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Raman and AFM studies of swift heavy ion irradiated InGaAs/GaAs heterostructures

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Abstract
The effect of swift heavy ion (SHI) irradiation on InGaAs/GaAs heterostructures is studied using Raman spectroscopy and atomic force microscopy (AFM). The structures consist of molecular beam epitaxy (MBE) grown InGaAs layers on GaAs(001), having layer thicknesses of 12, 36, 60 and 96 nm. After irradiation, the GaAs type longitudinal optical (LO) mode blue shifted to higher frequency in thin samples and red shifted towards lower frequency in thick samples. These results are discussed invoking the penetration depth of the probe radiation ($\lambda = 514.5$ nm) in InGaAs. Deconvoluting the Raman spectra of thin samples indicates a compressive strain developed in the substrate, close to the interface upon irradiation. This modification and diffusion of indium across the interface results in an increase of strain and reduction of the defect densities in the InGaAs layer. The variations in FWHM of the Raman modes are discussed in detail. The surface morphology of these heterostructures has been studied by AFM before and after SHI irradiation. These studies, combined with Raman results, help to identify different relaxation regimes.

1. Introduction

GaAs based heterostructures are promising materials due to their advantages in high frequency operational devices and laser diodes. InGaAs/GaAs heterostructures are important due to their built-in strain and the extra degree of freedom that they give to a variety of applications in opto-electronics [1, 2]. An enormous amount of work has been reported on the characterization

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of strain and the strain relaxation mechanisms of heterostructures grown beyond the critical layer thickness. The most suitable and well established techniques for such studies are high resolution XRD (HRXRD) [3–5], ion channelling [6, 7], electron microscopy [8, 9] and Raman spectroscopy [10–13]. Since the lattice dynamics are affected by stress-induced lattice deformation, the strain in epitaxial layers and its relaxation can be analysed through the evaluation of the phonon frequencies in the Raman spectrum. The relation between phonon frequencies and the strain tensor is described quantitatively by the phonon deformation potential tensor. Through the deformation potentials, the tetragonal distortion induces a splitting of the three-fold degenerate phonon oscillator into a singlet mode whose sub-lattice displacement is perpendicular to the interface and a doublet mode with a displacement parallel to the interface. For Raman experiments in backscattering geometry, the singlet mode is the longitudinal optical (LO) phonon, which is symmetry-allowed, while the doublet mode is the symmetry-forbidden transverse optical (TO) phonon mode. Strain effects of pseudomorphic layers can be observed for lattice mismatches beyond 0.1% [14].

The surface morphology of strained layers reflects the strain relaxation and defect generation at the interface. For thin epitaxial layers, steps are found to be the dominant morphology whereas for thicker films cross-hatch patterns are dominant [15–18]. Atomic force microscopy (AFM) is a simple and powerful tool for qualitative observation of dislocations and a number of surface features related to strain relaxation in the films. Modification of materials by energetic ions has attracted researchers in recent years; in particular swift heavy ion (SHI) modification of heterostructures has been reported by several groups [19, 20]. Recently, SHI-induced modifications of strained InGaAs/GaAs heterostructures and lattice-matched InGaAs/InP heterostructures have been demonstrated by our group [21, 22]. The present work involves Raman and AFM characterization of SHI-induced modification in partially relaxed InGaAs/GaAs heterostructures.

