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## X-ray reciprocal space mapping of coincided As-clusters/GaAs and $\delta$ -InAs/GaAs superlattices grown at low temperature

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**Abstract.** X-ray reciprocal space mapping was employed to assess the crystalline quality of the  $\delta$ -InAs/GaAs superlattices grown at low temperatures and structural transformations related to formation of As-cluster/GaAs superlattices upon post-growth anneal.

### Introduction

During the last few years InAs/GaAs superlattices with thin InAs layers have attracted much attention due to interesting electronic and optical properties. While perfect GaAs films are normally produced by molecular-beam epitaxy (MBE) at  $\sim 600^\circ\text{C}$ , the InAs/GaAs superlattices are grown at a reduced temperature (normally  $400\text{--}500^\circ\text{C}$ ) in order to eliminate indium segregation and produce abrupt interfaces. A decrease in the growth temperature leads to an increase of point defect concentration. When the temperature is extremely low ( $\sim 200^\circ\text{C}$ ), a high arsenic excess is incorporated in the growing film and the concentration of arsenic antisite defects is as high as  $10^{20}\text{ cm}^{-3}$  [1]. A post-growth anneal of the GaAs films grown at low temperature (LT) results in precipitation of the excess arsenic. The annealed LT GaAs exhibits a very high electrical resistivity and extremely short carrier lifetime [2]. This material is promising for application in ultra-fast devices.

The system of nano-scale arsenic clusters in the annealed LT GaAs films is random. It has been shown recently that InAs  $\delta$ -layers can be used to form two-dimensional cluster sheets in the GaAs matrix [3] and superlattices of these sheets coincided with the initial  $\delta$ -InAs/GaAs superlattices [4]. In this paper we employed x-ray reciprocal space mapping to assess the crystalline quality of both the initial  $\delta$ -InAs/GaAs superlattices grown at low temperatures and As-cluster/GaAs superlattices formed by the post-growth anneal.

### 1 Experimental

The  $\delta$ -InAs/GaAs superlattices were grown by MBE at  $150$  and  $200^\circ\text{C}$ . The nominal thickness of the InAs  $\delta$ -layers was  $\sim 1$  monolayer. The thickness of the GaAs spacers was  $30$  and  $50$  nm. All the samples were cut into several parts, one of them was kept as-grown, the other were annealed at  $400$ ,  $500$ , or  $600^\circ\text{C}$ .

X-ray diffraction (XRD) studies were carried out using high-resolution, double-crystal diffractometers. Studies of superstructural ordering and measurements of reciprocal space mapping (RSM) of diffracted intensity were made using a double-crystal  $\theta - \theta$  diffractometer with a twofold channel-cut Ge (001) monochromator crystal.  $\omega - 2\theta$  and  $\omega$ -scanning techniques for rocking curves (RCs) and the  $\theta - 2\theta(\omega)$  technique for RSM registration were used.  $\text{CuK}_{\alpha 1}$  radiation was used for the measurements. A narrow receiving slit in front of

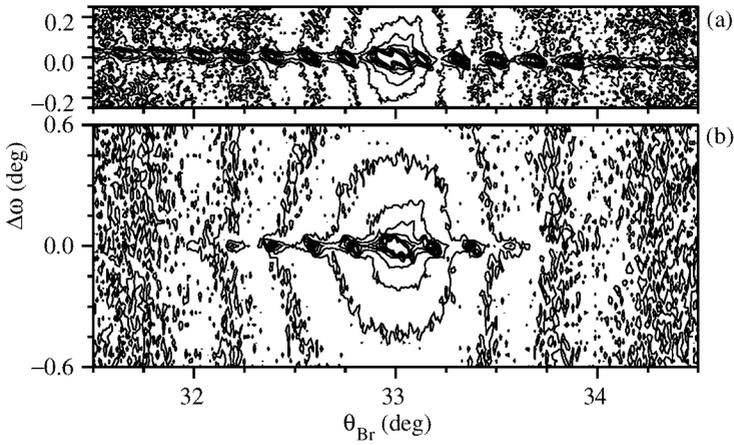
the detector enabled separation of the coherent and diffuse components of the diffracted x-ray beam.

