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Evaluation of antimony segregation in InAs/InAs$_{1-x}$Sb$_x$ type-II superlattices grown by molecular beam epitaxy

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InAs/InAs$_{1-x}$Sb$_x$ type II superlattices designed for mid-wavelength infrared photo-detection have been studied using several electron microscopy methods, with specific attention directed towards interface chemical diffusion caused by Sb segregation. Reciprocal-space image analysis using the geometric phase method showed asymmetric interfacial strain profiles at the InAs-on-InAsSb interface. Measurement of local Sb compositional profiles across the superlattices using electron energy-loss spectroscopy and 002 dark-field imaging confirmed asymmetric Sb distribution, with the InAs-on-InAsSb interface being chemically graded. In contrast, the InAsSb-on-InAs interface showed a small intrinsic interface width. Careful evaluation of the experimental Sb composition profiles using a combined segregation and sigmoidal model reached quantitative agreement. Segregation dominated over the sigmoidal growth at the InAs-on-InAsSb interface, and the segregation probability of 0.81 ± 0.01 obtained from the two microscopy techniques agreed well within experimental error. Thus, 81% of Sb atoms from the topmost layers segregated into the next layer during growth causing the interfaces to be broadened over a length of ~3 nm. This strong Sb segregation occurred throughout the whole superlattice stack, and would likely induce undesirable effects on band-gap engineering, such as blue-shift or broadening of the optical response, as well as weakened absorption. © 2016 AIP Publishing LLC. [http://dx.doi.org/10.1063/1.4942844]

I. INTRODUCTION

Mercury cadmium telluride (MCT) semiconductor alloys remain the most widely used materials for infrared (IR) photo-detection in mid-wavelength and long-wavelength ranges despite major disadvantages of intrinsic Auger recombination and small effective mass.1 Type-II superlattices (T2SL) have been proposed as promising alternatives, because they may overcome these MCT problems by providing flexible and more controllable band-gap engineering through design of the SL layer thickness/composition and coherency strain, as well as giving additional benefits including larger effective masses, better Auger suppression, higher mechanical strength, and lower relative cost.1,2 The most studied III-V T2SL system, InAs/Ga$_{1-x}$In$_x$Sb, suffers from short minority carrier lifetimes, most likely due to Shockley–Reed–Hall recombination induced by Ga-related native defects.3 Ga-free InAs/InAs$_{1-x}$Sb$_x$ T2SLs, on the other hand, have since demonstrated significantly improved minority carrier lifetimes, which are up to and exceeding an order of magnitude longer than measured for Ga-containing T2SLs, depending on the wavelength.4,5 Ga-free InAs/InAs$_{1-x}$Sb$_x$ T2SLs grown with carefully chosen SL design for strain balance and proper control over growth conditions have very low densities of extended defects.6,7 However, asymmetric SL peaks visible in high-resolution X-ray diffraction (HRXRD) patterns suggest the possibility of non-uniform InAs$_{1-x}$Sb$_x$ layer thickness and/or Sb composition throughout the SL stack. Interface chemical diffuseness that is deduced based on evidence from scanning tunneling microscopy and transmission electron microscopy (TEM) may also be present.6,8–11 Either type of compositional disorder could introduce perturbations to the SL band structure, and cause deviation from the design parameters and deterioration of the optoelectronic response.

TEM can be highly effective for studying structural and compositional properties of semiconductor structures with high spatial resolution. In this paper, the Sb compositional profile in a InAs/InAs$_{1-x}$Sb$_x$ T2SL has been investigated using three independent TEM techniques, namely, strain characterization using geometric phase analysis (GPA),12 compositional analysis using electron energy-loss spectroscopy (EELS) in scanning mode, and chemically-sensitive 002 dark-field (DF) imaging.13 The multiple linear least-squares (MLLS) method14 was used in EELS analysis to extract quantitative information about the Sb composition profile. While strain analysis using GPA serves as an indirect technique for providing qualitative information on Sb composition, EELS and 002 DF combined with kinematical calculations can provide quantitative characterization of Sb segregation.
compositional variation/evolution along the growth direction. All three techniques independently revealed the existence of Sb segregation at the InAs-on-InAsSb interface. Furthermore, detailed analysis of the Sb compositional profiles provided by EELS and 002 DF characterization allowed extraction of quantitative information, including interface width and segregation probability.15

