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Defects in GaInAsBi Epitaxial Films on Si(001) Substrates

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Growth of a thin GaInAsBi film was carried out on a Si (001) substrate by pulsed laser deposition. The growth was carried out in the Volmer–Weber. The grains are preferentially monophase, but are separated by dislocation network, and in some areas, there are antiphase boundaries. Investigation of the real structure by transmission electron microscopy and X-ray diffractometry shows that stress relaxation occurred due to plastic shears by means of a nucleation of dislocations and a slip close-packed $\{111\}$ planes, as well as twinning and a change in surface roughness. Using X-ray diffractometry, it was found that the GaInAsBi film has a lattice parameter of 5.856 Å. The root-mean-square roughness of the film surface, measured by atomic force microscopy, was 0.51 nm.

Keywords: III−V compounds, highly mismatched alloys, pulsed laser deposition, GaInAsBi, silicon.

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1. Introduction

GaInAsBi thin films are of interest for a wide range of practical applications. Grown on InP and GaAs substrates, they can be used in mid-infrared devices [1], thermoelectric [2] and terahertz [3–5] devices, as well as in telecommunications systems [6]. The use of Si as a substrate for epitaxial growth has a number of advantages, such as high quality, large area and low cost compared to substrates based on III−V group compounds [7–9].

Many III−V compounds on silicon are characterized by growth in the Volmer–Weber mode, when at the initial stage of growth the film consists of islands, and after a certain critical thickness their coalescence occurs [9–14]. When growing a GaInAsBi solid solution on Si, two stress relaxation mechanisms will compete due to strong lattice mismatch. The first is the appearance of plastic deformations, which lead to biaxial compression or tension through the nucleation and sliding of dislocations. With the second mechanism of stress relaxation, exclusively elastic deformations occur, manifested in a change in the roughness of the film surface (for example, the growth of quantum dots). Therefore, knowledge of the preferential mechanisms of stress relaxation in highly mismatched heterostructures is not only of fundamental scientific interest, but is important from a practical point of view for the epitaxial growth of III−V compounds on Si [9]. The GaInAsBi-Si heterosystem is promising for a new class of materials —highly mismatched alloys (HMAs) [15].

At the moment, the methods of molecular-beam epitaxy (MBE) [16] and vapor-phase epitaxy of metalorganic compounds (MOVPE) [6] are widely used for growing solid solutions with bismuth. According to the literature data [1–6], the properties of GaInAsBi solid solutions depend on the method of their growth. The complexity of growth is determined by the presence of droplets on the growth surface [17,18], as well as by the fact that the change in the band gap and lattice parameter is carried out by two elements — In and Bi. Nevertheless, methods for growing bismuth-containing solid solutions are being developed.

For the first time, a GaInAsBi solid solution was grown by MBE on an InP substrate with a Bi content of up to $x = 2.5\%$ [16]. It is known about the growth of MBE films of GaInAsBi on GaAs substrates with a content of 19.5% In and 9.5% Bi and roughness ~ 1 nm for a layer with a thickness of 30 nm [5]. An important detail of this study is the use of a 30 nm thick GaInAs buffer. In the work [6], a pseudomorphic GaInAsBi layer was grown on a buffer layer 250 nm thick on a GaAs substrate using the MOVPE method. The thickness of GaInAsBi, determined by X-ray diffractometry (XRD), was 66.5 nm, and the lattice parameter was 5.7 Å. In all studies on the growth of GaInAsBi by MBE and MOVPE methods, there is a general tendency towards a decrease in the substrate temperature due to the surfactant effect Bi [19] and segregation In [20–22].

