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Structural Analysis of Highly Relaxed GaSb Grown on GaAs Substrates with Periodic Interfacial Array of 90° Misfit Dislocations

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Abstract We report structural analysis of completely relaxed GaSb epitaxial layers deposited monolithically on GaAs substrates using interfacial misfit (IMF) array growth mode. Unlike the traditional tetragonal distortion approach, strain due to the lattice mismatch is spontaneously relieved at the heterointerface in this growth. The complete and instantaneous strain relief at the GaSb/GaAs interface is achieved by the formation of a two-dimensional Lomer dislocation network comprising of pure-edge (90°) dislocations along both [110] and [1-10]. In the present analysis, structural properties of GaSb deposited using both IMF and non-IMF growths are compared. Moiré fringe patterns along with X-ray diffraction measure the long-range uniformity and strain relaxation of the IMF samples. The proof for the existence of the IMF array and low threading dislocation density is provided with the help of transmission electron micrographs for the GaSb epitaxial layer. Our results indicate that the IMF-grown GaSb is completely (98.5%) relaxed with very low density of threading dislocations (10^5 cm^{-2}), while GaSb deposited using non-IMF growth is compressively strained and has a higher average density of threading dislocations (>10^9 cm^{-2}).

Keywords Semiconductor · GaSb/GaAs · Molecular beam epitaxy · Interfacial misfit dislocations (IMF) or Lomer dislocations · Strain relief · Structural properties · Moiré fringes

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Introduction

Antimonide semiconductors have potential application in a wide range of electronic and opto-electronic devices due to their unique band-structure alignments, and small effective mass as well as high mobility for electrons [1–4]. While recent technical advancements have enabled high quality lattice matched GaSb epitaxy on native substrates, for many applications GaAs substrates are desirable. This is because of the following reasons: GaAs is inexpensive, has favorable thermal properties, transparent to more (long wave length) active regions, forms excellent n and p ohmic contacts, and can be semi-insulating compared to GaSb. However, the high (7.8%) lattice mismatch between the GaSb epilayer and the GaAs substrate complicates the growth of sophisticated device structures. Currently, this mismatch is accommodated via metamorphic buffer layers [5] and strain-relief superlattices [6]. In metamorphic buffer layer approach, initially the strain within the critical thickness is accommodated by tetragonal distortion followed by defect formation and filtering. While this approach has enabled a number of device demonstrations [7], it exhibits several deficiencies such as the necessity to grow thick buffer layers (often >1 μm), poor thermal and

electrical conductivity, and has resulted in significant material degradation through the presence of threading dislocations (TDs).

Recently, a fundamentally different growth mode, interfacial misfit dislocation (IMF) growth mode, has been developed by our group [8, 9]. In this growth, the strain is relieved instantaneously at the mismatched heterointerface unlike the traditional tetragonal distortion approach that relieves the strain after reaching a critical thickness. The IMF growth offers a "buffer-free" approach to realize monolithic high quality GaSb deposited on GaAs substrate with exceptionally low threading dislocation (TD) densities ($\sim 10^5$ cm^{-2}), despite the high lattice mismatch. The strain created due to the 7.8% lattice mismatch is relieved at the GaSb/GaAs interface by the formation of a two-dimensional (2D), periodic IMF arrays comprised of pure-edge (90°) dislocations along both [110] and [1-10]. To facilitate the growth of "buffer-free" deposition of GaSb on GaAs substrate with low TD densities, in complex device structures, it is essential to understand the structural properties of IMF-grown GaSb epitaxial layers.

An attempt was made previously to show the proof of existence of the IMF array at the GaSb/GaAs interface along [1-10] using cross-sectional transmission electron micrograph (XTEM) and to calculate the TD density using KOH etching as shown in Ref. [10]. However, the XTEM images look only at one-dimensional sections and hence are not representative of the 2D interface. Also, the quantitative analyses like strain relaxation of bulk GaSb deposited on GaAs substrates, long-range uniformity of the IMF array in 2D, and accurate TD density calculation for GaSb that was not presented earlier, are very important in realizing high quality GaSb bulk layers on GaAs substrate. In this study, all the issues addressed earlier, namely the material quality of the GaSb epitaxial layer is quantified using various analyses like XTEM, selective area electron diffraction (SAED) double spot pattern, moiré fringe patterns, X-ray diffraction (XRD), and plan-view TEM.

