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Citation style: Papis-Polakowska E., Kaniewski J., Szade Jacek, Rzodkiewicz W., Jasik A., Reginski K., Wawro A. (2012). Study of interfaces chemistry in type-II GaSb/InAs superlattice structures. "Physics Procedia" 32 (2012), s. 184–190. DOI: 10.1016/j.phpro.2012.03.540



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Study of interfaces chemistry in type-II GaSb/InAs superlattice structures

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Abstract

There is a considerable interest in type-II GaSb/InAs superlattice system due to several modern applications including infrared detectors. In these studies X-ray Photoelectron Spectroscopy (XPS) and Spectroscopic Ellipsometry (SE) have been used to extensive characterization of the surface and interface of GaSb/InAs superlattice. Application of XPS and SE techniques provide precise information from topmost layers of structure and allow excluding presence of GaAs-type interfaces in GaSb/InAs superlattices. Simultaneously, these results indicate that InSb-type or GaInSb-type interfaces have been detected in the structures studied.

Keywords: superlattice, GaSb, surface, interface, X-ray Photoelectron Spectroscopy

1. INTRODUCTION

GaSb-based superlattices (SLs) are well recognized for obtaining high-performance modern micro- and optoelectronic devices including infrared detectors, long-wavelength lasers and high-frequency, low-voltage field effect transistors. In particular, the GaSb/InAs system is promising for the next generation mid-infrared photodetectors focal plane arrays as an alternative material to usually used HgCdTe alloy for better uniformity, reduced tunneling currents and suppressed Auger recombination [1-3]. For many of type-II GaSb/InAs superlattices applications the band alignment plays a critical role in the device performance, however, a determination of band alignments in mixed anion/cation systems is complicated by the fact that two different types of interfaces can be formed. In the case of an InAs/GaSb heterojunction, both InSb-like and GaAs-like interfaces can be formed [4] and it is very important to determine what effect changing the interface composition on band alignments in the GaSb/InAs. Because the InSb-like interface introduces compressive strain, but GaAs-like interface initiates tensile strain, the strain of multilayered complex superlattice structure can be modified and compensated by using proper sequence of interfaces in devices. Several studies have investigated interfacial control in mixed anion heterostructures, and have indicated that the exchange reaction is difficult to suppress [5-7]. In particular, it has been found that structures containing only In-Sb interface bonds have increased average lattice constants and reduced band gaps compared to structures containing only Ga-As interface bands. While a number of studies have been performed in order to present the competitive incorporation behaviour of arsenic and antimony during the Molecular Beam Epitaxy (MBE) growth of GaSb-based superlattices [8-10], little is known on chemistry and kinetics of the As-for-Sb and Sb-for-As anion exchange reactions, when GaAs surface is exposed to Sb flux and when GaSb surface is exposed to an As flux respectively. Specific complex multilayered construction of InAs/GaSb SLs requires high quality of surface and interface with good morphology and controlled composition. On the other hand, because of the critical dependence of the electronic and optical properties of the superlattice on the thickness and composition of individual layers, as well as of interfaces, non-destructive and atomic-level resolution techniques are required for characterization of surface/interface in SLs structures. In particular, X-ray Photoelectron Spectroscopy (XPS) [6] and High-Resolution X-ray Diffraction (HR XRD) [11] have been reported in literature as prominent methods for surface chemistry analysis and SLs structures characterization, respectively.

In particular, Losurdo *et al.* presented the extensive results of the superlattice surface and interface study carried out by XPS, XRD, AFM (Atomic Force Microscopy), SE (Spectroscopic Ellipsometry) and TEM (Transmission Electron Microscopy) complementary techniques [12].

The Fractional Derivative Spectrum (FDS) model in connection with the Spectroscopic Ellipsometry has been used for optical analysis of the superlattices layers as a helpful technique to extract basic information on relevant physical quantities from the $\epsilon E^2(E)$ spectra [13-15].

In the present study, 30-period GaSb/InAs superlattice structures were grown. The interfaces were formed by exposure of the GaSb and InAs layer to As_2 and Sb_2 flux respectively, The XPS, SE and AFM complementary methods were used for extensive characterization of the surface and interface in type-II GaSb/InAs SLs.

