Effect of growth technological conditions on the heterointerface thickness in the InAs/GaSb strained-layer superlattices grown by MOCVD

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Abstract. In this article, we investigated the effect of technological growth parameters by metal-organic chemical vapor deposition (MOCVD) method on the thickness of the transition layers, which affect the value of tensile strain in the structure, in the InAs/GaSb superlattice. We consider that the thickness of transition layers depends on the roughness of the growth surface and the technological conditions of growing single layers in the superlattice. At the first stage, we have determined the optimal annealing parameters for the minimization of the substrate roughness as T = 650 °C and t = 8 min, with a minimum value of 1.1 nm. At the second stage, studies were carried out on the effect of the sequence of elements and the delay time of reagent supply during the growth of InAs and GaSb layers on the quality of heterointerface. The stress analysis of the obtained structures was performed by reflectance anisotropy spectroscopy. It was shown that a superlattice becomes less tense with the increase in the number of pairs of alternating layers.

1. Introduction

The InAs/GaSb strained-layer superlattice (SLS) is a new material for manufacturing the 3 – 15 µm infrared devices. This material system can change the energy structure due to the tensile strain caused by the difference of parameters of the crystal lattice. Nowadays, the common method used for SLS growing is molecular beam epitaxy (MBE) [1], however, it is less productive in comparison with metal-organic chemical vapor deposition (MOCVD), which is more preferable, especially for SLSs with a large number of periods.

The main aim of this work is to study the effect of technological growth conditions on the thickness of transition layers. We consider that the thickness of transition layers depends on the roughness of the growth surface and the technological conditions of the growth of each layer. This work also proposes the method for estimation of elastic stresses in superlattice structures by reflectance anisotropy spectroscopy (RAS).

2. Experiment

The experiments were carried out on AIX-200 (AIXTRON, Germany) on n-GaSb (001) substrates. The pressure in the reactor: 76 Torr. The carrier gas: purified hydrogen with a dew point not worse than −100°C; total flow through the reactor: 5.5 standard litre per minute; further growth details are described in [2]. During the study of the surface roughness, annealing of substrates was performed at...
T=600 – 700°C for 2 – 8 minutes. The next stage of the study was focused on the effect of the flow sequence of reactants during the growth of InAs and GaSb layers on the quality of heterointerfaces. For this purpose, SLS composed of 8 pairs of InAs/ GaSb alternating layers was grown. During the growth process, one of the reactants was supplied 2 or 5 seconds earlier than the other and the growth of the layer was completed by the simultaneous stopping of the reactant flow. Growth sources: trimethylindium (TMIn), triethylgallium (TEGa), trimethylstiban (TMSb) and arsine (AsH₃). Growth temperature: 500°C. The pause time, when none of the reactants was supplied (the same for all pairs): 10 seconds. Figure 1 shows the timing diagram of the flow sequence of the reactants during the growth of the structure. The samples were examined using the transmission electron microscope (TEM) and the atomic force microscope (AFM).

![Figure 1. The timing diagram of the flow sequence of the reactants during the SLS growth of the structure.](image)

Residual elastic stresses in the grown SLSs were examined by RAS ex-situ at room temperature. In the RAS equipment described in [3], a xenon arc lamp was used as a source of excitation, and an ultraviolet (UV)-sensitive photomultiplier as a detector of the reflected light. The experimental setup allowed us to measure the reflection anisotropy (RA) signals within the spectral range from 1.5 up to 5 eV.

3. Experimental Results
The GaSb substrates were prepared by etching [4]. The surface roughness after etching was 30 nm. For SLSs, having layers not thicker than 10 nanometers, the morphology is of great importance, because it is impossible to produce high-quality thin layers when roughness is commensurate with the layer thickness. To possibly smooth the substrates, they were annealed for 2 minutes at 650°C and 700°C. After AFM examination, it was found out, that root mean squared (RMS) value of surface roughness after annealing at 650°C was less than that at 700°C. The data obtained after the annealing of substrates at different temperatures and annealing times are given in table 1. They demonstrate: the
longer annealing, the lower the roughness (Figure 2), the lowest RMS value $R_q = 1.1$ nm was obtained by annealing at 650°C for 8 minutes.

