Depth dependent lattice disorder and strain in Mn-implanted and post-annealed InAs thin films

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A B S T R A C T

The lattice order degree and the strain in as-grown, Mn-implanted and post-implanted annealed InAs thin films were investigated with depth resolution by means of Rutherford backscattering spectrometry in channeling conditions (RBS/C). Three main crystallographic axes were analyzed for both In and As sublattices. The behaviour of the induced defects was evaluated in two regions with different native defects: the interface and the surface. The results show that Mn implantation and post-implantation annealing are anisotropic processes, affecting in a different way the In and As sublattices. The mechanisms influencing the enhancement and deterioration of the crystal quality during the implantation are discussed in relation to the as-grown defects and the segregation of the elements.

Keywords:

III–V Semiconductor

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RBS channelling

lattice order degree and strain

1. Introduction

Implantation techniques have been traditionally used to modify the physical properties of semiconducting materials, such as the free carrier concentration [1] or the n- or p-type semiconducting behaviour [2,3]. More recently, ion implantation has been used to induce site-selective growth of semiconducting quantum dots for strong three dimensional carrier confinement [4–6] and magnetism in the so called diluted magnetic semiconductors (DMS) [7–9]. In particular, concerning the DMS, ion implantation has been used to introduce, in a homogeneous and controllable way, magnetic dopants. This approach can overcome the low solubility of magnetic ions into III/V semiconductors, avoiding the formation of phase separation above a certain doping [10].

Nevertheless, the ion implantation originates lattice damage and creates a great variety of mobile and stable defects, known as implantation-induced defects. If implantation-induced defects such as Frenkel pairs (interstitials and vacants) are mobile at the implantation temperature, then a significant annihilation of the damage can occur during the implantation because of the dynamic annealing processes [11,12]. Moreover, additional and selective extermination of the implantation-induced defects can be achieved by annealing the specimens at a desired temperature after implantation [13]. The mechanism of the formation and annihilation of

defects in implanted semiconductors notably influences their physical properties and, therefore, it is of great scientific and technological interest [14–17].

Normally, the effect of the implantation-induced defects is convoluted with additional as-grown defects. In particular, a high density of misfit dislocations is observed in several of the heterojunctions of pristine III–V semiconductors because of the large lattice mismatch commonly presents there, especially for InAs/GaAs [18]. These native defects can play an important role for the reconstruction of the lattice damage after the implantation (in the thermal annealing processes). The characterization of the lattice disorder induced by the misfit dislocation at the InAs/GaAs interface has been already carried out by Cheng et al. by transmission electron microscopy (TEM) [19] and by Williams et al. using Rutherford backscattering spectrometry in channeling geometry (RBS/C) [20].

Actually, RBS/C has been revealed as a precise and useful tool for the study of crystal quality and strain of epitaxial layers in heterojunctions or superlattices [21,22]. The main advantage of RBS/C versus other traditional techniques such as X-ray diffraction (XRD) lies in its capability to allow simultaneously an elemental analysis of the crystal order and strain with depth resolution. The crystal quality and lattice order of the epitaxial layers is described by the ratio of yields (number of backscattered atoms) measured in aligned and in random configuration, also called normalized yield (χ). Thus, a low χ is a sign of good epitaxial quality (high order degree or crystalline feature), whether a high χ would indicate a low epitaxial quality and/or lattice disorder.

RBS/C is also a sensitive probe for strain, which can be measured by means of the angular displacement (known as kink angle) between the dips of the layer and the substrate along an oblique axis [23,24]. For cubic lattices, this measurement is usually done in the $\langle 110 \rangle$ direction [25]. The kink angle can be related with the tetragonal distortion of the film (ϵ_T) using the geometrical relations between the axis angle and the lattice parameters, studying it for different elements and depths [26].