2. EXPERIMENTAL

The surface study was performed on a superlattice structure consisting of 30 period 10 monolayers (MLs) InAs/ 9ML GaSb. The SLs structures were grown on undoped (100) GaSb epi-ready substrates in a Riber 32P Molecular Beam Epitaxy (MBE) system equipped with group V solid sources specifically cracked As and Sb sources to provide dimmer As_2 and Sb_2 species. The thickness of individual InAs and GaSb layers was $d_{InAs}=3.02\text{\AA}$ and $d_{GaSb}=3.05\text{\AA}$ respectively. One pair of InAs/GaSb system together with the interfaces created during epitaxial growth characterized by 6.3nm thickness and the overall thickness of InAs/GaSb SLs structure was about 180nm. The GaAs- and InSb-like interfaces in superlattices were formed by the time controlled As_2 exposure of GaSb before InAs layer growth and Sb_2 exposure of InAs before GaSb layer formation, respectively. High-resolution X-ray Diffraction using PANalytical X'Pert PRO Diffractometer and High-resolution Transmission Electron Microscope JEOL JEM-2100 was used to provide preliminary information on the quality and lattice mismatch in GaSb/InAs periodical layered structures. The chemistry of surface and interfaces in InAs/GaSb SLs was analyzed by XPS using a Physical Electronics PHI 5700 spectrometer with monochromatized Al K_{α} radiation system. The energy resolution of the spectra presented within this work can be estimated as 0.35 eV. The detection limit for most elements is about 0.1 at. %. The XPS database from Physical Electronics Handbook of Photoelectron Spectroscopy [16] and National Institute of Standards and Technology (NIST) database were applied as the preliminary reference for identifying the chemical states of elements. Additionally, the reference compounds GaAs, GaSb and InAs have been studied at the same parameters of the XPS spectrometer. The MULTIPAK software from Physical Electronics was used to deconvolute the XPS spectra and calculate atomic concentrations. The XPS depth profile was obtained during the sputter etching sequences of GaSb/InAs structures using Ar^+ ion beam of energy 1 keV. The SL pseudodielectric function $(\epsilon)=\epsilon_1 + i\epsilon_2$ was measured in the 1.1-5.5 eV photon energy range with the energy resolution of 0.01eV using J. A Woollam Variable Angle Spectroscopic Ellipsometer (VASE) at multiple angles of incidence in the range 55-75°. Surface morphology was examined by AFM (Digital Instruments Nanoscope IIIa) technique.

3. RESULTS AND DISCUSSION

3.1 X-ray Photoelectron Spectroscopy results

Preliminary characterization of 30 period 10 GaSb MLs /9 InAs MLs superlattices carried out by HR XRD and HR TEM techniques indicated well-defined high quality multilayered SL structures with a smooth and sharp interfaces between InAs and GaSb layers. The results of these studies will be presented in a separate paper. The XPS analysis was focused on the spectra of Sb 4d, As 3d, Ga 3d levels with similar Binding Energies (BE) in order to assure similar surface sensitivity caused by the effect of energy dependent Inelastic Mean Free Path (IMFP) of photoelectrons. The value of IMFP for such electrons can be estimated to be about 30 \AA [17]. Only In 3d line is characterized by higher BE (above 400 eV) and slightly higher surface sensitivity.

The results of XPS measurements of GaSb/InAs superlattice structures (multilayer) compared to reference compounds GaSb, GaAs and InAs are shown in Fig. 1 where the spectra of the most

prominent Sb, As, Ga and In lines and valence band are displayed. Analysis of Sb 4d and As 3d XPS spectra (Figs. 1 a, b) indicates to the presence of GaSb and InAs, as expected for the main components of the multilayer although the In lines show different features as discussed later in the text.