II. EXPERIMENTAL

A. Growth

The InAs/InAs$_{1-x}$Sb$_x$ superlattices studied here were designed for mid-wave infrared photo-detection, and consisted of 58.5 periods of 1.8-nm-thick InAs$_{1-x}$Sb$_x$ layers (nominally $x = 40\%$; HRXRD simulation gave $x$ of 34$\%$ on average) and 6.8-nm-thick InAs layers, sandwiched between two 10-nm AlSb barrier layers and capped with an InAs layer (see Fig. 1 for a schematic of the structure). The superlattices were grown using molecular beam epitaxy without any special interface treatment on unintentionally doped 2"-diameter GaSb nominal-(001) wafers at a temperature of 405 °C (calibrated by pyrometer). Beam-equivalent pressure (BEP) of InAs was fixed at 5.5, and Sb flux was adjusted instead of As flux for alternating the growth of InAs and InAs$_{1-x}$Sb$_x$ layers.

B. Characterization

1. Geometric phase analysis

Cross-sectional TEM specimens suitable for imaging and spectroscopy studies were prepared along both the [110] and the orthogonal [1–10] directions using conventional mechanical polishing and dimple grinding, followed by argon ion milling (maximum energy of 2.1 keV) with liquid-nitrogen cooling to help reduce ion-beam damage. Scanning TEM (STEM) and high-angle annular DF (HAADF) images were recorded for GPA analysis using an aberration-corrected JEOL ARM200F operated at 200 kV, with a detector collection half-angle of 90–170 mrad: the collection inner acceptance of quantifiable information, including interface width and segregation probability.15

2. Electron energy-loss spectroscopy

EELS spectrum images were acquired using the JEOL ARM200F, which was equipped with a Gatan Enfinium EELS spectrometer capable of DualEELS acquisition. The instrument was operated at the accelerating voltage of 80 kV, for the purposes of reducing electron-beam-induced damage and enhancing the energy-loss signal by enhancing the inelastic scattering cross-sections.17 The beam convergence semi-angle of 30 mrad was defined by a 40-μm condenser aperture: the electron probe size obtained was ~0.2 nm. The EELS collection semi-angle of 60 mrad was defined using a 5-mm entrance aperture. A rough calculation of the most-probable scattering semi-angles using $\theta_B = \Delta E/E_0$ (where $\Delta E$ is the energy loss, and $E_0$ is the accelerating voltage) yielded 2.8 mrad for the In $M_{4,5}$ edge (edge onset at 443 eV) and 3.3 mrad for the Sb $M_{4,5}$ edge (edge onset at 528 eV) for this study. The difference between the EELS collection semi-angle and the incident-beam convergence semi-angle is 8–10 times larger than the most-probable scattering angle, so that a substantial fraction of the energy-loss signals of interest should have been collected. Moreover, the microscope-specimen geometry was kept fixed during acquisition of all EELS spectra, so that estimates of (changes in) Sb concentration vs. position should be reliable.

An energy dispersion of 0.25 eV/channel and a total of 2048 channels provided the total collected energy range of 512 eV. This particular dispersion setting was chosen to capture only the majority signals of the In and Sb $M$ edges, which are close to edge onset and relatively strong, so that these primary and less noisy portions of the spectra are employed for quantitative analysis. Zero-loss and core-loss spectra of interest were recorded simultaneously, where the zero-loss spectra were utilized to screen for thin areas of interest and avoid excessive plural scattering through examination of relative local thickness. Spectrum images were acquired with a scanning step size of 0.15 nm and a pixel time of 0.02 s. Drift correction was essential (even though the TEM specimen was loaded beforehand to stabilize overnight) to counteract unavoidable slight drift introduced by significant changes of the goniometer setting when locating suitable regions of interest, as well as some ambient interference, because the Sb segregation, which was the feature of interest, occurred over a very fine scale (as revealed by prior GPA analysis). Acquisition of a single EELS spectrum image capturing three superlattice periods typically took 10 min or more.