Experiments

The samples are grown on GaAs substrates in a VG V80H molecular beam epitaxy (MBE) reactor equipped with valved crackers for As and Sb, and an optical pyrometer for monitoring the substrate temperature. Various samples comprising GaSb bulk layers are grown on GaAs substrates, using IMF growth. The details of the IMF growth are presented elsewhere [10]. The thickness of the IMF-grown GaSb epitaxial layers used for various analyses range from 15 nm to 5 μm. For example, thick samples like 5, 0.5 μm are used for XRD analyses, and samples with medium thickness, like 120 nm, are used for XTEM and SAED analyses, respectively. For TD density analysis using plan-view TEMs, the sample is lapped down from 5-μm GaSb epitaxial layer to 45 nm. Very thin 15-nm sample is grown separately for moiré fringe analysis to facilitate the transmission of electrons through both the epitaxial layer and the underlying substrate. The sample required for moiré fringe analysis is prepared as follows, the substrate is lapped down to ~ 10 μm and ion milled to 30 nm, resulting in a net thickness of 45 nm that includes the 15-nm IMF-grown GaSb epitaxial layer. Another set of GaSb bulk samples, which are similar to those of the IMF samples are deposited using non-IMF growth on GaAs substrate for comparison with the former in various analyses as mentioned earlier. If the interface is As-rich instead of Ga-rich prior to the deposition of GaSb, no IMF is observed at the heterointerface and this growth mode is called non-IMF growth mode. Non-IMF growth is also similar to that of the IMF growth up to the deposition of GaAs smoothing layer. After the smoothing layer, Ga source is turned off and the As-overpressure is on while bringing the temperature down to 510 °C from 560 °C. When the substrate temperature is 510 °C, the resulting surface is As-rich. At this point, both Ga and Sb sources are turned on. In this case, IMF is not formed at the interface as is explained in the following paragraphs.

Results and Discussion

Figure 1 shows the high-resolution TEM (HR-TEM) image of the GaSb/GaAs interface. The Burgers circuit completed around each misfit indicates a pure-edge dislocation along [1-10]. One of such misfit dislocations are shown in Fig. 1 as a bright spot representing the IMF dislocation. Similar type of burgers vectors are observed along [110] as well. Hence the dislocation network associated with the IMF array formation along both [110] and [1-10] is characterized as a 2D Lomer dislocation network. In general, relaxation kinetics favors the formation of 60° dislocations over 90° dislocations as the former dislocation can glide to the surface from the interface. However, the latter is more preferable as it is more efficient in relieving the strain compared to the 60° dislocations and can be formed under favorable conditions as shown in Fig. 1.

Figure 2a shows the bright-field XTEM image of a 120-nm TD free IMF-grown GaSb epitaxial layer on a GaAs substrate along zone axis [110]. The IMF is seen as dark spots in this figure with a periodicity of 5.6 nm. This periodicity corresponds to exactly one misfit dislocation for every 14 lattice sites of GaAs or 13 lattice sites of GaSb. This value is in good agreement with the theoretical periodicity for a relaxed GaSb deposited on GaAs [8]. The strain created by the lattice mismatch is relieved

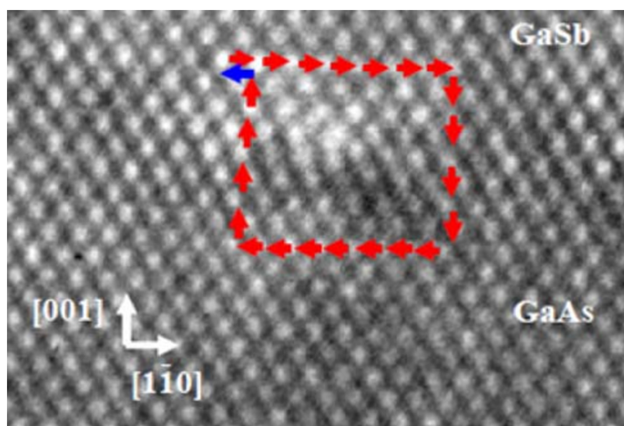

Fig. 1 Burgers circuit completed around one misfit dislocation of the IMF array at the GaSb/GaAs interface shown with the help of HR-TEM image, where the dislocation is shown as a *bright spot*

spontaneously by the formation of the IMF at the GaSb/GaAs interface. Further proof of spontaneous relaxation of IMF-based samples is provided via the SAED double spot pattern as shown in Fig. 2b, which is imaged along zone axis [110]. The highly resolved diffraction spots in SAED demonstrate two separate lattice constants associated with GaAs ($a_s = 5.65$ Å) and GaSb ($a_f = 6.09$ Å), respectively. The alignment of the 000 diffraction spot with, for instance, the two 220 spots indicates that there is no lattice rotation. In the IMF growth, a sheet of Sb atoms are deposited on Ga-rich GaAs surface before starting the growth of bulk GaSb epitaxial layer. If Sb is deposited on As-rich GaAs surface instead of Ga-rich GaAs surface, the resulting epitaxial layer will have high defect density as shown in the bright-field XTEM of Fig. 2c, which is imaged along [110] for non-IMF grown GaSb sample.