In the last decade, the method of pulsed laser deposition (PLD) has become increasingly used for growing thin films [23–27]. Its main advantages over other methods of physical deposition are the ability to control the stoichiometry of films [28], the opportunity of reducing the substrate temperature for growing thin films based on III−V compounds [29], discrete flow of material from target to the substrate in the time intervals between laser pulses. Due to the presence of droplets in the grown layer and the relatively small area of film deposition, it is too early to talk about the widespread industrial application of the method. Nevertheless, the method is suitable for the synthesis of many semiconductor materials. This study presents the results of a study of growing a GaInAsBi solid solution on a silicon substrate using the PLD method.

An analysis of the literature showed a small number of works devoted to the study of the structural properties and mechanisms of defect formation in GaInAsBi films, and in the case of growth on Si, their complete absence. Studying the initial stage of growth of GaInAsBi thin films is important from a practical point of view to achieve their heteroepitaxial growth. In this regard, the goal of the work was to grow a GaInAsBi film on a Si (001) substrate using the PLD method, to study defects in them and the mechanisms of layer relaxation.

2. Experiment procedure

The growth was carried out on an experimental equipment of PLD [23]. A $YAG:Nd^{3+}$ laser with a wavelength of 532 nm (second harmonic) was used as a target sputtering source. The target-to-substrate distance was 50 mm. The target was formed by cold pressing. For this purpose, GaAs, InAs and Bi powders were mixed in the required proportion. Then the mixture was sifted through a sieve with a mesh size of $20\,\mu$m. The target was formed using an isostatic press at a pressure of 207 MPa. Then the target was sintered in an atmosphere $H_2:N_2\,(1:1)$ at a temperature 850°C and a pressure $13.2 \cdot 10^4$ Pa for 2 h. For the experiments, an *p*-Si substrate with crystallographic orientation (001) and a thickness of $390\,\mu$m was used. At the first stage of preparation, the Si substrate was degreased in acetone and then in isopropyl alcohol. Then it was washed in deionized water to remove solvent residues and particles of dissolution products from the surface. At the second stage, chemical etching of the silicon surface was carried out in a 5% solution of hydrofluoric acid for 60 s and washing in deionized water, drying and loading into the growth chamber. The vacuum in the chamber was pumped out to a residual pressure of $\sim 2.3 \cdot 10^{-4}$ Pa. The substrate was heated to a temperature of 500°C and annealed for 15 minutes. Next, the substrate temperature was reduced to 350°C and the GaInAsBi film was grown at laser fluence $F = 2.3$ J/cm^2, pulse duration 10 ns, pulse repetition rate 15 Hz. The film deposition time was 60 min.

Structural defects in GaInAsBi thin films on Si were studied by XRD and transmission electron microscopy (TEM). The crystalline state and lattice parameter were determined on a Bruker D8 Discover instrument with $CuK\alpha_1$-radiation in the Bragg–Brentano $\theta - 2\theta$ geometry. The lattice parameter of the GaInAsBi layer in the growth direction was calculated from the diffraction maximum from the (004) plane on the XRD curve, in accordance with the Wulff–Bragg law: $2d\sin\theta = n\lambda$ (where d — interplanar spacing, λ — wavelength of characteristic X-ray radiation, n — maximum order ($n = \pm 1, \pm 2 \ldots$), θ — Bragg angle for a family of planes (hkl)). To study the bulk structure, we used the bright field mode, and to study local areas of the structure, microdiffraction mode and high resolution on a Jeol JEM-2100F microscope. The thickness of the GaInAsBi films was determined from cross sections using TEM.

The study of morphology and measurement of root-mean-square roughness was carried out using atomic-force microscopy (AFM) on an NT-MDT SI „Integra academia“ microscope. Scanning was carried out in semi-contact mode at atmospheric pressure. An NS15 silicon cantilever with a radius of curvature of 10 nm was used as a probe. Scanning area $1 \times 1\,\mu$m^2. AFM image filtering operations included subtraction of the 3rd surface, removal of steps in the X direction, and removal of scratches. The root-mean-square roughness parameter was determined based on the scan area (S_q).