3. 002 dark-field imaging

The 002 DF investigations were performed on a JEOL JEM 3010 transmission electron microscope operated at 300 kV using an objective aperture size of ~5 μm. To set up the correct $g_{002}$ diffraction condition, the amount of tilt of both the incident electron beam and the TEM specimen was
carefully chosen. In particular, the specimen was tilted 8°–10° from the (110) zone axis towards the [001] pole, while keeping the interface edge-on. The 002 Bragg beam was then selected using an objective aperture to form the DF image. The size of the objective aperture defined the spatial resolution, which was ~0.5 nm in this case.

002 DF imaging is a non-standard TEM imaging technique that uses a chemically-sensitive reflection to form the DF images, which is the 002 reflection in the case of zinc-blende III-V semiconductor compounds. As opposed to HAADF imaging, the contrast in this “structure-factor imaging mode” mainly arises from the difference in the atomic-scattering factors between the group-III and group-V elements. In III–V alloys, the diffracted intensity for the 002 reflection is proportional to the square of the structure factor $F_{002}$, which in turn depends on the difference in the atomic-scattering factors of the alloy components ($f_{III}$ and $f_{V}$ for the group-III and group-V elements, respectively), thus $I_{002} \propto F_{002}^2 \propto |f_{III} - f_{V}|^2$. Hence, when the 002 imaging condition is properly set up for a specimen region of interest with thickness well below the extinction distance for (002) reflections, compositional information can be directly extracted following the procedure proposed by Bithell and Stobbs, which is based on analysis of the DF image contrast using a kinematical calculation.

III. RESULTS AND DISCUSSION

A. Geometric phase analysis

The GPA technique can be used to extract information on both in-plane and out-of-plane strain across the epitaxial layers. The out-of-plane strain is usually defined relative to the (c-axis) growth direction as follows:

$$\varepsilon_{zz} = \frac{c_{\text{alloy}} - c_{\text{reference}}}{c_{\text{reference}}}$$

where $c$ corresponds to the lattice parameter along the growth direction. Extended structural defects were completely absent over very large lateral distance in this particular sample (i.e., the defect density was below the detectability of plan-view TEM), and coherent interfaces were confirmed by high-resolution HAADF STEM images (see Fig. 3(a)). Thus, it was reasonable to assume tetragonal distortion of the lattice (see Fig. 2) and calculate the lattice parameter $c$ using Vegard’s law and Poisson’s ratios (or equivalently, elastic constants). The out-of-plane strain in the growth direction calculated for the different layers is listed in Table I.

HAADF STEM images, rather than HREM, were chosen for strain analysis here, because the contrast in HAADF STEM images is more intuitively interpretable over a greater thickness range and relatively immune to small thickness/focus changes. Moreover, strain in thicker specimen regions can be more reliably measured, since strain relaxation at the specimen surface due to thinning is less severe. In this study, the sample thicknesses were typically ~5–10 times greater than the layer period. The influence of distortions in the scanning system on strain analysis was accounted for by recording successive images in two orthogonal orientations. The images recorded with the scanning direction along or perpendicular

| TABLE I. Calculation of out-of-plane strain (vs GaSb). |
|-----------------|-----------------|-----------------|-----------------|
|                | GaSb            | InAs            | InAs$_{1-x}$Sb$_x$(x = 0.34) | AlSb            |
| $a$ ($\text{Å}$) | 6.0959          | 6.0584          | 6.2012          | 6.1355          |
| $\nu$           | 0.313           | 0.352           | 0.351           | 0.331           |
| $\varepsilon_{zz}$ | 0               | -1.2%           | +3.6%           | +1.3%           |
to the growth direction were used to analyze either the out-of-plane strain or the in-plane strain, respectively.

