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Mechanisms of strain release in molecular beam epitaxy grown InGaAs/GaAs buffer heterostructures

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Abstract

Strain release and distribution in double InGaAs/GaAs heterostructure buffer layers were studied. A higher misfit dislocation density at the inner interface between the InGaAs layer and the substrate was found in all the samples. This corresponded to a strain release of the inner ternary layers which was much larger than that predicted by equilibrium theories. The residual parallel strain of the external layers as a function of the thickness was found to follow a curve of slope −0.502, in agreement with previous results on single InGaAs layers. These results were interpreted as evidence that the elastic energy per unit interface area remains constant during growth. The presence of numerous dislocation loops inside the substrate was considered to be responsible for the strain relaxation occurring through dislocation multiplication due to Frank–Read sources activated during growth. A comparison with InGaAs/GaAs step graded heterostructures is also discussed. Finally, lattice plane tilts between epilayers and substrates were found and attributed to the imbalance in the linear density of misfit dislocations with opposite components of the Burgers' vector ($b_{\text{eff}}^{\perp}$) perpendicular to the interface.

Keywords: Defect formation; X-ray spectroscopy; Electron microscopy; Interfacial stress

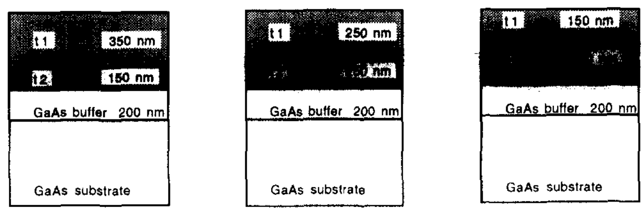
1. Introduction

Highly mismatched epitaxial layers with very low dislocation densities can be grown on GaAs substrates by using suitable buffer heterostructures able to confine dislocations far from the active regions of the devices [1–3].

In order to study the confinement of dislocations in the buffer–substrate interface and the mechanism of strain release in multiple structures (following our previous work on InGaAs/GaAs single and superlattice buffer layers [4, 5]), molecular beam epitaxy (MBE) grown $In_xGa_{1-x}As/GaAs$ double heterostructures, with nominal In content of $x = 0.05$ and $x = 0.10$ and a total nominal thickness of $t = 500$ nm (Fig. 1) were studied by Rutherford backscattering spectrometry (RBS) and channelling techniques, high-resolution X-ray diffraction (HRXRD) and transmission electron microscopy (TEM). The top InGaAs layer corresponded to the higher In content. RBS and HRXRD techniques were employed to measure the composition, thickness and degree of strain release of the samples. Cross-sectional TEM (XTEM) investigations were carried out to study the dislocation nature, distribution and density inside the structures.

2. Experimental details

The structures were grown at ICMAT in a conventional MBE system on (001) oriented, Si-doped GaAs substrates with an average dislocation density of



S 88 S 89 S 90

Fig. 1. Sketch of the structures investigated. $In_xGa_{1-x}As/GaAs$ double heterostructures; $x_{\text{nom}}(\text{t1}) = 0.10$, $x_{\text{nom}}(\text{t2}) = 0.05$.

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5.5×10^2 cm^{-2}. The growth temperature was 530 °C for both buffer and ternary layers.

RBS channelling measurements were carried out at the Laboratori Nazionali di Legnaro using a high precision goniometer sample holder and a $^4He^+$ beam of 2 MeV energy [6].

TEM analyses were performed at MASPEC in a 2000FX JEOL microscope working at 200 kV on (110) oriented, cross-sectioned samples mechanochemically thinned and then finished by room temperature Ar ion milling.

X-Ray measurements were performed at MASPEC on a double crystal diffractometer in the 117 Cu Kα parallel geometry, corresponding to a Bragg angle of 76.64° and an asymmetry angle $\phi=\pm11.4°$ depending on the reflection geometry.