3. Experimental results

3.1. Structural defects in the bulk of a GaInAsBi film

To determine the crystal lattice parameter and crystalline state, XRD measurements were performed (Figure 1).

The diffraction pattern (Figure 1) shows intense diffraction maxima from the (002) and (004) planes of the Si substrate at the angles $2\theta = 33.05°$ and $2\theta = 69.21°$, respectively. $2\theta = 63.49°$ angle corresponds to the main diffraction maximum from the (004) plane of the GaInAsBi layer (see inset in Figure 1). The appearance of the diffraction pattern indicates that the film has a predominant crystallographic orientation (001). The calculated lattice parameter of the GaInAsBi layer based on the (004) peak from the Wulff-Bragg expression was $a = 5.856$ Å.

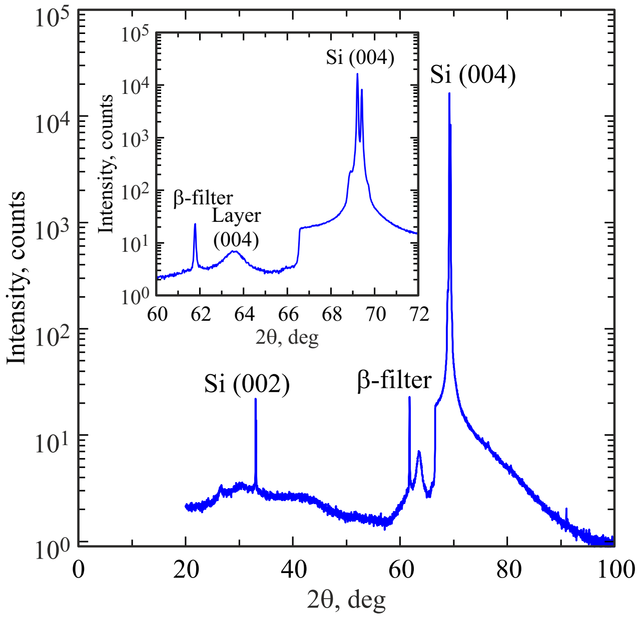

Figure 1. X-ray diffraction pattern of GaInAsBi on Si (001) for $2\theta = 20-100°$.

In this case, the mismatch value is $\Delta a/a_{\mathrm{Si}} = 7.84\%$. The calculated average size of the coherent scattering region according to the Scherrer model ~ 12.6 nm.

Figure 2, *a* shows a bright-field TEM image of a GaInAsBi/Si heterointerface. The thickness of the GaInAsBi layer reaches ~ 45 nm. Stacking faults (SF) are visible in the bulk of the film and grain boundaries. Some SF extends to the surface of the film. Determination of the crystalline state of the GaInAsBi film and indexing of crystallographic directions were carried out using a microelectron diffraction pattern (Figure 2, *b*).

There is presence of plastic deformations in the film volume in the form of linear reflections, as well as reflections from twins (Figure 2, *b*).