The aim of this paper is to study the depth dependent lattice disorder and strain (for both In and As sublattices) in InAs thin films grown on (100) GaAs substrates after Mn-implantation and rapid thermal annealing processes. With this goal, RBS spectra were measured in channeling configuration along the $\langle 100 \rangle$, $\langle 110 \rangle$ and $\langle 111 \rangle$ crystal axes. Considering the high density of dislocations typically present at the InAs/GaAs heterojunctions, two regions were selected to carry out the analysis: one close to the (InAs/GaAs) interface and the other one near to the InAs surface. The influence of the as-grown defects on the lattice disorder and its dependence on the crystal orientation, as well as its relation with implantation-induced defects is investigated.

2. Experimental

InAs layers with a thickness of 250 nm were grown by atomic layer molecular beam epitaxy (AL-MBE) [27] on commercial GaAs(100) substrates. The substrate temperature was $T_s = 440^\circ\text{C}$, and the growth rate 0.5 monolayers per second (ML/s), using an arsenic beam equivalent pressure BEP (As₄) of 2×10^6 Torr. Previous to the growth of InAs, a 200 nm thick GaAs buffer layer was grown at $T_s = 580^\circ\text{C}$ and 1 ML/s by MBE.

The samples were implanted with Mn ions at a fluence of $1 \times 10^{16} \text{ cm}^{-2}$. The implantations were performed at 190 keV under room temperature conditions, and tilting the sample from the normal direction to avoid possible effects of channeling. The projected range of the implanted ions was calculated from TRIM code [28], showing a Gaussian-like shape with the center located at 125 nm depth (half of the thickness of the layer). The maximum number of target atom displacements per ion is calculated to be 3.7. One of the samples was rapid thermal annealed (RTA) after the implantation in Ar atmosphere for 30 s at a temperature of 750°C . During the annealing, the sample surface was covered with an InAs/GaAs sample to prevent As evaporation.

RBS/C experiments along the $\langle 100 \rangle$, $\langle 110 \rangle$ and $\langle 111 \rangle$ crystallographic axes were carried out in as-grown, implanted and post-annealed (from now on Mn-RTA) layers. The measurements were performed using 3 MeV $^4\text{He}^+$ beam (spot of $1 \times 1 \text{ mm}^2$) at the Centre of Micro-Analysis of Materials (CMAM) [29,30]. For the measurements, the samples were mounted on a three axis goniometer with an accuracy of 0.01° . The backscattered ions were detected by two standard Si-barrier detectors located at angles of 165° and 170° in IBM geometry, avoiding possible blocking effects. In all the cases, the random spectra were obtained by rotating the sample to average over azimuthal crystallographic orientations. Some special precautions were taken in order to achieve reliable and reproducible dose measurements: a bias voltage of +180 V was applied to the sample holder to avoid a possible contribution of the secondary electrons to the current integration, and the dead-time correction was applied. More detailed information about dead-time corrections is reported in reference [31].

3. Results and discussion

3.1. Elemental composition

Measured random spectra of as-grown, Mn-implanted and Mn-RTA InAs samples are shown in Fig. 1a. The elemental areal density

of InAs layers, as well as its homogeneity along depth, was deduced for each sample by fitting the random spectra with the simulation code SIMNRA [32]. The data reveal that the as-grown film is stoichiometric (50 at.% In and 50 at.% As) with a very homogeneous composition along the whole sample thickness. After Mn implantation, RBS data evidence a slight variation in the spectrum due to the incorporation of Mn. Despite the low cross section of Mn when compared with In or As, and the overlapping signals of the elements, the fitting of the data evidence that ~ 4 at.% of Mn is incorporated into the InAs film. The half width of the Mn distribution has been estimated to be ~ 25 . Interestingly, after the RTA treatment of the samples, Mn distribution becomes not uniform. Fig. 1b shows the simulations of the Mn-RTA spectrum using two models; one assumes a homogeneous Mn distribution within the InAs film and with the other assumes that Mn is only present in a small region close to the InAs surface. For the sake of clarity a simulated spectrum without Mn is also depicted. A good fit between experimental and calculated RBS data is only achieved by assuming that Mn implanted atoms are distributed within a narrow near-surface region, corroborating that the RTA treatment results in a non-homogeneous film. Such behaviour has been also observed for rapid thermal annealed GaAs samples implanted with Be, Mg or Zn [33].

