Structural and morphological features of ultrathin epitaxial InSb films in AlAs matrix

D A Kolotovkina1,2, A K Gutakovskii1,2

1A. V. Rzanov Institute of Semiconductor Physics, SB RAS, Novosibirsk, Russia
2Novosibirsk National Research State University, Novosibirsk, Russia

E-mail: koldarya@yandex.ru

Abstract. This work presents results of the investigation of structural and morphological features of epitaxial InSb layers in the AlAs matrix. Our research group used transmission electron microscopy (TEM). The specimens were grown by molecular beam epitaxy and prepared in the cross section (110) and plan view foils (100). We found a formation of the embedded epitaxial layer of solid solution InxAl1-xSb1-yAs in the AlAs matrix during precipitation of In and Sb on the AlAs surface. The embedded layer had continuous area (wetting layer) and islands. The study revealed two types of islands in the epitaxial layer the first having coherent interfacing with the matrix lattice and the second a relaxed island. We estimated concentration of In, Sb in the solid solution by the indirect method. We used the method of geometric phase to analyze the distribution of misfit dislocation cores on the interface. Every misfit dislocation was formed by two close 60°-dislocations with the Burgers vectors like \( \frac{a}{2} <110> \). The sum Burgers vector of the dislocation pair was in the plane of the interface.

1. Introduction

Today the system AlAs-InSb-AlAs intrigues scientists because of the interesting features of its energy spectrum. Quantum dots (QD) of indium antimonide formed in the aluminum arsenide matrix are of the greatest interest, a structure of QD energy spectrum suppresses photon and phonon scattering of electrons. Theoretically it can provide a long life time (up to a few milliseconds) of exciton spin polarization (excitons localized in QD) [1]. In this case theory allows to predict an indirect QD energy-band structure, namely, the energy-band structure of the first type with the main electron state belonging to X-minimum of conduction band. It leads to the increased life time of the exciton up to few milliseconds that gives a chance to study the spin relaxation of exciton.

The theoretical calculations [1] point to energy spectrum of the first type of the system InSb/AlAs even if mixing of the materials of QD and matrix takes place during the process of growth. Thus, the InSb/AlAs system allows to form QD having different sizes and composition, so it gives an opportunity to study parameters of the exciton spin relaxation with various values of exchange interaction between an electron and the hole.

Structural data of the system are necessary for a correct interpretation of future experimental results. Unfortunately, at present any data about formation and structure of the InSb QD in the wide-band-gap AlAs matrix are absent in literature. A goal of the work is to study structural features and...
morphology of epitaxial indium antimonide layers in the wide-band-gap aluminum arsenide matrix by transmission electron microscopy (TEM).

2. Objects of investigation
The object of the investigation was GaAs-AlAs-InSb-AlAs-GaAs (001) heterosystem, grown by molecular beam epitaxy (MBE) on “RIBER compact 21T” (Figure 1). For electron microscopy (EM) investigations we chose six samples, which had different MBE modes (atom layer epitaxy or ALE and simultaneous deposition or standard MBE) and conditions (an order of the alternation, a number of the deposition cycles of In-Sb and deposition time of the flows of indium and antimony for each cycle). An effective thickness of the epitaxy layer depended on the growth conditions and was 1.3-2.0 ML. The main growth parameters of epitaxy are in Table 1.

All samples were prepared in the cross sections (CS) (110) and plane view (PV) (001) foils by means of ion etching for CS and chemical etching using standard solution 3H_{2}SO_{4} : 1H_{2}O_{2} : 1H_{2}O for PV.

Table 1

| Specimen | Mode of epitaxy | The expected thickness of the InSb layer, ML |
|----------|-----------------|------------------------------------------|
| 938      | ALE             | 1.5                                      |
| 939      | ALE             | 2.5                                      |
| 940      | ALE             | 2.0                                      |
| 941      | ALE             | 1.3                                      |
| 1025     | ALE             | 1.5                                      |
| 1027     | MBE             | 1.5                                      |

3. Experimental results and their discussion
We carried out the investigations by transmission electron microscopy (TEM) in the scanning mode (STEM), in the two-beam diffraction contrast mode and in the multi-beam interference contrast mode (HREM) using JEOL-4000EX and TITAN 80-300 microscopes.