## 2 Results and discussion

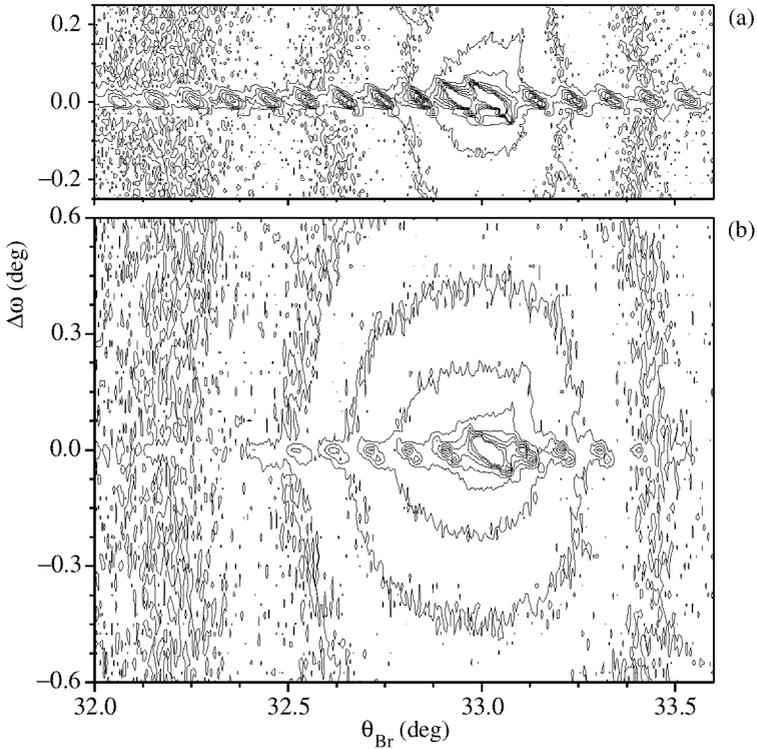
The structures investigated have high crystal lattice perfection, which was demonstrated by measurements at a high angular resolution (about 1.0 arcsec) in the vicinity of GaAs (004) reflection. The parameters of crystal perfection are determined from the extended interference patterns arising from structures under Bragg diffraction conditions. This means that the density of small structural defects in as grown samples is insignificant. Furthermore, coarse defects such as dislocations are entirely absent. The elimination of grown-in structural defects in these samples and the high degree of crystal lattice perfection enable structural investigations and modelling of samples containing real quantum-sized objects with extremely high accuracy. In the case of perfect structures, the main structural features observed in experiments must be correlated with the real structures of scattering objects, including quantum sized objects. Wide-angle XRD RCs have shown the existence of superstructural ordering along the direction of epitaxial growth for both investigated samples. The coherent approximation of the dynamical theory of XRD based on Takagi's equations [5] was used for the simulation of RCs. Fitting of RCs has shown that the period of multilayer structures is close to the value estimated from the growth conditions, and that the average thickness of the InAs layers is about 0.20–0.25 nm, which is close to the expected value. Evaluation of the roughness of the interfaces in the periodic parts of structures has been obtained from the entire angular range of coherent RCs. It was shown that the roughness of the InAs/GaAs interfaces of the as-grown samples is equivalent to about 3–4 monolayers and is typical for a GaAs(001) surface [6].

Figs. 1 and 2 show the RSM of diffracted intensity in the vicinity of GaAs(004) reflection for two samples grown at different temperatures. For as-grown samples one can see a large number of superlattice interference peaks and low diffuse scattering. This is clear evidence of high crystalline quality of these samples in spite of a very high concentration of excess-arsenic-related point defects ( $(1-2) \times 10^{20} \text{ cm}^{-3}$ ). The annealing at 600 °C leads to a significant diminishing of the interference patterns and strong increasing of diffuse scattering (part (b) in Figs. 1 and 2). The diffuse scattering is symmetrically distributed around GaAs(004) and zero-order superlattice (0SL) peaks. In as-grown samples the point defects do not influence the crystal perfection of as-grown samples, and the planarity and roughness of the InAs/GaAs interfaces. The scale of non-uniformity of deformation fields around the interfaces is comparable with the roughness of the interfaces. Formation of As clusters at the InAs  $\delta$ -layers upon annealing leads to increasing roughness of the interfaces. In addition, migration of native point defects causes enhanced In-Ga intermixing. These processes produce a significant distortion of the local strains around the  $\delta$ -layers and, as a result, lead to the intensive diffuse scattering in the vicinity of central diffraction peaks, and to diminishing extension of interference patterns.

A strong lattice relaxation was detected after annealing of the samples. The lattice parameter of the annealed material is slightly less than that of stoichiometric one. This relaxation was stronger for the sample grown at lower temperature. However, comparison of experimental data in Figs. 1 and 2 shows that the spatial distribution of the x-ray diffracted intensity is very similar for the samples grown at 150 and 200 °C. The decrease of the growth temperature by 50 °C results in the increase of the excess arsenic by the factor of two. However, higher supersaturation by excess As causes more intensive precipitation rate and the sheet cluster concentrations at InAs  $\delta$ -layers turn out to be approximately equal in



**Fig. 1.** X-ray reciprocal space mapping in the vicinity of GaAs(004) for the sample grown at 200 °C before (a) and after (b) annealing at 600 °C.



**Fig. 2.** X-ray reciprocal space mapping in the vicinity of GaAs(004) for the sample grown at 150 °C before (a) and after (b) annealing at 600 °C.

both samples. Analysis of reciprocal space patterns shows that formation of nano-scale As clusters does not produce extended defects such as dislocations and stacking faults.

### 3 Conclusions

Multilayer  $\delta$ -InAs/GaAs periodical structures grown by MBE at low temperature were studied by high resolution x-ray diffraction methods. It was shown that in spite of the initial high concentration of point defects in these structures they have high crystal lattice perfection and flat abrupt interfaces. A strong increase in the x-ray diffuse scattering was detected by reciprocal space mapping of the samples annealed at 500 and 600 °C. This effect is accompanied by remarkable decrease of the interference patterns. Both phenomena can be attributed to increasing roughness of the interfaces due to formation of As clusters superlattices coincided with the initial  $\delta$ -InAs/GaAs periodical structures. Formation of nano-scale clusters was found to produce no extended defects such as dislocations and stacking faults. The lattice parameter of the clusters contained material is slightly less than that of stoichiometric one.

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