The 117 reflection gave a large peak splitting, thus allowing the better separation of the layer peaks in the diffraction profile. In order to obtain the lattice mismatches parallel ($(\Delta d/d)^{\parallel}$) and perpendicular ($(\Delta d/d)^{\perp}$) to the (001) surface, the measurements were performed in both the grazing incidence (positive ϕ) and grazing emergence (negative ϕ) geometry. For each geometry, four independent measurements were repeated after successive 90° rotations around the surface normal. To avoid the effect of the small (less than 0.5°) deviations of the physical surface from the nominal (001) crystallographic plane and of rotation of the layer lattice with respect to the substrate because of the dislocation network at the interfaces, the averages of the peak splitting after 180° rotations were taken. In this way, two independent measurements of $\Delta d/d$ in the scattering planes corresponding to the 0°–180° and 90°–270° rotations were obtained. The mismatch values $\Delta d/d$ were calculated from the measured values of the peak splitting (Δ_{Tot}) using the exact formula

$$\Delta_{\mathrm{Tot}}=\Delta\Phi+\Delta\Theta_{\mathrm{B}}=\Phi-\tan^{-1}\left[\tan\Phi\,\frac{1+(\Delta d/d)^{\perp}}{1+(\Delta d/d)^{\parallel}}\right]+\sin^{-1}\left[\sin\Theta_{\mathrm{B}}\left(\frac{\sin^2\Phi}{[1+(\Delta d/d)^{\parallel}]^2}+\frac{\cos^2\Phi}{[1+(\Delta d/d)^{\perp}]^2}\right)^{1/2}\right]-\Theta_{\mathrm{B}} \quad (1)$$

where $\Delta\phi$ is the tilt of the lattice planes due to the deformation of the layer lattice and $\Delta\Theta_{\mathrm{B}}$ is the change of the Bragg angle.

3. Results and discussion

The results of both RBS and HRXRD measurements are reported in Table 1. The residual parallel strain values $\varepsilon^{\parallel}$ were calculated from the composition values determined by RBS and HRXRD from the following equation

$$\varepsilon^{\parallel}=\frac{a^{\parallel}-a^0}{a^0} \quad (2)$$

where a^0 is the relaxed lattice parameter determined from Vegard's law and the RBS and HRXRD composition values.

The residual strain values vs. the layer thickness are shown in Fig. 2. The points labelled t2, corresponding to the deeper buffer layers, are reported considering their individual thickness. The top layers show residual strain values much larger than predicted by the model of Matthews and Blakeslee [7]. In contrast, despite the lower In content, the deeper InGaAs buffer layers exhibit a much larger strain release and appear nearly completely relaxed. This simply shows that the strain release of the first ternary layer depends on the total thickness of the structure which must be considered as a whole and not as two individual layers.

In all the samples, (110) oriented XTEM investigations showed a higher misfit dislocation density at the inner interface between the InGaAs layer and the substrate (Fig. 3). Besides the misfit dislocations, dislocation loops extending from the deeper interface inside the GaAs substrate were also found, as shown in Fig. 4 for specimen S88. Both misfit dislocations and dislocations in each set of loops were of 60° type with a Burgers' vector of $a/2[110]$ type on a similar {111} glide plane.

Since the dislocation density at the InGaAs/GaAs interface is nearly ten times higher, this demonstrates that suitable buffer layers of intermediate composition

Table 1
Experimental values of composition x, thickness t and residual strain $\varepsilon_{\mathrm{res}}$ of the specimens investigated. The average error values are also reported

Sample	$x(t_1)$ (%) ($\pm0.3\%$)	$x(t_2)$ (%) ($\pm0.3\%$)	t_1 (nm) (±5 nm)	t_2 (nm) (±5 nm)	$\varepsilon_{\mathrm{res}}(t_1)$ ($\pm1\times10^{-4}$)	$\varepsilon_{\mathrm{res}}(t_2)$ ($\pm1\times10^{-4}$)
S88	12.1	6.45	340	143	3.11×10^{-3}	0.44×10^{-3}
S89	12.3	6.8	244	239	3.49×10^{-3}	0.33×10^{-3}
S90	11.0	5.8	145	343	4.16×10^{-3}	0.92×10^{-3}

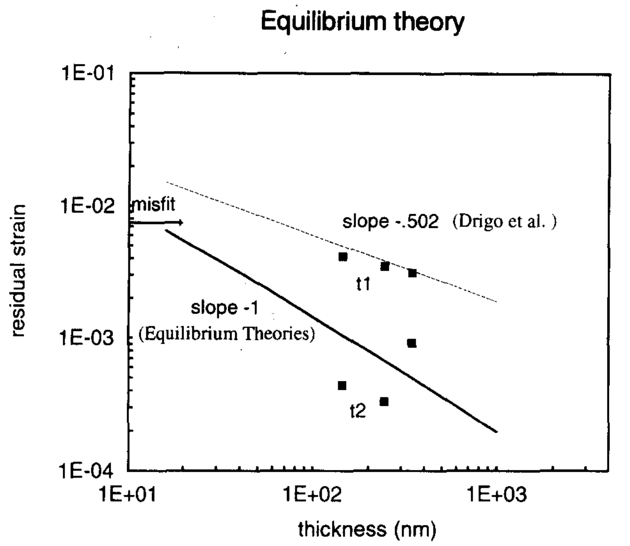

Fig. 2. Residual strain vs. specimen thickness. Full line, equilibrium theories; broken line, experimental curve previously obtained [4] for different InGaAs/GaAs single buffer layers. The points labelled t1 correspond to the three upperlayers of the structures reported in Fig. 1. The points labelled t2 correspond to the inner layers.