The GPA analysis was carried out by selecting two non-collinear 111 Bragg spots with smoothed reciprocal-space masks that provided a spatial resolution of 1 nm, and by using the InAs layers as reference since the unstrained GaSb buffer is far out of the field of view. In particular, the InAs reference region was close to the next InAsSb layer in the growth direction, thus away from any possible influence of Sb segregation across the InAs-on-InAsSb interface, as later justified by EELS and 002 DF analysis of the segregation broadening length. The strain map was then converted with respect to the unstrained GaSb. The standard deviation of the strain measurements was 0.3% judging from variation in the random strain present in the unstrained GaSb buffer layer. The out-of-plane strain in the AISb barrier layer was also measured by precession electron diffraction (not shown here) to be 1.3%, agreeing well with the calculation shown in Table I.

The in-plane strain was found to be negligible over all SLs examined (not shown here), which agreed with the previous observations of no extended defects and coherent interfaces. A representative summary of the out-of-plane strain analysis is listed in Fig. 3. It is evident from the out-of-plane strain maps in Fig. 3(b) that the InAs layers were under biaxial tension and the InAsSb layers were under biaxial compression, each with a strain value in fair agreement with the strain calculation for the case of tetragonal distortion (Fig. 3(c)). The transition from tensile strain in the InAs layer to compressive strain in the InAsSb layer occurred relatively quickly indicating sharp interfaces and effective Sb incorporation upon introduction of the Sb flux. In comparison, the transition from compressive strain in the InAsSb layer to tensile strain in the InAs layer was gradual and extended over longer distances. The explanation for such a graded strain profile compared with the InAsSb-on-InAs interface must be related to how the Sb composition evolved, which is the only varying factor across the SL structure. Thus, the graded Sb composition profile is likely to be due to segregation, which was reported previously to occur during MBE growth. As a result, the intended step-like strain profile was altered to a significantly graded profile for the InAs-on-InAsSb interface, in addition to reduced maximum compressive strain in the InAsSb layers and locally weakened tensile strain in the InAs layers. All of these factors would alter the intended band structure and cause the optical response to deviate from the target design.

It is difficult to reliably determine Sb composition or estimate Sb segregation from strain measurements, mainly because (a) at a crystal discontinuity such as an interface, the GPA algorithm averages over both sides of the interface such that the absolute strain value is not the true local strain; (b) there is an inherent error associated with choosing the 111 Bragg diffraction spots: in addition to geometric phase associated with the true strain, an extra non-zero phase term related to the sub-cell structure contributes as false strain, even though relatively small; (c) the spatial resolution is limited, as defined by the masks applied in reciprocal space. Nevertheless, GPA strain analysis is straightforward to implement and provides useful preliminary insight into the existence of fine-scale grading of the Sb compositional profile that is likely to be due to Sb segregation.

B. Electron energy-loss spectroscopy

The major challenge for EELS investigations of these InAs/InAs1-xSbx T2SL samples lies in the fact that the core-loss edges of interest, namely, Sb and In M4,5 edges (edge onsets at 528 eV and 443 eV, respectively), are two closely positioned delayed edges with significant overlap. As a result, regular EELS quantification routines using background subtraction and peak integrals would likely fail due to unreliable extraction of individual signals. In this study, MLLS fitting was implemented to achieve reliable separation of the Sb M4,5 edge from the In M4,5 edge. The Sb and In reference spectra were taken from the AISb barrier layer and from the InAs capping layer, respectively. Thus, the reference and target spectra were acquired from the same TEM specimen with identical acquisition parameters under similar chemical environment (same valence state). Zero-loss spectra were also recorded to calculate the relative sample thickness (in units of k, the mean free path for inelastic scattering) in order to select suitably thin regions of the TEM specimens, so that both two-dimensional spectrum images and reference spectra could be acquired from relatively thin regions with similar thicknesses to make allowance for any plural scattering.