For the analysis of the structural features we used EM images filtered from “noise”, associated with non-elastic scattering. We obtained two types of the cross sectional images. The first (Figure 2) is typical only for sample № 940. The second type is characteristic for the other samples (Figure 3). The analysis showed a formation of continuous (wetting) layer with islands for all specimens. The wetting layer is coherent interfaced with the crystal lattice of the matrix, its average thickness was 3-4 nm having statistical deviation about 0.5 nm. The experimental thickness of the epitaxial layer in the AlAs matrix differed from the expected thickness, so we supposed formation of the solid solution In\textsubscript{x}Al\textsubscript{1-x}Sb\textsubscript{y}As\textsubscript{1-y} in the matrix instead of the binary compound. We measured the interplanar distance on the obtained HREM images (Figure 2) using digital diffraction patterns. The interplanar distances in continuous areas of the embedded epitaxial layer differed from the corresponding values of the matrix less than 3%, indicating the concentration of indium and antimony in the solid solution is low. To
estimate chemical composition of $\text{In}_x\text{Al}_{1-x}\text{Sb}_y\text{As}_{1-y}$ we used Vegard’s law. The possible values of «$x$» and «$y$» in the solid solution do not exceed 0.2, if «$x$» is equal «$y$».

Figure 2. The cross sectional dark field EM images of sample № 940 (the top), where large islands in the AlAs matrix appear. The arrows point to STEM (the left) and HREM (the right) images of the according cross sectional areas. Titan 80-300, 300 keV and JEOL-4000EX, 400 keV.

Figure 3. The characteristic cross sectional bright field EM images of samples № 938, № 939, № 941, № 1025, and № 1027. The dotted rectangle on the enlarged image of the epitaxial layer corresponds to an island. The arrow points to the typical HREM image of the islands. JEOL-4000EX, 400 keV.

We got TEM images of the PV foils to study an island film which had the definite MBE conditions (Figure 4, Figure 5). These images revealed small and large islands in the epitaxial layer. The small
islands have the contrast, which is characteristic for coherent inclusions, meaning their crystal lattice is interfaced with the lattice of the matrix coherently (Figure 4a, Figure 5a, Figure 5e, Figure 5f, Figure 5i, and Figure 5l). The large islands are relaxed, so parallel moiré pattern originated in the TEM images of these islands (Figure 4b, Figure 5b, Figure 5c, Figure 5g, and Figure 5j). Structural defects in a volume of the islands result in the break and local non-parallelism of the moiré pattern’s fringes.

![Figure 4](image1.png)

Figure 4. The bright (a) and dark (b) field EM images of the island film (sample № 940): the arrows correspond to typical large and small islands. The characteristic cross sectional images of the islands are at insertions. JEOL-4000EX, 400 keV.

![Figure 5](image2.png)

Figure 5. The dark field EM images of the island films of samples № 938 (a), № 939 (c, f), № 941 (b, d, e), № 1025 (g, h, i), and № 1027 (j, k, l). Figure 5d and Figure 5e, obtained under the precise Bragg condition and the “weak beam” condition correspondingly, present the enlarged image of the rectangle in Figure 5b. Figure 5f is the enlarged image of the rectangle in Figure 5c. PV (001), JEOL-4000EX, 400 keV.
For specimen № 938 the contrast on the island TEM images (Figure 5a) indicates the existence of a disk-shaped "pedestal" under each island. The contrast increases along the outer perimeter, meaning the lateral surface of the island has a step form. The islands have coherent interfacing with the crystal lattice of the matrix and uniform distribution. In this case, we did not detect the large islands shown in Figure 2, Figure 4, and Figure 5 during the TEM study. In other specimens the small coherent islands were localized near the large islands.