2. Experimental details

The $In_{0.18}Ga_{0.82}As$ layers with thicknesses of 12, 36, 60 and 96 nm were grown on GaAs by molecular beam epitaxy (MBE). The layers were grown at 500 °C with a growth rate of 0.2 nm s^{-1}. SHI irradiation was done at room temperature with 150 MeV Ag^{12+} ions with a fluence of 1×10^{13} ions cm^{-2} from the 15 MV pelletron at IUAC. To avoid heating the samples a low beam current (0.5–2 pnA) was maintained. The samples were oriented at an angle of 5° with respect to the beam axis to minimize channelling. The Raman scattering measurements were carried out at room temperature in backscattering geometry with a 514.5 nm argon-ion laser beam of 75 mW power. The laser beam was focused to a spot size of 50 μm on the sample surface. The scattered photons were collected using a camera lens (Nikon) and a focusing lens. A double monochromator (model Spex 14018) was used for dispersion, and photons were detected using a cooled photomultiplier tube (model ITT-FW 130) operated in counting mode. The slit width of the monochromator corresponding to a spectral line width of about 3 cm^{-1} in terms of the full width at half maximum (FWHM) of the resolution function was employed in the experiments. Surface morphology of the samples was characterized by AFM (in DFM mode using an SPA400, Seiko Instruments Inc.). Hereafter, U and I in the text, table and figures refer to unirradiated and irradiated samples.

3. Strain measurement

Under strained conditions the LO phonon frequency of the InGaAs layer is given by [11, 23, 24]

$$\omega = \omega_0 + 2\Delta\Omega_H - \tfrac{2}{3}\Delta\Omega \tag{1}$$

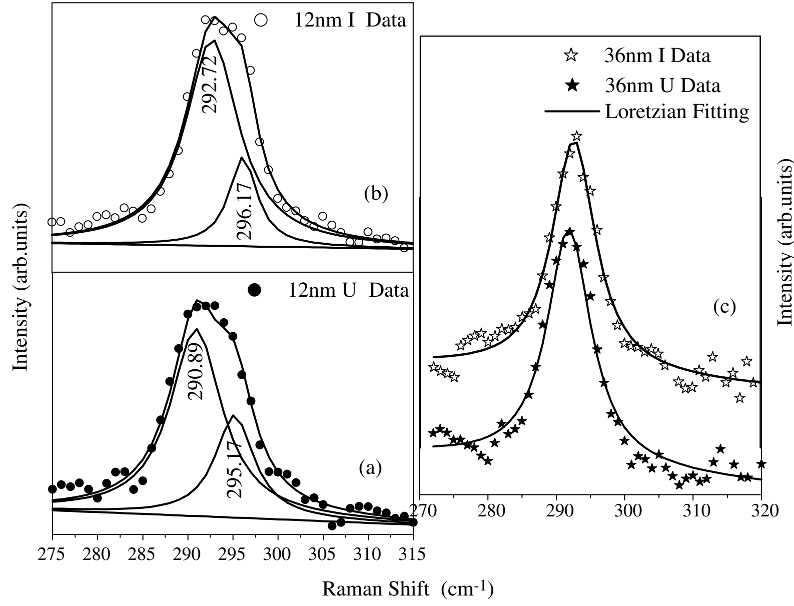

Figure 1. (a) and (b) Deconvoluted Raman spectra of 12 nm thick U and I samples, respectively, and (c) Raman spectra of 36 nm U and I samples.

where ω_0 is the unstrained (bulk) frequency of InGaAs and $\Delta\Omega_{\mathrm{H}}$ and $\Delta\Omega$ are given by [23]

$$\Delta\Omega_{\mathrm{H}} = \frac{p+2q}{6\omega_0^2}\left(\frac{S_{11}+2S_{12}}{S_{11}+S_{12}}\right)\omega_0\varepsilon \tag{2}$$

$$\Delta\Omega = \frac{p-q}{2\omega_0^2}\left(\frac{S_{11}-S_{12}}{S_{11}+S_{12}}\right)\omega_0\varepsilon \tag{3}$$

where the terms involving p and q are deformation potentials and S_{11} and S_{12} are elastic constants. These standard formulae have been used extensively in the literature and show the strain dependence of the LO phonon frequency.

The values of deformation potentials $(p+2q)/6\omega_0^2$ and $(p-q)/2\omega_0^2)$ are $(-)0.891$ and 0.185, respectively, whereas the values of $(S_{11}+2S_{12})/(S_{11}+S_{12})$ and $(S_{11}-S_{12})/(S_{11}+S_{12})$ are 0.028 and 0.047, respectively. These values are obtained from [23] assuming Veggard's law for $\mathrm{In_{0.18}Ga_{0.82}As}$. A negative sign for compressive strain is the convention used here. Replacing the values of these terms in equation (1) we obtain a simple relation

$$\omega = \omega_0 - 1.293\omega_0\varepsilon \tag{4}$$

with ω and ω_0 in units of $\mathrm{cm^{-1}}$. This equation predicts the shifts due to the strain in the layer.