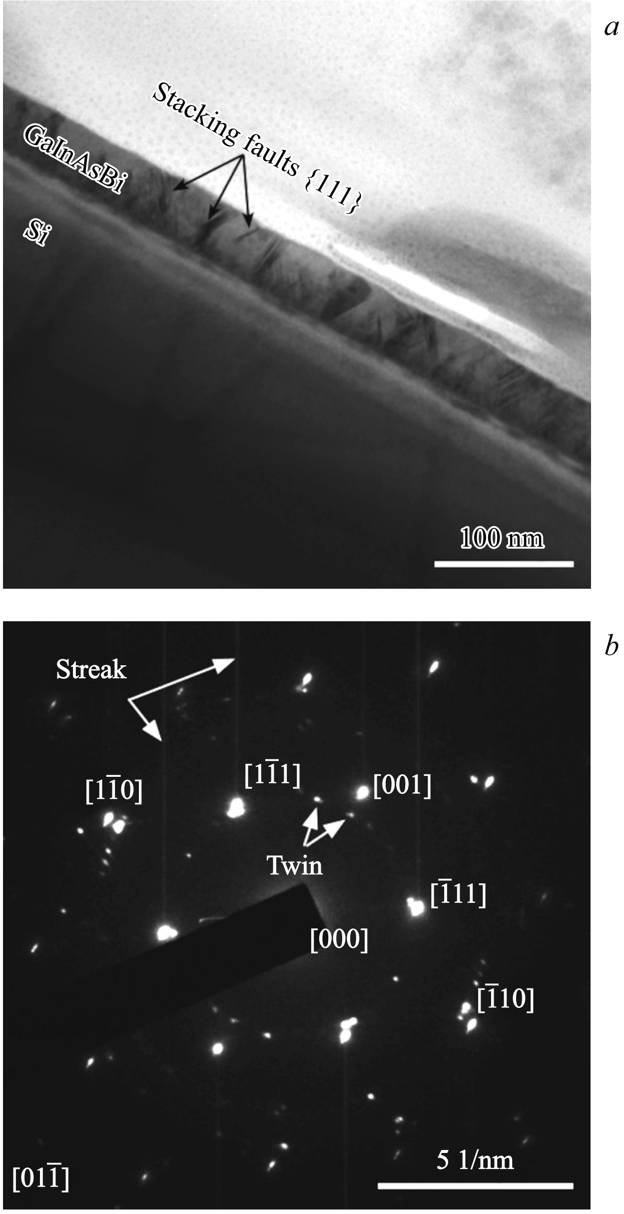

Figure 2. Bright-field TEM image (*a*) and microelectron diffraction pattern (*b*) of a GaInAsBi film on Si (001).

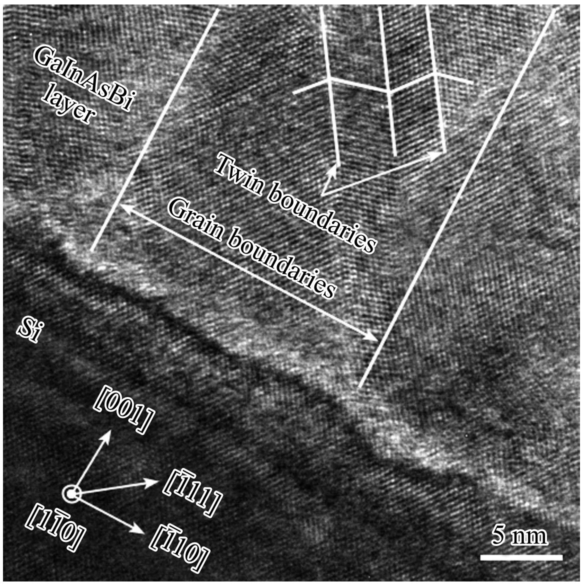

Figure 3. High resolution TEM image of a GaInAsBi/Si heterointerface.

Figure 3 shows a high-resolution TEM image of a cross section of a GaInAsBi/Si heterointerface with a transition amorphous layer of thickness ~ 3 nm. Grain boundaries are also visible, indicating that the film is textured [30,31]. Analysis of several sections of the heterointerface showed that the centers of nucleation of plastic deformations are located on the surface of the transition amorphous layer.

Another type of structural defects found in the GaInAsBi film are twins. Figure 3 shows a twin, which is also confirmed by the results on the microelectron diffraction pattern in the form of two diffraction maxima (Figure 2, *b*). The size of the twin in the observation plane is ~ 5 nm.

3.2. GaInAsBi film surface morphology

The AFM results of the surface of the silicon substrate and GaInAsBi film are shown in Figure 4. The surface of the silicon substrate (Figure 4, *a*) is represented by a smooth relief with a root-mean-square roughness parameter $S_q = 0.23$ nm. The growth surface is represented by terrace-like domains [31], which are limited on the sides by planes $\{110\}$.

