## Original Paper

# **Composition Modulation in Low Temperature Growth** of InGaAs/GaAs System: Influence on Plastic Relaxation

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Abstract. This work focuses on the characteristics of composition modulation in InGaAs/GaAs layers grown by ALMBE at 200 °C, and its influence on the degree of plastic relaxation that these layers achieve. The asymmetry in the composition modulation for both  $\langle 110 \rangle$  directions lying in the (001) growth plane observed by Transmission Electron Microscopy is proposed to explain the asymmetry enhancement in misfit dislocations density, as has been found in these structures. Internal stresses in the material associated with this phase separation could be responsible for the hardening of these low temperature grown InGaAs layers.

**Key words:** Composition modulation; InGaAs/GaAs; TEM; strain-hardening; low temperature growth.

Over the last decade, InGaAs/GaAs hetrostructures have been of great interest because of the possibility of reaching the technologically important 1.3–  $1.55 \mu m$  wavelength range. Nevertheless, as a result of structural problems related to plastic relaxation that appear when increasing the In content, nowadays dilute nitrides alloys (InGaAsN), that present an anomalously large bandgap bowing, are being considered as an alternative for optoelectronic applications. Recent studies of InGaAsN alloys [1] have shown, however, that in this promising alloy the problems of phase separation observed previously in InGaAs seem to be more intense and even more complex because there is In and N segregation involved.

The aim of this article is to provide further insight into the role of the composition modulation in the plastic relaxation behaviour of InGaAs, knowledge that could be extended to the quaternary system in a future. Although a large number of works have focused on composition modulation and on plastic relaxation in III–V alloys separately, just a few studies about the correlation between them have been developed. We report a Transmission Electron Microscopy (TEM) study of the influence of phase separation on plastic relaxation in modulated InGaAs layers grown at low temperature (200 °C).

### **Experimental**

InGaAs layers with a nominal Indium content of 20% were grown on on-axis GaAs(001) by atomic layer molecular beam epitaxy (ALMBE) at 200 °C. The thickness of the layers are 80 nm, 150 nm and 300 nm. These three samples underwent a postgrowth thermal annealing (TA) at 500 °C for half an hour. A layer of 300 nm without thermal annealing has also been considered. All the structures were prepared for cross section transmission electron microscopy (XTEM) by mechanical grinding and polishing followed by  $Ar^+$  ion beam etching to electron transparency. High resolution Xray diffraction (HRXRD) characterization has been carried out, too.

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#### Results

Figure 1 shows g220 bright field TEM micrographs of both [110] and  $[1\overline{1}0]$  orientations lying in the (001) growth plane of the 300 nm thick InGaAs film without thermal annealing. In the image taken from the [110] orientation, vertical periodic bright and dark contrasts are observed in the epilayer, with a wavelength of approximately 25 nm. These contrasts have been widely associated to composition modulation [2, 3] produced by a spinodal decomposition process. The perpendicular section  $[1\overline{1}0]$  exhibits long-wavelength contrasts (60–70 nm), showing that this layer is highly asymmetric with regard to contrast modulation. On the other hand, this sample remains nearly coherent to the substrate, given that the plastic relaxation degree measured by HRXRD for both  $\langle 110 \rangle$  directions is  $1\% \pm 1\%$  (although few areas of high dislocations density have been observed by TEM, see Fig. 1b). In the annealed sample of the same thickness (300 nm) similar periodic contrasts have been observed, which indicates that the application of this





Fig. 1. XTEM images of the 300 nm thick InGaAs sample without thermal annealing with g220 BF reflections: (a) [110] orientation, where fine speckle contrasts are observed; (b)  $[1\bar{1}0]$  orientation, where coarse contrasts appear



Fig. 2. g220 BF image of the homogeneous 80 nm thick InGaAs film

thermal treatment seems not to affect phase separation in these structures. Nevertheless, the TA does influence on plastic relaxation increasing it in an asymmetric way, since the plastic relaxation degree in the annealed structure is  $37\% \pm 2\%$  for [110] direction and  $21\% \pm 2\%$  for the perpendicular one.

On the contrary, TEM study of samples with epilayer thickness of 80 nm (Fig. 2) and 150 nm show that these layers do not exhibit the composition modulation associated contrasts mentioned above, therefore these layers seem to be homogeneous. These results indicate that composition modulation is highly dependent on the film thickness of these low temperature grown samples, appearing when a critical layer thickness is reached, as we discussed in a previous work [4]. With regard to plastic relaxation, the values measured by HRXRD are  $32\% \pm 3\%$  for [110] direction and  $30\% \pm 2\%$  for [110] in the 80 nm film and  $51\% \pm 1\%$  ([110]) and  $45\% \pm 3\%$  ([110]) for the layer of 150 nm.

