is at a grazing angle, and the wavelength of the light probe is shorter, so the penetration depth is not enough

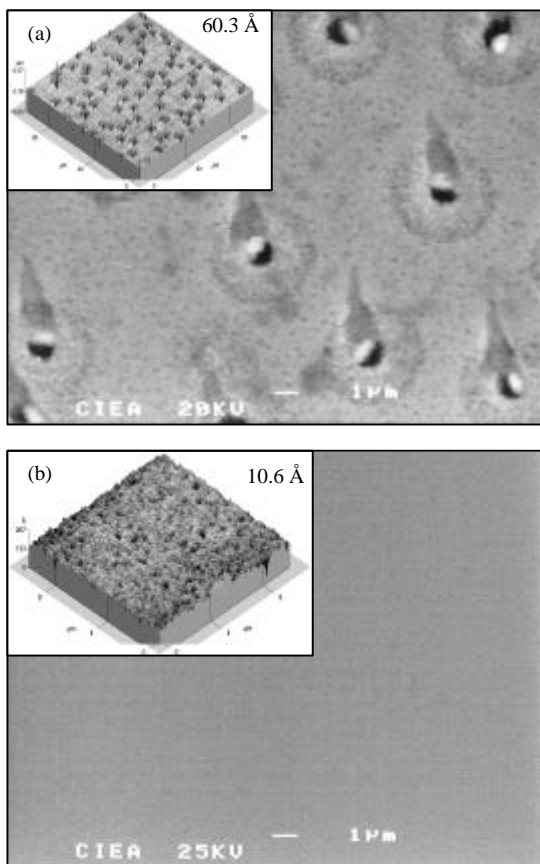


Fig. 3. Surface SEM images with AFM images insets, of CdTe layer grown on InSb substrates for: (a) sample A (b) sample B.

to produce interference from the back side of the wafer. Thus, the oscillations in Fig. 4 are originated from the interference between light scattered at the substrate surface and at the growing layer surface. We can estimate in real time the thickness of the epilayer using a geometrical model of interference in thin films:

$$\Delta q \approx \frac{2p}{l} (2nd \cos j')$$

where d is the film thickness, n is the real refraction index of CdTe, and j' is the angle of the refracted beam on the film. By making $Dq=2p$, we can evaluate the d value. Film thicknesses obtained by this equation were in good agreement with those measured by cross sectional SEM. Now, for the CdTe growth on InSb substrates two principal features are observed in Fig. 4(b). At the start of growth, a strong decrease in LLS intensity was observed, which is attributed to a smoothing of the surface. As growth proceeds the dominant feature is the oscillation in LLS signal due to interference. It is important to note the difference in the LLS behavior for the CdTe growth on InSb and that on GaAs substrates. Growth difficulties are expected for the CdTe growth on GaAs due to the high

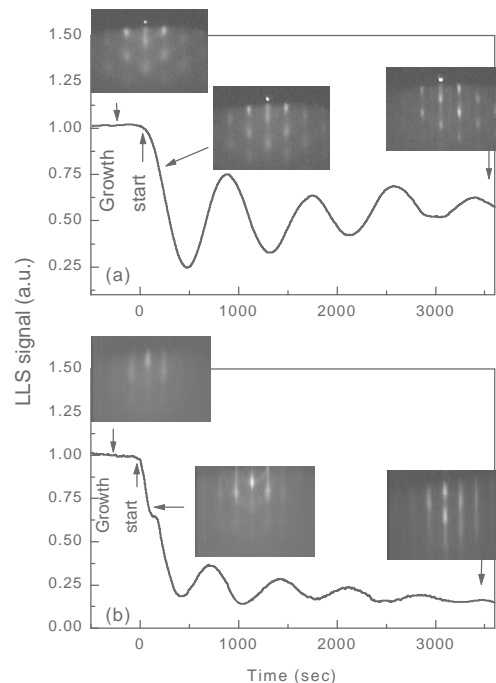


Fig. 4 LLS signal normalized to initial value, during one-hour CdTe growth on: (a) GaAs substrate, (b) InSb substrate. RHEED patterns has been added at different steps of growth.

lattice mismatch (~15%) between these compounds. These difficulties can be observed in the RHEED patterns added in Fig 4(a). Spotty RHEED patterns were observed at the initial stage of growth, indicating an initial three-dimensional growth mode. The RHEED patterns became streakier as growth proceeds indicating a smoothing of the surface. The average LLS signal at the end of growth was about 40% smaller than initial value before the growth. On the other hand, we can expect a smoother growth of CdTe on InSb because the lattice mismatch between these compounds is only 0.05%. This is supported by the streaky RHEED patterns observed during the whole growth process as observed in Fig. 5(b). Note that in this case the final LLS signal was about 80% smaller than value measured before the growth.

Finally it is worth to comment that we have performed the LLS measurements with and without rotating the substrate, obtaining similar results.

4. Conclusion

We have demonstrated specific uses of LLS during MBE growth as *in-situ*, non-destructive, non-invasive, real time probe. We showed that LLS measurements at a grazing angle using a semiconductor laser is a direct and simple way to determine the critical temperature during substrate annealing, surface roughness, and thickness in real time during the growth.

Acknowledgments

The authors acknowledge the technical support provided by A. Guillen, Z. Rivera, R. Fragoso. Also, it is acknowledged the help given by Prof. J. Salas in taking the images of RHEED patterns. J. Huerta acknowledges the financial support granted by CONACyT. This work was partially supported by CONACyT-México.

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