The surface of the GaInAsBi film has a more rough morphology (Figure 4, *b*) than the Si substrate. Root-mean-square roughness parameter over scan area $S_q = 0.51$ nm. The dimensions of the islands are 10–70 nm. The film is a continuous layer that was formed as a result of multiple nucleation of islands and their subsequent coalescence.

4. Results and discussion

The presence of V-shaped SF (Figures 2, *a* and 3) in the volume of the film is explained by the fact that in solids

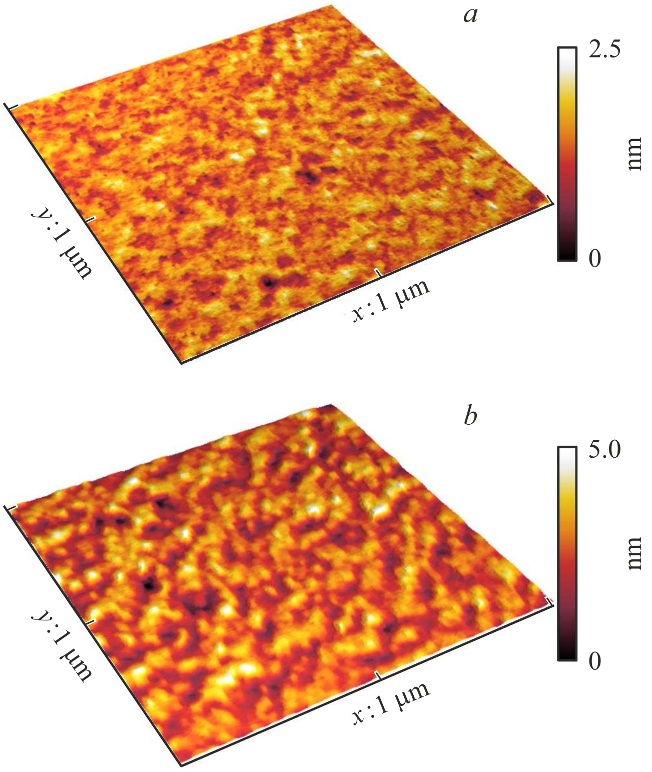

Figure 4. *a*) — $1 \times 1\,\mu\mathrm{m}^2$ 3D AFM surface Si (001); *b*) — $1 \times 1\,\mu\mathrm{m}^2$ 3D AFM surface of a GaInAsBi thin film. (The colored version of the figure is available on-line).

with a face-centered (FCC) lattice, with lattice mismatch over 1%, due to the formation of partial dislocations, sliding of the $\{111\}$ planes occurs. A SF can intersect with another one lying in a different plane $\{111\}$ and form V-shaped configurations. We believe that the reason for their appearance in GaInAsBi is due to the addition of bismuth to the film composition, which led to a strong lattice mismatch (7.84%) between the film and the Si substrate. The result of this was a stress relaxation process, which caused the formation of defects at the interface and in the bulk of the film. However, the process of nucleation of mismatch dislocations in heteroepitaxial semiconductor systems has not yet been fully studied. Among the various models proposed in the literature for dislocation nucleation, only two of them are most consistent with experimental observations in heteroepitaxial systems: 1) nucleation from surface steps; 2) agglomeration of point defects, which can be sources of gliding dislocations [32]. Due to the peculiarity of the growth process in our samples, at the „film-substrate“ interface, there is an amorphous layer with a thickness of I ~ 3 nm. It can be seen that on the surface of this layer, nucleation of SF occurs along the $\{111\}$ planes due to the appearance of point defects. It should be noted that, due to the existence of a transition amorphous layer in the film, no growth of dislocations from the substrate into the bulk of the GaInAsBi layer and back was detected, as was observed in the works [7,33]. In turn, point defects in FCC lattices lead to the formation of two types of partial dislocations:[34–36]: 1) partial Shockley dislocations associated with slip, which have a Burgers vector $a/6\{112\}$ (30° Shockley dislocations); 2) partial Frank dislocations, including interstitial or vacancy planes, with the Burgers vector $a/3\{111\}$ (60° dislocation).