We estimated the average lattice constant of the relaxed islands \(a_d\) using the moiré pattern period and digital diffraction patterns:

\[
\begin{align*}
\text{specimen № 939}, & \quad a_d \approx 5.96 \text{ Å} \\
\text{specimen № 940}, & \quad a_d \approx 6.15 \text{ Å} \\
\text{specimen № 941}, & \quad a_d \approx 5.90 \text{ Å} \\
\text{specimen № 1025}, & \quad a_d \approx 5.87 \text{ Å} \\
\text{specimen № 1027}, & \quad a_d \approx 6.00 \text{ Å}
\end{align*}
\]

An evaluation of the chemical composition of the formed solid solution \(\text{In}_x\text{Al}_{1-x}\text{Sb}_y\text{As}_{1-y}\) was done assuming that the hetero-epitaxial stress was relaxed and Vegard’s law was kept. If \(x\) is equal \(y\), then values of \(x\) and \(y\) in \(\text{In}_x\text{Al}_{1-x}\text{Sb}_y\text{As}_{1-y}\) do not exceed 0.35 (specimen № 939), 0.59 (specimen № 940), 0.25 (specimen № 941), 0.27 (specimen № 1025), and 0.40 (specimen № 1027).

We used the geometric phase analysis to visualize the distribution of misfit dislocation cores on the interface (Figure 6). The Burgers vectors were determined by classic method with the Burgers contour (Figure 7).

![Figure 6](image)

Figure 6. The dark field TEM image of interface of large island and matrix (specimen № 940) (a) and the distribution map of interplanar distances for (1-11) (b) and (-111) (c). Figure 6d is superposition of Figure 6b and Figure 6c. We used the geometric phase analysis in the dotted rectangle in Figure 6a to obtain Figure 6b and Figure 6c.

![Figure 7](image)

Figure 7. The digital images, which were filtered near misfit dislocation core using reflexes (1-11) (a) and (-111) (b). The Burgers contour of the misfit dislocation of specimen № 940 (the dotted line) and its Burgers vector (the solid line). The letters “A” and “B” point to start and end of the Burgers contour.
The method revealed, that a base type of dislocations in the InSb/AlAs system was 60°-dislocation with the Burgers vectors like $\frac{a}{2}<110>$, but the dislocation cores were splintered. Every misfit dislocation was formed by two close 60°-dislocations with the Burgers vectors like $\frac{a}{2}<110>$. The sum Burgers vector of the dislocation pair was situated in the plane of the interface. Thus the dislocation pair was similar to edge misfit dislocation.

4. Conclusion
We revealed origination of the embedded In$_x$Al$_{1-x}$Sb$_y$As$_{1-y}$ epitaxial layer in the AlAs matrix (instead of a pure binary compound InSb) during the precipitation of In and Sb on the AlAs surface (and the following deposition of aluminum arsenide layer). Its characteristics depend on the MBE conditions, but the continuous epitaxial layer with islands was grown in all cases. To use ALE is not a critical requirement in this case, because the growth mechanism was uniform for specimen № 1025 (ALE) and № 1027 (standard MBE). The main morphological and structural features of the formed epitaxial film are:

1. There is the wetting layer with possible concentration of In ($\langle x \rangle$) and Sb ($\langle y \rangle$) up to $\langle x \rangle=\langle y \rangle=0.2$. The continuous layer has coherent interfacing with the crystal lattice of matrix.
2. In sample № 938 we found small islands with uniform distribution on the wetting layer’s surface. They have coherent interfacing with the AlAs lattice too. Their average size and density are 100 nm and $10^8$ cm$^2$ accordingly.
3. In the other samples the small coherent islands were localized around large islands.
4. The epitaxial film contains incoherent (relaxed) large islands with possible concentration of In ($\langle x \rangle$) and Sb ($\langle y \rangle$) up to $\langle x \rangle=\langle y \rangle=0.35$ (specimen № 939), $\langle x \rangle=\langle y \rangle=0.59$ (specimen № 940), $\langle x \rangle=\langle y \rangle=0.25$ (specimen № 941), $\langle x \rangle=\langle y \rangle=0.27$ (specimen № 1025), and $\langle x \rangle=\langle y \rangle=0.40$ (specimen № 1027). The parallel moiré pattern appears in the TEM images of these islands. The defects of crystal structure originated in specimen № 940.

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