Plastic relaxation in InGaAs/GaAs(001) homogeneous layers is slightly asymmetric, due to the differences both in the activation energy of formation [5] and in the glide velocity [6] for  $\alpha$  and  $\beta$  dislocations. Our results indicate that this asymmetry is highly accentuated when an asymmetry in composition modulation is observed. In the low temperature grown samples studied in this work, plastic relaxation occurs mainly during the postgrowth thermal annealing, when the asymmetric composition modulation (if occurs) has already been developed. This fact suggests that the asymmetry in plastic relaxation may be related to the presence of composition modulation features.

#### Discussion

# Asymmetric Composition Modulation Features

By XTEM, we have observed an asymmetry in the wavelength of composition modulation contrasts for both  $\langle 110 \rangle$  directions in the growth plane of the 300 nm thick samples. This asymmetry was reported by Mirecki et al. [7] in AlAs/InAs heterostructures and it was explained as a result of a difference in the adatom diffusion length related to (2×4) surface reconstruction. However, Mahajan [8] argued that the wavelength of coarse contrast modulation is too high to be formed at the surface in the time available between deposition of two consecutive monolayers.

McDevitt et al. [9] tried to bear out Mahajan's hypothesis [8] about the coupling between fine speckle contrasts and coarse modulations. Annealing experiments were carried out in samples with both types of features, and it was observed that they disappeared simultaneously. If coarse contrasts were true composition modulations, they were expected to take longer to disappear, because of the longer distance that atoms had to diffuse. Thus, they proposed that coarse contrasts are a thickness-dependent artefact of thin foils that appear to accommodate the stresses due to the fine speckle modulations. This argument was also corroborated by the work of Choi et al. [10]. Fine speckle contrasts were observed in the whole TEM thin foil of an InGaAs/InP sample, whereas coarse contrasts just appear in some areas, depending on the foil thickness. Therefore, the authors consider coarse contrasts as an artefact of TEM thin foils.

Thus, in this work we are going to consider coarse contrasts modulations as an artefact, and not as a true phase separation. In this way, the studied samples would still be asymmetric, given that only the [110] direction would be modulated, and the perpendicular section would remain as a random alloy. This new type of asymmetry has been previously found in the studied system (InGaAs layers) by Okada et al. [3], and also in (InAs)<sub>n</sub>/(GaAs)<sub>m</sub> superlattices [11], therefore the assumption taken into account may be appropriate.

## Strain Hardening by Composition Modulation

Our results have shown that the existence of composition modulation could affect the plastic relaxation of the studied structures. The strain hardening of an alloy due to spinodal decomposition have been proposed theoretically [12] as well as experimentally [13]. Composition modulation carries the appearance of alternating fringes in the material with different Indium content and, therefore, different reticular parameter. The atomic planes between these fringes should be stressed due to the reticular mismatch. The existence of a considerable amount of stressed planes in the material could make it difficult for the dislocations to glide through it, causing the hardening of the alloy. This internal stress is strongly dependent on the wavelength of the composition modulation [12] and only short spacing modulations produce an appreciable effect in the retarding force on dislocation glide. Thus, independently of the exact origin of the coarse modulation, we can only consider the strainhardening behaviour of the fine speckle contrast.

Cahn [12] demonstrated that only in the case that the glide plane or the Burgers vector of a dislocation is parallel or perpendicular to the wave vector of the composition modulation k, the internal stresses due to this phase separation do not affect that dislocation, and they will not induce hardening. According to this model, only the dislocations that glide in a plane which contents the wave vector of the modulations would not suffer the internal stresses due to it. In our 300 nm thick samples, the wave vector of the modulations is parallel to [110] direction. Dislocations observed in Fig. 1b glide in {111} planes which contain this vector k, and therefore they would not suffer the stresses of the composition modulation, and a considerable plastic relaxation degree in this direction would be expected. However, dislocations parallel to the [110] direction glide in planes which does not contain the wave vector  $[1\overline{1}0]$ . In this case, the internal stresses due to the phase separation difficult the dislocation formation by glide, decreasing the density of these defects in that direction, in agreement with the experimental results obtained in the present work. This result implies that the modulation composition formation has to be taken into account in the theoretical models to predict plastic relaxation in ternary and quaternary semiconductor alloys.

#### Conclusions

Composition modulation and plastic relaxation in low temperature grown  $In_{0.2}Ga_{0.8}As/GaAs(001)$  epilayers have been studied. Our results show the presence of composition modulation with an asymmetric wavelength in  $\langle 110 \rangle$  directions for 300 nm thick samples.

Coarse contrasts modulations are considered an artefact of TEM thin foils due to the accommodation of the stresses of fine speckle modulations. Moreover, an asymmetry in plastic relaxation is found, which is considered to be enhanced as a consequence of the asymmetric phase separation: composition modulation difficults the glide of dislocations, causing the strain hardening of the alloy.

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