A representative STEM-EELS analysis is summarized in Fig. 4. The EELS spectrum image was recorded from the region indicated by the solid blue rectangle in Fig. 4(a), and a closely adjacent region, indicated by the dashed yellow square, was used for drift correction. The core-loss spectrum image was acquired with both In and Sb M4,5 edges included to enable MLLS fitting. The relative thickness map shown in Fig. 4(d) indicated that the thickness in this region was relatively uniform and considerably less than one inelastic mean free path (average thickness of 0.7 k), similar to the thickness of the regions where the reference spectra were acquired (not shown here). Under this well-controlled acquisition condition, the ratio of MLLS fitting coefficients for the corresponding Sb/In 2D map in Fig. 4(b) and the extracted averaged line profile in Fig. 4(c) (averaged over two pixels) give the projected areal density of Sb compared with In in the scanned region. Thus, since the In content is presumed to be constant throughout the growth of the InAs/InAs1-xSbx SL stack, the Sb composition can be extracted. Note that the Sb composition is the average for the examined region projected along the electron beam direction.

Closer examination of Fig. 4(c), which is the averaged projected line profile of Sb composition, confirmed the asymmetric interfaces in the superlattices, as previously indicated by GPA analysis. The InAsSb-on-InAs interface was relatively sharp, while the graded InAs-on-InAsSb interface demonstrated a typical segregational feature of an exponential-like tail with Sb atoms migrating from the InAsSb layer into the following InAs layer. The maximum Sb composition in the InAsSb layers here is about 35%. 

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A typical 002 DF image of the superlattices is shown in Fig. 5(a), while Fig. 5(b) shows the averaged Sb composition profile extracted from the region marked by the white dashed rectangle (averaged over 30 pixels, which corresponds to about 4 nm). The averaged intensity line profile was initially calibrated using a characteristic dark-line feature (minimum-intensity line) at the AlSb/InAsSb interface. Quantitative chemical determination was then carried out by analyzing the (002) diffracted intensity assuming the kinematical scattering approximation (with atomic-scattering factors adapted from Doyle and Turner). The influence of electron redistribution due to the bonding of atoms, local structural distortions, and thin-foil surface relaxation is not considered here. Note that the layer contrast in the 002 DF images is not the same as for the HAADF images used in GPA analysis, i.e., the InAsSb layers appear darker than the InAs layers because of different contrast mechanisms for the two techniques. The maximum Sb content of about 35% estimated by 002 DF analysis is in remarkable agreement with the value obtained from EELS. Furthermore, the presence of asymmetric Sb profiles on either side of the InAsSb layers is confirmed, in agreement with the information revealed previously by GPA and EELS analysis. The 002 DF imaging results corroborate that the InAsSb-on-InAs interface is relatively sharp, while the InAs-on-InAsSb interface is graded and resembles a typical segregation profile with a decreasing exponential-like tail penetrating into the next InAs layer.

The main challenge associated with implementation of 002 DF imaging is to tilt the specimen with the 002 beam at or close to the Bragg condition while imaging the interface closely edge-on. We estimate a small tilting offset of the interface from the exact edge-on condition, which would broaden the projected interface by 0.5–1 monolayer (ML) for specimen thicknesses of 50–100 nm. Such broadening is negligible provided that careful choices of specimen/beam tilting and specimen thickness are made, and is much smaller than the interface width measured here, as discussed in Sec. III D. For other MBE-grown III-V heterostructures, the typical interface width (10%–90% criterion) ranges between 4.4 and 7.5 ML. In addition, although this imaging method is aperture-limited, it has been shown elsewhere that detailed analysis of the shape of the composition profiles, supported by structural modeling, allows quantification of the chemical interface, and variations in interface widths and layer thicknesses as small as 0.1 ML can be measured.