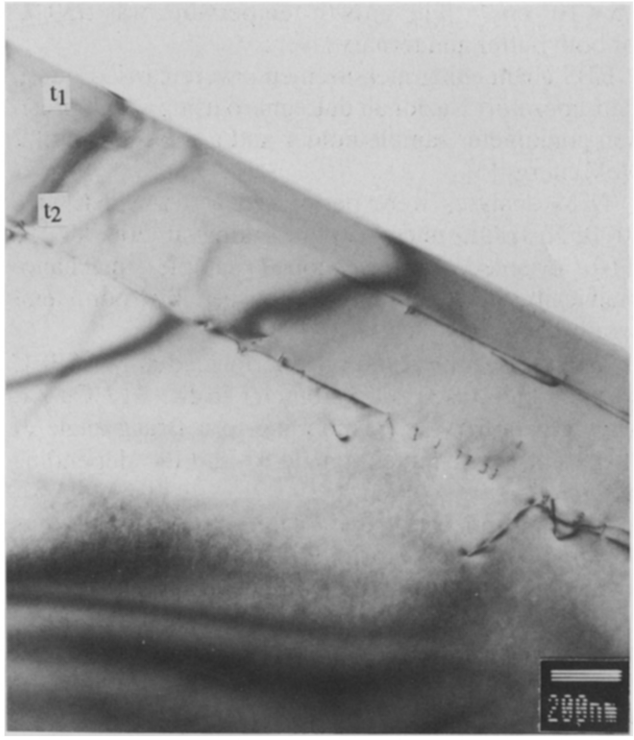

Fig. 3. (110) Oriented, cross-sectional transmission electron micrograph of sample S90. The higher dislocation density at the deeper heterointerface is shown. $g=004$ type.

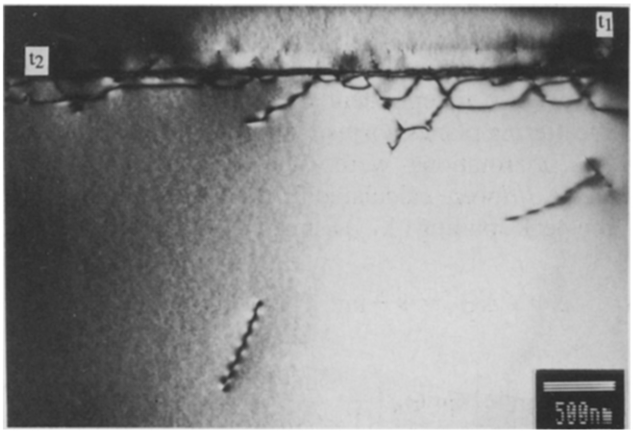

Fig. 4. Bright field, zone axis, (110) oriented, XTEM picture of sample S88. Extended dislocation loops inside the GaAs substrate are clearly visible.

are effective in reducing the dislocation density in regions with higher In content.

The residual parallel strain values of the three upper layers (t1) are compared in Fig. 2 with previous results (broken line) on InGaAs/GaAs single heterostructures of similar composition and thickness [4]. As in the previous experiments, $\varepsilon^{\parallel}$ vs. t values for the three structures investigated in this work approximately follow a unique curve of $-1/2$ slope (the error bar is slightly larger than the dimension of the filled squares) independent of misfit [4] and in disagreement with equilibrium theories [7, 8]. This result leads to the assumption that, once the critical thickness is overcome, the elastic energy per unit interface area remains constant [9].

On the basis of this assumption, we can also explain the behaviour of the double layer heterostructures. In particular, it follows that the strain release of the inner layer must continue during the growth of the top InGaAs layer. The linear dislocation density of the inner interface evaluated from the measured $\varepsilon^{\parallel}$ values is about $(3-4)\times 10^5$ cm^{-1}, in good agreement with the misfit dislocation linear density measured by XTEM maps. The number of misfit dislocations per unit length inside the structures is about one order of magnitude higher than that which could be induced by the pre-existing dislocations threading from the substrates used in this experiment. As a consequence, in order to release the excess of strain inside the inner layer due to the growth of the upper layer, new dislocations must be nucleated. Since the dislocation density is higher at the ternary-binary interface, it follows that the strain release occurs first at the InGaAs/GaAs interface.