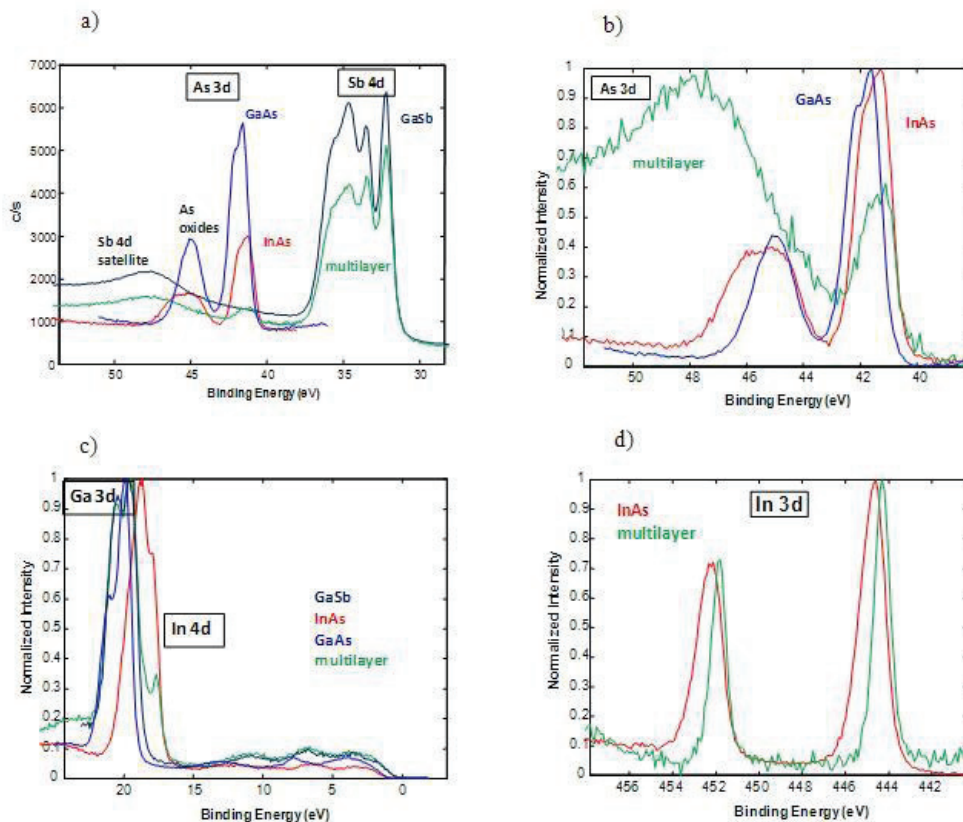


Fig. 1. Comparison of Sb 4d and As 3d (a) and (b); Ga 3d (c) and In 3d (d) XPS spectra for GaSb/InAs SLs (multilayer) and GaSb, GaAs and InAs reference compounds.

The broad line with maximum at the energy of 48 eV corresponds to a satellite of the Sb 4d photoemission line. Similar feature is seen in the spectrum obtained for pure GaSb (Fig. 1a). Therefore, this structure cannot rather be ascribed to formation of AsSb_y compounds as suggested by Losurdo *et al.* [12]. Moreover, satellites separated by about 16 eV observed in pure GaSb are accompanying by other Sb lines. The presence and shape of satellites depend on the Sb chemical state, since we did not find them in strongly oxidized GaSb surface. The spectral line at about 44–46 eV is related to arsenic oxides. Analysis of As 3d and Ga 3d spectra (Fig. 1 b, c) prove that GaAs does not exist in the SL structure. The more detailed analysis of the Ga 3d line is presented by Papis *et al.* [18]. One has to remember that Ga lines have strong contribution from the surface oxides. Interpretation of the In 3d spectrum is more complex. The shift of the In 3d line towards lower binding energy with respect to pure InAs (Fig. 1d) suggests a modification of the chemical bonding of indium in the superlattice. One of the reason can be related to the presence of InSb or GaInSb in the superlattice structures. Formation of such compounds was reported previously. Simultaneously the As 3d line fits well to the ones related to InAs - see Fig. 1a, b. It may be related to the structure of the last interface where Sb_2 exposure of InAs before GaSb layer formation was used during superlattice growth. In order to clarify this problem we performed the depth profile for the superlattice of 30 periods 10 GaSb MLs/9 InAs MLs. The low energy and low dose of Ar^+ ions and short sputtering cycle enabled detailed analysis of the topmost layers including the oxidized surface. The sputtering rate was estimated to be about 2–4 Å/min.