3.2. Lattice disorder and strain

The lattice order degree was characterised prior to and after the different processes (implantation and RTA) by means of RBS/C. A deep analysis of the depth dependent lattice disorder from channeling measurements requires separating the contribution to the backscattering yield from In and As atoms (referring to the In and As sublattices) and selecting two energy windows for studying each sublattice: one located near to the InAs/GaAs interface and therefore, containing a high density of as-grown defects (dislocations), and the other one close to the InAs surface, with a much lower density of as-grown defects. Because of this reason five windows were selected in the RBS spectra corresponding to In (w1 for surface and w2 for interface), As (w3 for surface and w4 for interface) and GaAs substrate (w5). The RBS/C spectra of the different samples composing this study are shown in Fig. 2, together with the selected windows. The main features of each particular sample are discussed separately on the following.

3.3. As-grown InAs film

In general, large differences in the normalized yield are measured between RBS spectra taken in random and aligned configuration along all three investigated axes for the as-grown sample. In all cases, a high χ ($\sim 55\%$) is observed at the InAs/GaAs interface. As mentioned before, this is due to the presence of a high density of 90° and 60° misfit dislocations originated to release the stress derived from the 7% lattice mismatch between epilayer and substrate [19]. The χ value notably decreases with rising film thickness, reaching a minimum value $\chi_{\min}$ of $\sim 34\%$ (for w1 and w3 in the $\langle 100 \rangle$ axis). This gradual drop of χ is due to an improvement of the lattice order degree in the film. These data are in very good agreement with earlier observations with TEM carried out for similar films which evidence a reduction of the dislocation density of several orders of magnitude between the heterojunction and the bulk film [19]. Nevertheless, it is worthwhile to mention that even when the lattice order degree gets better with increasing thickness, the high $\chi_{\min}$ observed at the InAs surface as compared to that expected for a standard single crystal (typically $\chi_{\min} = 2\text{--}3\%$ [19]) indicates that the crystalline quality of the surface layer of a 250 nm InAs film is still affected by the defects generated at the heterojunction, being not fully free of disorder and/or strain. This

Fig. 1. (a) RBS spectra measured in random configuration for as-grown (black line), Mn-implanted (red line) and Mn-RTA (green line) InAs films. (b) Measured (points) and simulated (lines) RBS spectra for a Mn-RTA InAs film. For the simulation two different distributions of the Mn atoms are assumed: (i) Mn atoms homogeneously disseminated along the whole InAs layer (red line) (ii) Mn atoms distributed within a near-surface region (green line). For clarity sake a simulated RBS spectra without (WO) Mn is also presented (black line). (For interpretation of the colour in this figure legend, the reader is referred to the web version of this paper.)

result agrees quite well with that previously reported by Williams et al. which shows that an excellent crystalline quality is only achieved after the first 500 nm of similar InAs film [20].

The evaluation of windows w2 and w4 shows a different χ value along the $\langle 100 \rangle$ axis for the As ($\chi = 68\%$) and In ($\chi = 57\%$) sublattices, illustrating that, in the region close to the heterojunction, the As sublattice is notably more affected by dislocations and/or defects. However, in the shallower region (w1 and w3), both As and In sublattices present a similar crystalline quality. These features are also visible in the $\langle 110 \rangle$ axis, although a different situation is observed along the $\langle 111 \rangle$ axis, where $\chi(\text{In})$ is slightly higher than $\chi(\text{As})$ in both depth regions. This anisotropy in the normalized yield is normally ascribed to a different concentration of defects parallel to the $\langle 100 \rangle$ axis than perpendicular to it [19].