The inner layer can be considered to be free standing during growth and its $\varepsilon^{\parallel}$ value should follow the broken line in Fig. 2. The points labelled t2 in Fig. 2 indicate that a stronger strain relaxation occurs because of the growth of the external InGaAs layer. The real lattice mismatch at the beginning of the growth of the upper layer is that which competes for the residual strain inside the inner layer. The relative lattice mismatch between the InGaAs layers depends on the strain relaxation of the inner layer and changes continuously during growth, until complete strain release occurs at the inner layer. This mechanism is not surprising, since it is more convenient from the point of view of energetics to release the maximum amount of strain at the first ternary-binary interface rather than equally distributing the strain release between the two interfaces.

From the experimental observation that the number of misfit dislocations at the deeper heterointerface is higher than the available sources in the substrates, it follows that a multiplication process must be active to create new dislocations to release the strain. Transmission electron micrographs show the presence of several extended dislocation loops at the InGaAs/GaAs interface, propagating into the substrate. The presence of these loops has been related to Frank-Read sources that are activated during growth for generating misfit dislocations [10]. The dislocation intersections inside the network of misfit dislocations, due to the pre-existing threading dislocations from the substrate, behave like Frank-Read sources [11].

XTEM maps obtained from micrographs, such as that shown in Fig. 4, seem to reveal that the density and dimensions of the extended loops are much higher in the two specimens that present the highest strain release (S88 and S89 in Table 1). This observation suggests that both the loop dimensions and density are releated to the strain relaxation of the inner layers. Therefore the misfit dislocation density at the deeper interface must increase by means of some dislocation multiplication process within the crystal at a lower energy barrier than that required for direct nucleation of dislocations. As a consequence, this result seems to indicate that, as suggested by Matthews [12] for similar misfit values, it is not necessary to invoke the generation of half loops from the specimen surface to account for the strain release.

As a natural extension of this work, a similar study was performed on an MBE grown InGaAs/GaAs step graded buffer structure such as that sketched in Fig. 5(a). XTEM investigations (Fig. 5(b)) revealed that misfit dislocations are mainly concentrated at each interface and propagate from the InGaAs/GaAs interface through the structure with decreasing density, leaving some thousand Ångströms from the surface with a very low density of defects. This shows the reliability of the structures as buffer layers for reducing the dislocation density at the specimen surface as already found by other groups [1, 13].

The behaviour of the step graded structure, very similar to the double structure described above, indicates that the number of layers necessary to design prefixed surface lattice parameter and residual strain values is related to the maximum composition step between successive layers. The maximum composition step is determined by the two-dimensional-three-