The composition of the main elements as a function of sputtering time is presented in Fig. 2. One has to remember that the value of IMFP causes that information at every point comes from several atomic layers and sputtering may cause some interlayer mixing.

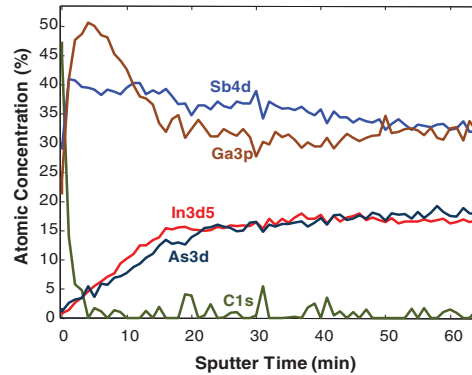


Fig. 2. The depth profile of GaSb/InAs SLs obtained using Ar⁺ ion beam etching.

Information is thus partly integrated for the depth of the order of 3-4 nm. The evolution of the particular XPS spectra is shown in Figs. 3-5. The topmost layers are influenced by oxidation. An exact XPS characterization of GaSb surface has been previously reported by Papis-Polakowska [19].

The Ga oxides and Sb oxides are well visible in the front spectra. A slight shift of the Sb lines was observed during sputtering which probably can be attributed to some mixing of Sb and In in the region of interface. Also the composition changes in the region 10-25 min of sputtering can be assigned to the first interface (Fig. 3). The excess of In and Sb with respect to their standard neighbours from the main layers may indicate formation of InSb or GaInSb at the interface. The In 3d line (Fig. 5) shows a slight shift of the peak maximum position with sputtering time, that can be related to the reactions at the interface. Generally, after 45 min of sputtering the profile shows a relatively uniform composition with two main components - GaSb and InAs, which may be caused by partial mixing of layers due to sputtering.

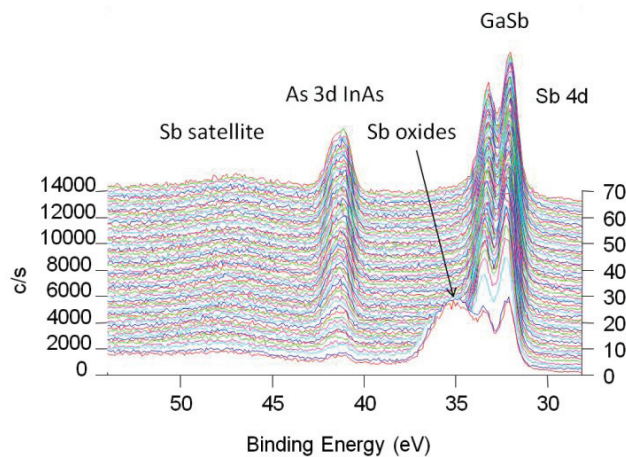


Fig. 3. XPS spectra of Sb 4d and As 3d region from GaSb/InAs SLs obtained during depth profiling. The right axis corresponds to the numbers of sputtering cycles and cumulated sputtering time in minutes.

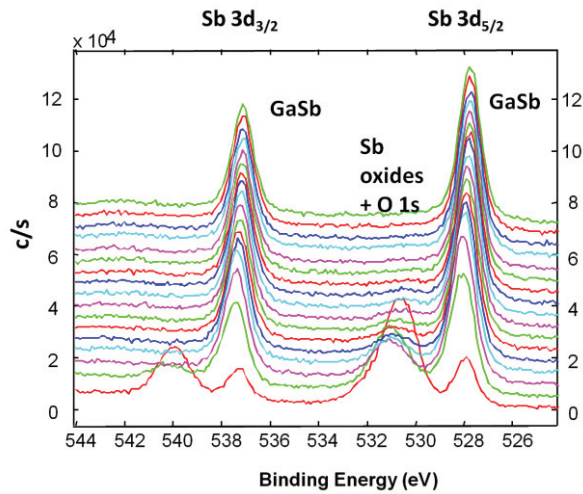


Fig. 4. XPS spectra of Sb 3d (a) and In 3d_{5/2} (b) of GaSb/InAs SLs obtained during depth profiling.