The cause of twinning is shift due to the passage of a partial Shockley dislocation with Burgers vector $\vec{b} = 1/6[112]$ through each plane $(\bar{1}11)$ above the twinning plane. In FCC crystals there are four close-packed planes $\{111\}$, each of which contains three $\langle 112 \rangle$ directions. Thus, there are twelve independent twinning systems. For the cross section $(1\bar{1}0)$ in Figure 3, as a result of re-indexing, the plane $(\bar{1}11)_M$ of the film is transformed into the plane $(1\bar{1}\bar{1})_T$ of the twin [37,38]. The observed twins cannot be caused solely by deformation of the crystal lattice. The twinning mechanism depends not only on the crystal lattice, but also on the growth mechanism and the stage of film formation [11].

Films of III–V compounds on silicon are characterized by growth according to the Volmer–Weber mechanism, when three-dimensional nucleation of islands occurs at the initial interval of growth, and after a critical thickness and surface density coalescence occurs [9,11–14]. In this case, the film is textured [30,31] due to the presence of grain boundaries, and 3D nucleation predominates over 2D nucleation on the growth surface. As a result, plastic relaxation of internal stresses leads to changes on the growth surface, expressed in changes in surface roughness. The terraces on the tops of the islands (Figure 4, *b*) indicate that the grains in the film are predominantly oriented in the [001] growth direction; this is also confirmed by XRD results (Figure 1). Most of the islands are monophasic, but are separated by a network of dislocations, and in some areas there are antiphase boundaries. The high mismatch (Figure 1) of lattice parameters, due to the high Bi concentration, did not lead to the formation of extended mismatch dislocations on the film surface, which are characteristic in the case of pseudomorphic or layer-by-layer growth [31,39]. Stress relaxation in the film and the roughness on its GaInAsBi surface are determined by the fact that almost complete stress relaxation occurs at grain boundaries during their coalescence, and stresses increase inside [10]. For stress relaxation on the growth surface, surface diffusion must be maintained to move atoms to places with the lowest free energy, i. e. to the tops of the islands. This process explains the persistence of island growth when growing a GaInAsBi film on Si. Therefore, in a GaInAsBi layer with a thickness of ≈ 45 nm, plastic relaxation occurs through partial Shockley dislocations, Frank dislocations, as well as twins (Figure 3) and antiphase boundaries, which appear in the surface relief (Figure 4, *b*).

The XRD, TEM, and AFM results are in good agreement and show that GaInAsBi films grown by pulsed laser deposition have a pronounced texture in the (001) direction and are completely relaxed.

5. Conclusion

Thin films of GaInAsBi were grown on a Si (001) substrate by the PLS method and the structural properties and morphology were studied, and the mechanism of stress relaxation was established. The results of XRD, TEM and AFM allow us to conclude that the GaInAsBi film was grown in the Volmer–Weber mode and has a pronounced texture in the growth direction [001]. The study of relaxation processes and types of defects allows us to conclude that in a highly mismatched $(\Delta a/a_{\mathrm{Si}} = 7.84\%)$ GaInAsBi-Si heterosystem, stress relaxation occurred due to plastic shears, through the nucleation of dislocations and sliding along close-packed planes $\{111\}$, as well as through twinning and changes in surface roughness. Meanwhile, significant stresses remain inside the grain. The grown layers have a lattice parameter $a = 5.856\,\mathrm{\AA}$ and a root-mean-square roughness of 0.51 nm. The results indicate the potential of the pulsed laser deposition method for the epitaxial growth of multicomponent semiconductor solid solutions, including on silicon substrates.

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Conflict of interest

The authors declare that they have no conflict of interest.

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