4. Results and discussion

The Raman spectra of the samples are given in figures 1 and 2. InAs type modes were hardly observed, probably due to a lower indium concentration [10, 11]. In the present experimental configuration, the GaAs type TO mode is forbidden. However, a weak TO mode at about 270 $\mathrm{cm^{-1}}$ appears due to strain relaxation-induced defects in the samples. The usual intense peak of the GaAs type LO mode around 290 $\mathrm{cm^{-1}}$ is also observed [12, 13]. The results are discussed in the light of penetration depth of the probe laser beam used in the experiment. The

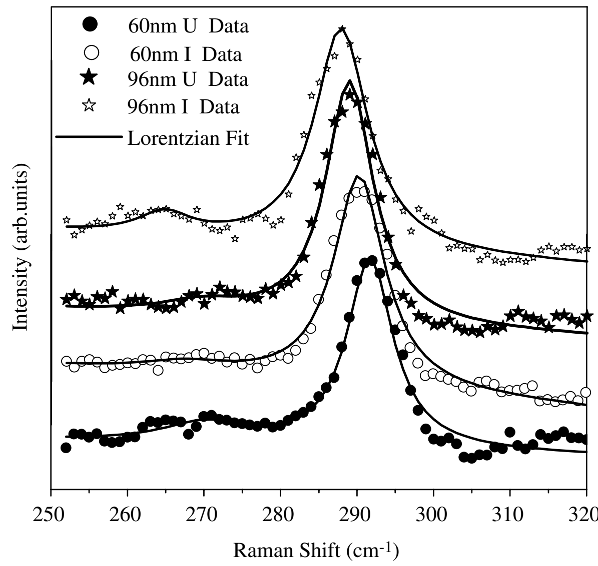

Figure 2. Raman spectra of 60 and 96 nm thick U and I samples.

Table 1. Parameters measured from Raman and AFM.

$In_{0.18}Ga_{0.82}As/GaAs$ samples (layer thickness) (nm)	FWHM of GaAs type LO mode (cm^{-1})		Blue shift in GaAs type LO mode (cm^{-1}) U		Compressive strain (%) using equation (4)		AFM-RMS roughness (nm)	
	U	I	U	I	U	I	U	I
12	5.224	3.780	7.63[a] (4.64[b])	8.63[a] (6.12[b])	2.05[a] (1.25[b])	2.32[a] (1.67[b])	3.96	3.05
36	7.823	7.747	4.26	5.36	1.20	1.44	1.54	1.30
60	6.958	8.655	4.06	2.73	1.09	1.01	2.62	4.31
96	7.984	9.466	1.30	0.38	0.35	0.12	2.62	2.36

[a] Strain values calculated from the deconvoluted spectra.
[b] Strain values calculated from the as-recorded spectra.

penetration depth of a 514.5 nm laser beam in GaAs is about 55 nm and in InAs it is about 15 nm in defect free crystals. These values are significantly reduced by disorder present in the crystal [12]. Hence, in the present study, the interface of 12 and 36 nm thick samples is within the penetration depth. However, for 60 and 96 nm thick samples, the interface is beyond the penetration depth of the probe beam.