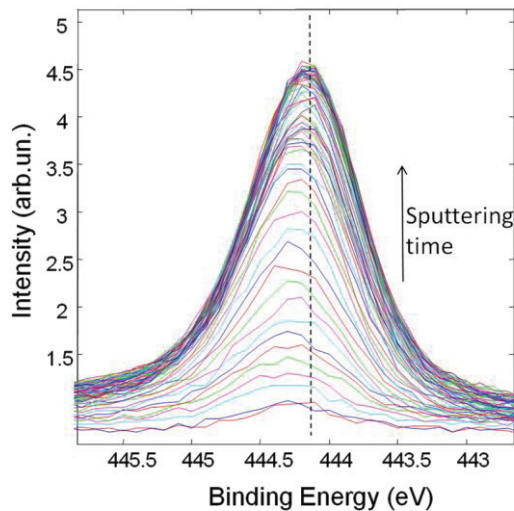


Fig. 5. XPS spectra of In 3d_{5/2} of GaSb/InAs SLs obtained during depth profiling.

3.2. VASE analysis

The real (ϵ_1) and imaginary (ϵ_2) components of the effective complex dielectric function versus photon energy E , and the second energy derivative $d^2\epsilon_2/dE^2$ of 30 period 10 GaSb MLs /9 InAs MLs superlattice structures are shown in Fig. 6 a, b. Critical Points (CP) are resolved as minima in $d^2\epsilon_2/dE^2$ at energies of 1.81 eV, 2.08 eV, 2.54 eV and 4.07 eV. These critical points have been assigned to the E_1 interband transition in InSb, to the E_1 transition in GaSb, to a superposition of the GaSb $E_1+\Delta_1$, to E_2 transition in GaSb and E_1 transition in InAs, respectively. The critical point at energy 2.21 eV corresponds to a superposition of the GaInSb $E_1+\Delta_1$ [20]. One can notice that the VASE results do not indicate critical point corresponding to GaAs. The detailed description of optical analyses of GaAs substrate by EDS model has been reported by Kulik *et al.* [21].