As discussed previously, the strain in each In and As sublattices was calculated from the $\langle 110 \rangle$ angular scans in the established re-

gions, using the GaAs substrate (w5) as a reference. Fig. 3a shows the angular scan for the shallower region, while Fig. 3d shows the dip close to the interface. In both cases, there is no significant shift (within the experimental error) with respect to the GaAs substrate ($\theta = 45^\circ$). Consequently, for the as-grown sample, no clear signs of strain are visible by RBS/C for both As and In sublattices.

3.4. Mn-implanted InAs films (Mn-implanted)

The effect of the Mn implantation on the generation of defects was also evaluated by RBS/C along the three main axes discussed before. Interestingly, the defects induced by the ion implantation affect In and As sublattices in different ways depending on the orientation. Fig. 2a shows that, along the $\langle 100 \rangle$, minor changes are produced with respect to the as-grown sample. On the contrary, Fig. 2b and c show that significant differences take place along

Fig. 2. RBS/C spectra along the $\langle 100 \rangle$, $\langle 110 \rangle$, and $\langle 111 \rangle$ crystallographic axes for as-grown, Mn-implanted, and Mn-RTA InAs films. For comparison the random spectrum for the as-grown film is also shown in all figures.

the $\langle 110 \rangle$ and $\langle 111 \rangle$ axes, respectively. In particular, both In and As sublattices show a clear decrease of the normalized yield at the interface along these $\langle 110 \rangle$ and $\langle 111 \rangle$ axes, what evidences certain reconstruction of the crystal lattice. A different situation is ob-

served at the surface region, where $\chi(\text{As})$ and $\chi(\text{In})$ increases with respect to the as-grown sample (especially in the $\langle 110 \rangle$ direction, Fig. 3b and e).

These results indicate that the original lattice disorder at the interface is mitigated by the Mn implantation, although the Mn-implantation produces a completely different effect in the surface region, where the disorder increases. Since this behaviour only appears in the $\langle 110 \rangle$ and $\langle 111 \rangle$ axes, it should be mainly related with the increase (for the surface) or decrease (for the interface) of the defect density parallel to the growth direction.

Regarding the behaviour of In and As sublattices, there are no significant differences in the normalized yield along the $\langle 100 \rangle$ axis. The $\langle 110 \rangle$ angular scans (Fig. 3b and e) also show a similar behaviour in the implantation induced damage for both sublattices, with only slight differences regarding the variations in the minimum yield. Actually, SRIM calculations show a similar number of displacements per atom (dpa) ~ 1.2 for In and As atoms when implanting Mn under these conditions. Nevertheless, the previously reported PIXE/C data on similar samples have shown that most of the Mn atoms occupy substitutional positions into the In site [9], which could explain some of the features observed in the dips (e.g., the appearance of strain as discussed below).

The different effect of the ion implantation on the lattice order for the interface and the surface might be related with some kind of radiation-induced damage recovery processes taking place

Fig. 3. Angular scans along the $\langle 110 \rangle$ axis for the In and for the As sublattices for the as-grown, Mn-implanted, and Mn-RTA InAs films. (a–c) Represent the integration for the windows related to the surface region (w_1 and w_3 for In and As, respectively), while figures (d–f) represent the windows close to the InAs/GaAs interface (w_2 and w_4).

during irradiation [11]. The selective enhancement at the interface suggests that this recovery is somehow linked to the presence of native defects in this region (i.e., dislocations). To deepen in this issue, angular scans along the $\langle 110 \rangle$ axis were analyzed (Fig. 3). Concerning the surface (Fig. 3b), a clear shift of $\Delta\theta = -0.10(2)^\circ$ is detected with respect to the position of the relaxed GaAs substrate. This shift is the same for both In and As sublattices, which indicates that Mn-implantation induces a similar strain in both sublattices. The tetragonal distortion associated with this shift is $\varepsilon_T \sim 0.4\%$ and, therefore, the Mn-implantation induces a significant strain at the surface of the film. The accommodation of the Mn atoms in this region, therefore, introduces an additional deformation of the InAs lattice (normally relaxed after the dislocations at the interface) and explains the rising in the minimum yield.