The bulk equivalent (strain free) GaAs type LO mode (ω_0) for $In_{0.18}Ga_{0.82}As$ is taken from [24] as 287.54 cm^{-1} and is used in equation (4) for strain measurements. A blue shift for compressive strain (negative ε) is observed as predicted by equation (4) and the calculated strain values are given in table 1. For the unirradiated samples, the strain decreases as a function of thickness; this indicates that the onset of strain relaxation is around 12 nm. Very low strain values for thick samples indicate a strong relaxation of strain in the near-surface regions. After irradiation, a blue shift of the Raman mode, implying an increase of strain in thin layers, and a red shift, implying a decrease of strain in thick layers, were observed. This is probably due to the difference in the ion beam modifications near the interface and near the surface. Additional compressive strain would have been induced in the regions close to the interface

upon irradiation. But close to the surface, irradiation might have reduced the strain. Beyond the critical layer thickness, the strain relaxes giving rise to dislocations. But this relaxation is not abrupt at the critical thickness as there are various stages of relaxation. Hence, beyond the critical thickness the strain energy decreases at various stages. The above conjecture about different effects of ion beams at the interface and near-surface regions suggests that initial strain energy plays a crucial role in such modifications.

The 12 nm thick samples have a broad GaAs type LO mode, which is due to the contribution from the GaAs substrate as the probe beam also penetrates into the substrate. Figures 1(a) and (b) shows the deconvoluted spectra of 12 nm thick U and I samples, respectively. The mode observed around 290.89 cm^{-1} corresponds to the bulk GaAs (reported earlier [25]) and the mode around 295 cm^{-1} is from the InGaAs layer. Calculation of strain using this value in equation (4) gives a higher value than the ones measured from the convoluted spectra as given in table 1. In other samples, such a difference in strain was negligible due to a lesser or no contribution of GaAs LO mode from the substrate. After irradiation, the GaAs type LO mode from the layer as well as from the substrate of the 12 nm thick sample shifts towards higher frequency (figure 1). This indicates that a compressive strain has been developed in the substrate near the interface due to irradiation. This may reduce the misfit at the interface and induce a compressive strain in the over layer as evidenced from the increase in the blue shift of the LO mode contribution from the InGaAs layer. A compressive strain in the substrate region near the interface was observed in all the irradiated samples using HRXRD [26]. Such a compressive strain in the GaAs substrate was attributed to ion-induced damage below the interface region. The HRXRD [26] and RBS/channelling [27] results on the same set of samples indicate that the strain is induced and defect densities are reduced after irradiation in thick samples also. The reduction of defects due to irradiation as characterized by HRXRD and RBS/channelling was attributed to ion-induced damage in the GaAs substrate near the interface and inter-diffusion of indium across the interface.

The FWHM of GaAs type LO modes are given in table 1. The FWHM decreases for thin samples and increases for thick samples upon irradiation. It is to be noted that the irradiated samples were not subjected to a thermal annealing process after irradiation. An intense LO mode comparable to that of as-grown samples was observed, and the intensity of the TO mode decreased after irradiation. This is in contrast to the low energy irradiations where a broad and intense disorder-activated TO (DATO) mode is observed. Also, the intensity of the symmetry allowed LO mode decreases and post-annealing is required to recover the symmetry allowed phonon modes [28–31]. This highlights that in the present work the modifications are solely by electronic energy loss and that discernible lattice damage has not been created due to irradiation. For thin samples, the SHI irradiation results in decrease of FWHM, increase of strain values (table 1) and decrease of the TO/LO mode intensity ratio (not shown). This may be attributed to the reduction in defect density and probably not to the inhomogenity in strain and/or composition. In contrast, for thick samples, the FWHM increases, the strain value decreases (table 1) and there is no significant change in the TO/LO mode intensity ratio after irradiation. This may be due to inhomogeneity in strain and/or composition [32].