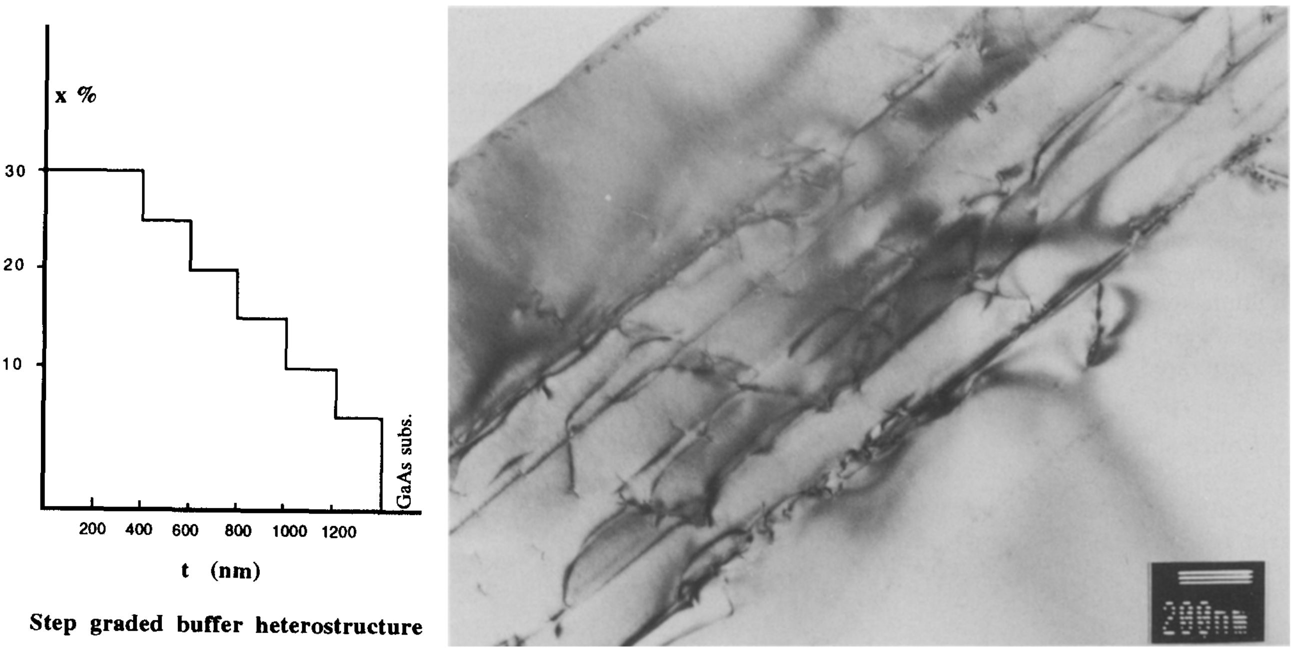

Fig. 5. (a) Sketch of the step graded buffer heterostructure. (b) (110) Oriented, bright field, zone axis, cross-sectional transmission electron micrograph of the step graded structure. Dislocations propagating from the deeper interface through the structure are shown. The highest dislocation density is at the InGaAs/GaAs interface.

dimensional growth transition regime. Work is in progress to develop a numerical model to account for the strain release in multiple structures.

Following the work of Ayers et al. [14] and Kavanagh et al. [15], further HRXRD investigations were performed to study the correlation between the dislocation type and the tilts between epilayers and substrate. Within the experimental accuracy, which is ±1% for the determination of the perpendicular strain and ±5% for the parallel strain, no asymmetry in the parallel strain release was observed. Nevertheless, evidence of a small tilt (300–400 arcsec) beween the buffer layer lattice and the substrate lattice was found in all the samples. This tilt value, observed by HRXRD after a 180° rotation along the sample surface axis with the same diffraction geometry, was due to the low angle grain boundary produced by the dislocation network at the buffer layer–substrate interface. The tilt value α is related to the unbalance $(\rho^+ - \rho^-)$ in the linear density of dislocations having opposite $b_{\mathrm{eff}}^{\perp}$ components of the Burgers' vector perpendicular to the interface [16]

$$\alpha = b_{\mathrm{eff}}^{\perp}(\rho^+ - \rho^-) \tag{3}$$

Assuming that only 60° dislocations with $b^{\perp} = a/2$ are present, a tilt of 400 arcsec corresponds to a linear dislocation density $\rho = 6.84 \times 10^4$ cm^{-1}. A comparison with the dislocation density determined by TEM and HRXRD, 1.14×10^5 cm^{-1} for sample S88 (that presents the highest density), indicates that the majority of the dislocations have the same perpendicular component of the Burgers' vector. A similar result obtained by Kavanagh et al. [15] in InGaAs/GaAs samples cut 2° off the (100) planes was interpreted as being due to the different strain release associated with dislocations having opposite perpendicular components of the Burgers' vector. In the present case, the very low surface miscut angle cannot explain such a difference. Since the low dislocation density for the Si-doped GaAs substrate (about 5.5×10^2 cm^{-2}) can accommodate only a small part of the strain according to the Matthews model, we conclude that the dislocation multiplication mechanism is responsible for such a difference, and that mainly dislocations of the same type are generated.

4. Conclusions

Double InGaAs/GaAs heterostructure buffer layers were revealed to be effective in confining misfit dislocations at the deeper interface. The strain release behaviour was explained on the basis of previous results on single InGaAs/GaAs layers, showing that the elastic energy per unit interface are remains constant. The strain relaxation occurs through dislocation multiplication due to Frank–Read sources activated during growth, as shown by numerous dislocation loops inside the substrate. Lattice plane tilts between epilayers and substrates of the order of magnitude of 400 arcsec were found. The tilt was attributed to the unbalance in the linear density of misfit dislocations with opposite $b_{\mathrm{eff}}^{\perp}$ components of the Burgers' vector perpendicular to the interface. Finally, the possibility of growing buffer layers with prefixed residual strain and composition has been shown to be related to the maximum concentration step between successive layers.

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