**Table 1.** The roughness obtained after annealing of substrates at different temperatures and annealing times.

| Temperature, °C | Time, min | Root mean squared roughness $R_q$, nm |
|-----------------|-----------|--------------------------------------|
| 650             | 2         | 6.7                                  |
| 650             | 4         | 4.0                                  |
| 650             | 8         | 1.1                                  |
| 700             | 2         | 13.8                                 |

**Figure 2.** Graph of roughness versus annealing time and temperature.

A structure having 8 pairs of alternating layers InAs and GaSb was grown. The boundaries of the layers are planar and look the same for all 8 pairs of layers regardless of supply time; the growth rate was 1.5 nm/min. At the boundaries, there are 0.5 – 0.6 nm transition layers with small deviations in the lateral direction (Figure 3). The boundaries are darker as compared to GaSb and InAs due to the fact that the contrast intensity of the transition layers in the selected image conditions is less than that of GaSb and InAs.
Figure 3. Dark-field cross-sectional sample image (1-10) obtained by transmission electron microscopy under two-beam conditions with \( g = (002) \). Odd layers: InAs, even layers: GaSb.

To study stress in SLSs, a special series of structures was grown on \( n \)-\((5-7) \times 10^{17} \text{ cm}^{-3}\) GaSb. They are schematically shown in Figure 4.

Stress diagnostics of these structures was performed by RAS [3]. Unlike the electron diffraction, allowing to detect stress only in few uppermost monolayers, RAS diagnoses stress in a layer thickness of which is limited by the light penetration depth. This modulation method measures the spectral dependence of the normalized difference between reflectance coefficients for two orthogonal polarization directions of light normally incident on the surface. Its sensitivity up to \( 10^{-4} \) allows detecting uniaxial stress corresponding to the difference between bond length in orthogonal directions 0.1%. These features make RAS preferential for stress diagnostics of multilayer heterostructures [5].

Figure 5(a) shows RA spectra of the samples under investigation, the substrate covered by the buffer layer (solid line), the sample with 4 nm InAs, grown on buffer layer (dash line) and a superlattice SLSx2 (dash-dot line). As stress in the structures occurs simultaneously both in the layer and in the substrate, we focus on contribution in RA signal from the GaSb substrate near the
GaSb/InAs interface. To estimate stress numerically, we analyze spectral feature near the optical transition $E_1$, whose position for GaSb is marked by an arrow on Figure 5(a). It was earlier shown that the amplitude of this feature is proportional to the electric field component perpendicular to the surface in the RA spectra of (001) surfaces of cubic semiconductors III-V [3]. The in-plane uniaxial stress in the structures induces an electric field in (001) direction as a result of the piezoelectric effect in III-V. Thus the stress value should be proportional to the near-surface electric field, and to the amplitude of the feature marked by the arrow. As one can see, the GaSb substrate is tensed approximately equally in the structures with one layer InAs (dash line) and SLSx2 (dash-dot line). This stress diminishes rapidly with increasing the number of the pairs of alternating layers, as it can be seen from Figure 5(b). The phase of the feature at $E_1$ allows identifying the uniaxial stress direction. In all the obtained structures the near-interface region of GaSb is compressed in [-110]. The amplitude of the RA signal in the UV spectral region corresponds to the anisotropic roughness value [6]. The decrease of the UV signal in the spectrum of SLSx10 testifies the less tension compared to SLSx2.

RA spectra also demonstrate the presence of InGaAsSb solid solutions forming the interfaces between alternating layers. This is evidenced by blue shifting and blurring of the sharp spectral singularities at the energies of optical transitions $E_1$, $E_1+\Delta$.

![Figure 5](image)

**Figure 5.** RA spectra of the structures under investigation: (a) – solid line: buffer layer of undoped GaSb on GaSb n-(5-7)$\cdot 10^{17}$, dash line: 4nm InAs on the previous structure, dash-dot line: SLSx2; (b) - dash-dot line: SLSx2, solid line: SLSx10.

As a result of the study, it was shown, that the smoothest surface (RMS roughness $R_q = 1.1$ nm) of the GaSb substrate was obtained at the optimal temperature 650°C in the H$_2$ flow without reactants at the annealing time of 8 minutes. At a delay time of 5 seconds, the boundaries of the layers in the structure with InAs/GaSb alternating layers were planar with the transition layer thickness of 0.5 – 0.6 nm. The stress analysis by RAS technique has shown that the strain in the InAs/GaSb structures decreases with an increase in the number of pairs of InAs/ GaSb alternating layers.

References

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