D. Evaluation of antimony segregation

This section investigates in more detail the asymmetric interfaces revealed by the three independent (S)TEM techniques. The graded/broadened InAs-on-InAsSb interface demonstrated an exponential-like descending tail characteristic of surface segregation. Thus, the Sb composition profiles across this interface obtained from 002 DF and EELS measurements were evaluated using Muraki’s phenomenological segregation model, which initially assumes a step-like interface, as summarized by.
between the Sb composition profiles and the segregation fitting at this interface. This is unlikely to be due to Sb segregation; rather, this non-ideal interface is due to an intrinsic minimum interface width that is dictated by the molecules–surface interaction potential during growth, and can be approximated with a set of sigmoidal functions, as represented by Equation (3). For layers centered at \( z = 0 \), the sigmoidal expressions are

\[
x = \frac{x_0(l)}{1 + e^{\frac{z - x_0}{L_{lower}}}}, \quad \text{for } z < 0;
\]

\[
x = \frac{x_0(u) - x_0(l)}{1 + e^{\frac{z - x_0}{L_{upper}}}}, \quad \text{for } z > 0,
\]

where \( x_0(l) \) and \( L_{lower} \), \( x_0(u) \) and \( L_{upper} \), denote the composition and the intrinsic interface width at the lower \( (l) \) and upper \( (u) \) interfaces, respectively, and \( N \) is the layer thickness. This sigmoidal-type interface exists naturally at both interfaces in this T2SL system. However, the segregation effect across the InAs-on-InAsSb interface is so strong that it dominates over the intrinsic sigmoidal profile (Figs. 6(b) and 6(d)), and the Sb composition evolution can be reasonably reproduced using only the segregation model (Figs. 6(a) and 6(c)). Hence, the Sb composition profile across a three-layer structure of InAs/InAsSb/InAs was better described using a piece-wise function, using the sigmoidal model for the InAsSb-on-InAs interface, and Muraki’s segregation model for the InAs-on-InAsSb interface. The connecting point of the two models is marked with an arrow, as indicated in Fig. 7 for profiles obtained from 002 DF imaging and EELS analysis. Experimental Sb composition profiles obtained from these

FIG. 6. Experimental Sb composition profiles and corresponding segregation and sigmoidal fitting curves obtained from (a) and (b) 002 DF imaging; and (c) and (d) STEM EELS.

FIG. 7. Experimental Sb composition profiles and corresponding fitting curves using combination of sigmoidal function for InAsSb-on-InAs interface and Muraki’s segregation model for InAs-on-InAsSb interface, as obtained from (a) 002 DF imaging; and (b) STEM EELS. Arrows indicate connection points of the fitting curves.

\[x = x_0(1 - R^z), \quad \text{for } 0 \leq z \leq N;
\]

\[x = x_0(1 - R^N) \cdot R^{z-N}, \quad \text{for } z \geq N.\]
two independent microscopy techniques were very well reproduced using the combined models with the same fitting parameters. In addition to the segregation probability of 81%, an intrinsic interface width of 1.2 ML was obtained for the InAsSb-on-InAs interfaces, which is relatively small compared to the broad InAs-on-InAsSb interface, confirming segregation as the dominant mechanism.

Thus, evaluation of the Sb composition profiles that has been obtained from these two independent microscopy techniques, with the aid of segregation and sigmoidal models, demonstrates remarkable agreement: both yield Sb segregation probability of 81% and a segregation broadening length of about 3 nm. This significantly modified composition profile which in turn alters the effective superlattice layer thickness and period, and related strain engineering, thus would induce significant deviations in the energy-band structure and the optoelectronic response.

**IV. CONCLUSIONS**

Electron microscopy characterization techniques with high spatial resolution have been used to provide insight into probable performance-limiting factors of state-of-the-art InAs/InAsSb superlattices that are largely free of extended defects. Three separate and independent studies, using GPA strain analysis, STEM EELS, and 002 DF imaging, showed that superlattices grown without any special incorporation in the InAsSb layers, which would induce blue shift of the optical response; (c) effectively thicker InAsSb layers due to segregated Sb, which would reduce the overlap of electron and hole wavefunctions and thus lead to weakened absorption; (d) smaller maximum compressive strain in the InAsSb layers and reduced tensile strain in the first half of each InAs layer, which would mean reduced effect of strain on band-gap engineering. All of these effects must be taken into account during structure design to avoid any undesirable or even detrimental impact on device performance.

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