The surface morphology of 36 and 60 nm thick samples is shown in figures 3 and 4, respectively. The 3 μm^2 area scans for both unirradiated and irradiated samples are given for comparison. The surface morphology of 36 and 96 nm thick samples is qualitatively similar showing protrusions (though the protrusion density and size differ) and cross-hatch patterns. The morphology of the 60 nm thick sample is different, showing deep holes varying in width between 175 and 200 nm and with a depth (Δz) variation between 9 and 16 nm; the cross-hatch pattern is also very faint. For the 60 nm thick irradiated sample, the surface roughness increased (table 1) with increase in hole depth varying between 11 and 22 nm, and the width

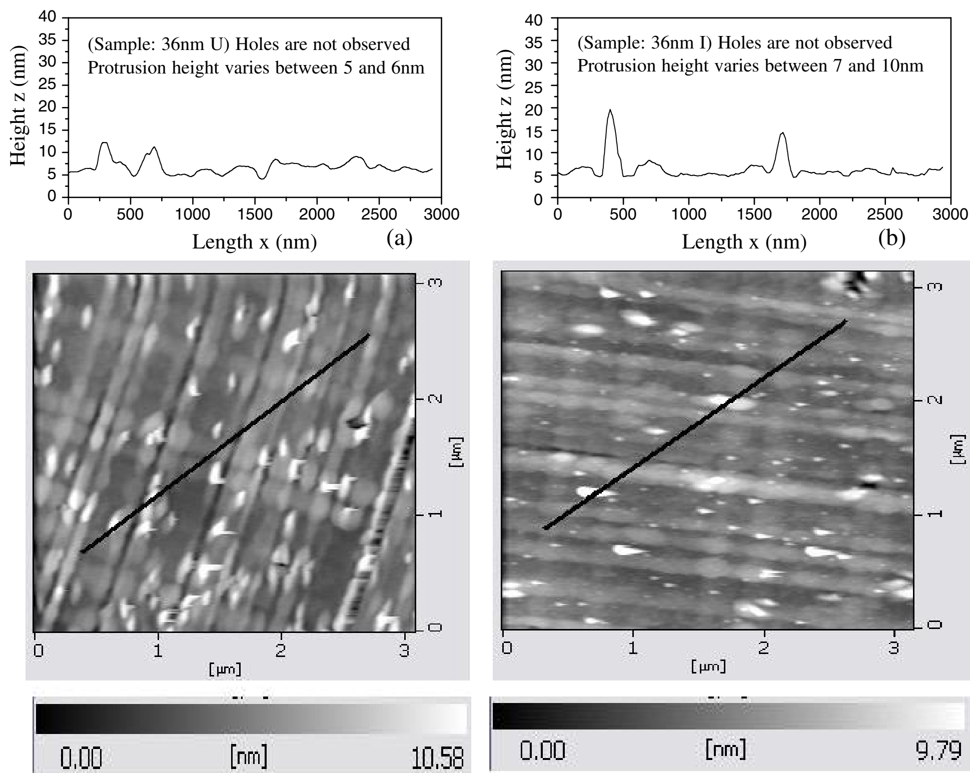

Figure 3. AFM of 36 nm thick (a) unirradiated and (b) irradiated samples.

did not change noticeably. The observed reductions in surface roughness after irradiation for 12, 36 and 96 nm thick samples are smaller, but the increase in roughness for the 60 nm thick sample after irradiation is comparably high. Recently, AFM studies on similar samples have been reported, attributing different surface morphologies to slow, rapid and saturated regimes of strain relaxation [18]. The HRXRD results [26] on strain measurements for the present set of samples indicate that the relaxation is about 55% for the 96 nm thick sample. In comparison with the strain relaxation curve as a function of thickness in [18], the present set of samples have a higher relaxation and hence higher slope. Even in the present studies, the morphological differences may be due to different relaxation regimes, as reported in [18]. Due to higher relaxation, different relaxation regimes may be observed at much smaller thicknesses than those reported in [18] as evidenced from different surface morphologies. The irradiated samples show better surface morphology and reduced roughness values (except for the 60 nm thick sample). The increase in strain and reduction of FWHM of the Raman modes in thin samples, implying the reduction in defect densities, are consistent with the reduction in surface roughness obtained from AFM. The reduction of strain, increase in FWHM of Raman modes and increase in surface roughness in the 60 nm thick sample obtained from AFM may be due to inhomogeneity in strain and composition. Further relaxation of strain and reduction of surface roughness in the 96 nm thick sample is also expected as higher strain relaxations reduce the surface roughness [18]. Thus, different relaxation regimes and different effects of irradiation have been characterized by combining Raman results and AFM.