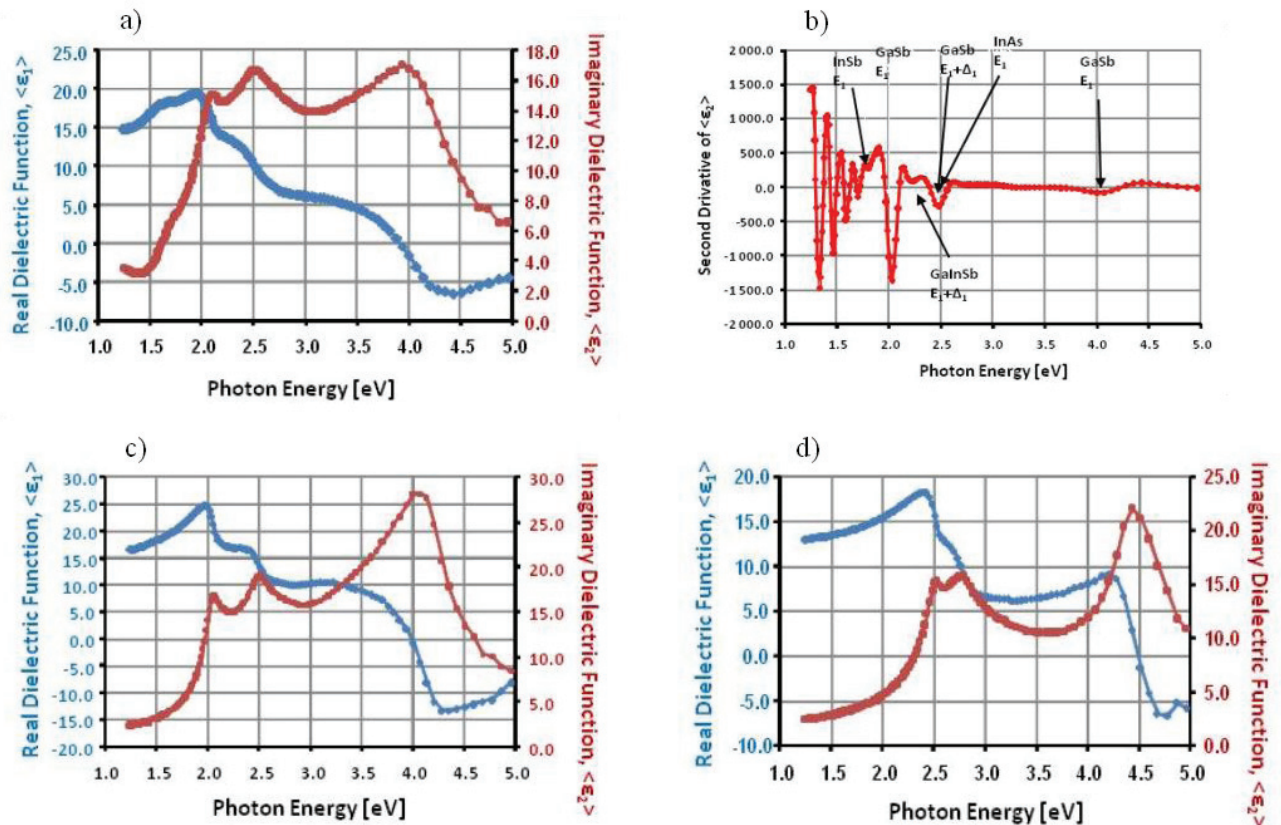


Fig. 6. The spectral characteristics of: (a) $\epsilon_1(E)$ and $\epsilon_2(E)$ for GaSb/InAs SLs, (b) the second energy derivative of ϵ_2 for GaSb/InAs SL, (c) $\epsilon_1(E)$ and $\epsilon_2(E)$ of GaSb substrate, (d) $\epsilon_1(E)$ and $\epsilon_2(E)$ of InAs substrate.

The values of CP in the second energy derivative of GaSb/InAs superlattices structures were extracted by using FDS and VASE techniques for GaSb, InAs and InSb standard samples. The $\epsilon(E)$ spectra of GaSb and InAs are presented in Fig. 6 c, d. The effective pseudodielectric function of GaSb/InAs SL was calculated in a multilayer model, describing the dielectric functions of the individual layers by ensemble of three Gaussian oscillators to model to critical point structures.

The VASE measurements of GaSb/InAs superlattice structure are very sensitive to their surface and interface morphology. The AFM measurements show that superlattice structures have high quality, smooth surface with RMS=0.2 nm.

4. CONCLUSIONS

Summarize, one can conclude, that XPS and SE are suitable methods for superlattice characterization. Using these methods, the complementary studies of the GaSb/InAs SLs surface/interface chemistry were performed. Anion exchange reaction as a result of exposure of GaSb under As_2 and InAs under Sb_2 fluxes, respectively have been investigated. X-ray photoelectron spectroscopy was used to gain knowledge about the electronic structure and chemical composition of the topmost layers from the superlattice. The depth profile XPS analysis performed with low energy Ar^+ ions beam allow to get insight to the interface region. Despite the main components, GaSb and InAs, Ga and Sb oxides were detected on the surface. Analysis of the As and In lines indicates to formation of InSb-type or GaInSb-type compounds at the layer interface.

The presence of GaAs-type interface has been excluded in the structures studied. It means that Sb-for-As anion exchange does not exist. Moreover, formation of AsSb_y compounds, suggested in the literature, was not detected in our samples and we believe that the interpretation of the XPS peak at about 48 eV should be revised. The results of SE accompanied with FDS procedure confirmed these conclusions. Additionally, it is shown that using GaSb, GaAs and InAs reference compounds is a good method to study interface chemistry from both XPS and SE measurements. This approach to type-II GaSb/InAs superlattice structures is innovative and have not been applied so far.

Acknowledgements – Authors would like to thank Dr I. Sankowska and Dr J. Kubacka-Traczyk for HR XRD measurements, and Dr. A. Laszcz and Dr. M. Wzorek for HR TEM measurements.

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