Only a slight shift of $\Delta\theta = -0.04(2)^\circ$ is visible for the interface of the InAs layer, which indicates an almost zero tetragonal distortion in this region. Since the InAs/GaAs interface already contains a large density of dislocations, the presence of Mn atoms in this region does not modify significantly the strain state. On the contrary, such Mn atoms might induce atomic mixing, what could be a plausible reason for the observed enhancement of the $\chi_{\min}$. The exact mechanisms producing such effect, however, remain unclear and would require a more systematic study.

3.5. Mn-implanted and post-implanted annealed films (Mn-RTA)

Typical annealing treatments aim to reduce the generation of defects during the ion implantation, but can induce other physical phenomena such as interdiffusion or intermixing affecting in a different way As and In atoms. In the current study, the thermal treatment was carried out at 750°C . The spectra measured for the annealed samples are depicted in Fig. 2. In this figure it is observed that after the RTA the order degree of In atoms is very similar to that of the as-grown sample along all three crystallographic axes, what indicates a good recovery of the crystal quality in this sublattice. Interestingly, some clear differences are observed in $\chi(\text{As})$ when comparing the RBS/C spectra of Mn-RTA and as-grown samples. In particular, along the $\langle 100 \rangle$ axis, the $\chi(\text{As})$ for the Mn-RTA film is smaller at the interface region and higher at the surface than those for the as-grown sample. This fact indicates that RTA increases the As sublattice order degree at the interface region and it significantly deteriorates it in the surface region.

As mentioned before, the Mn incorporated into the film migrates towards the sample surface during the RTA treatment (Fig. 1). Since Mn in as-implanted sample enhanced the crystal ordering at the interface, the out-diffusion of Mn (and perhaps of As too) during the RTA, is one possible mechanism associated with the detriment of the crystal quality in the As sublattice at surface. These processes would also explain the negative effect of the RTA at the interface and, furthermore, the absence of strain in the $\langle 110 \rangle$ angular scans (Fig. 3c and f). Actually, it is important to mention that $\chi(\text{GaAs})$ is higher for the Mn-RTA film than for the other two samples. This effect could be due to a higher dechanneling in the InAs layer as a consequence of the formation of non-crystalline phases during the Mn migration to the surface [8].

4. Conclusions

The elemental composition, lattice order degree and strain for as-grown, Mn-implanted and Mn-RTA 250 nm InAs films grown by MBE were investigated. The as-grown film was found to be stoichiometric. After implantation, the Mn incorporated in the film was $\sim 4\text{ at.}\%$, and the RTA promotes Mn atoms to diffuse towards the film surface, generating an inhomogeneous elemental distribution.

For the as-grown film, the In and As sublattice order degree in the surface region has been observed to be significantly better than in the interface region along all three investigated axis. At the interface, the disorder in the As sublattice has been found to be larger than in the In. No signs of strain were detected for any sublattice in this sample.

After the Mn implantation, the In and As lattice order degree improves along the $\langle 110 \rangle$ and $\langle 111 \rangle$ axis at the interface but deteriorates at the surface. These effects were correlated with the generation of strain for both sublattices at the surface, and with the possible reconstruction of the crystal lattice via Mn recombination at the interface. The absence of significant changes in the $\langle 100 \rangle$ direction reveals the anisotropy of the effects induced by Mn-implantation.

The RTA treatment after the implantation leads to an order degree very similar to that observed for the as-grown sample for the In sublattice. However, the As sublattice shows a different behavior with the annealing, improving the lattice order at the interface and deteriorating it at the surface. This effect cannot be ascribed to strain but to other diffusion effects already observed in the Mn (and suggested for As).

In general Mn-implantation and post-implantation annealing have been found to be anisotropic processes, affecting in a different way the In and As sublattices.

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