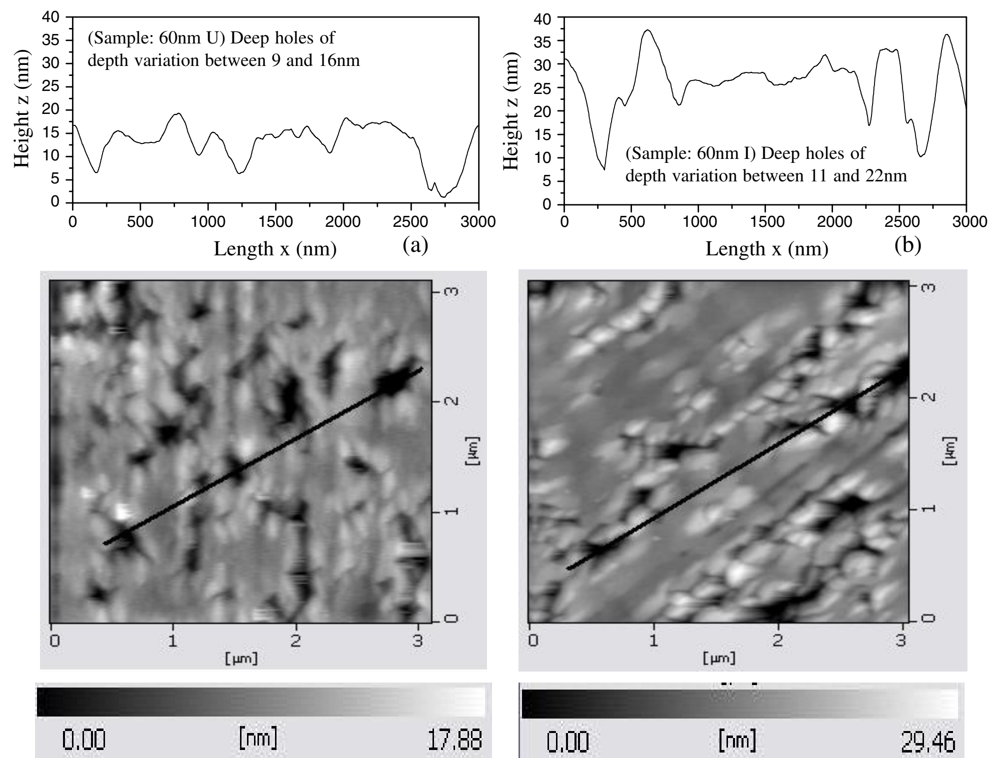

Figure 4. AFM of 60 nm thick (a) unirradiated and (b) irradiated samples.

5. Conclusions

Strain modification of SHI-irradiated InGaAs/GaAs heterostructures using Raman spectroscopy has been reported for the first time. The Raman results are discussed in the light of the penetration depth of the probe beam. The intense LO mode and weakening of disorder-induced modes observed in all irradiated samples without post-thermal annealing highlights the lower damage-creating capacity of SHIs in contrast to low energy ions. Different effects of heavy ion irradiation are observed near the surface and at the interface. The role of initial strain energy for different irradiation effects is highlighted. A compressive strain in the substrate region near the interface and diffusion of indium across the interface due to irradiation is responsible for the observed modifications. The decrease of FWHM of the Raman modes after irradiation may be due to reduction in the defect density and not due to inhomogeneity in strain and composition. The corresponding increase may be due to inhomogeneity in strain and composition. The surface modification of the samples studied by AFM is correlated with the Raman results and different relaxation regimes could be identified. The results are comparable with other complementary techniques also, namely HRXRD and RBS/channelling, where it has been concluded that the strain is induced in all the samples after irradiation irrespective of the thickness. In conclusion, modification of InGaAs/GaAs heterostructures without considerable lattice damage is possible using SHI irradiation. Correlation of Raman and AFM results gives a better insight into the interface and surface modifications.

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