The ω-2θ scan of symmetric (004) XRD spectra for a 0.5- μm thick GaSb epitaxial layers deposited using IMF and non-IMF growths, and 5- μm thick sample deposited using IMF growth are shown in Fig. 3a, b, respectively. In addition to the broad full width at half maximum (FWHM), the non-IMF spectrum differs to the IMF spectrum due to the presence of additional peak near the GaAs substrate as shown in Fig. 3a. This additional peak in the non-IMF sample is attributed to the tetragonally distorted GaSb. This means that initially the in-plane lattice constant of the epitaxial layer and of the substrate are equal up to critical thickness, after which the epitaxial layer slowly relaxes to the original lattice constant of GaSb by relieving the strain via the formation of misfit and often threading dislocations. In non-IMF spectrum, this transition of lattice constant is represented by a negative slope via the transition from additional peak to the epi-peak. Similar type of behavior was not observed in the IMF samples, and hence no tetragonal distortion is attributed to the IMF-grown GaSb epitaxial layers. The relaxation of the IMF-grown GaSb epitaxial layer is determined from the analysis of XRD. The calculation based on the symmetric (004) and asymmetric (115) XRD measurements show approximately 98.5% (complete) relaxation of the GaSb epitaxial layer, and similar type of relaxation is observed in GaSb grown on GaAs with AlSb nucleation layer [11]. We believe that the broad FWHM (194 arcsecs) of GaSb layers, thinner than 1 μm, as shown in Fig. 3a is due to the small amount of residual strain (<2%) in the epitaxial layers after the creation of the IMF array [10]. As per our observations, with thicker layers (5 μm) the FWHM decreases considerably to $\sim$20 arcsecs in IMF-grown GaSb epitaxial layers as shown in Fig. 3b.

Figure 4a, b show the bright-field plan-view TEMs imaged along zone axis [001] for the center and edge of the IMF sample, respectively. The average TD density was calculated to be 10^5 cm^{-2} from the plan-view TEMs. Even though, no TDs are observed at the center, very few TDs are observed at the edge of the IMF sample and are attributed to the un-optimized IMF growth at sample edges. Using the plan-view TEM images, the dislocation density

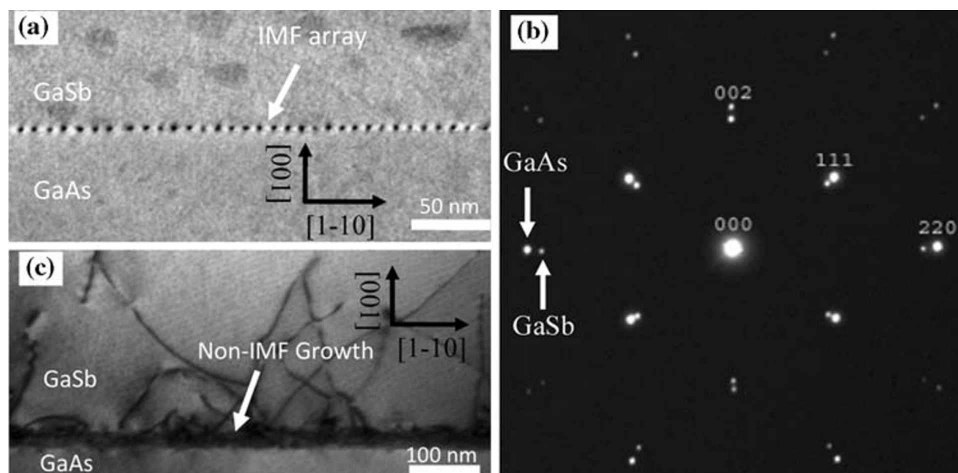

Fig. 2 **a** XTEM showing a periodic IMF array with a periodicity of 5.6 nm, as *dark spots*, at the GaSb/GaAs interface **b** SAED double diffraction pattern of IMF growth mode, and **c** XTEM of non-IMF growth mode with high threading dislocation density compared to the IMF growth mode

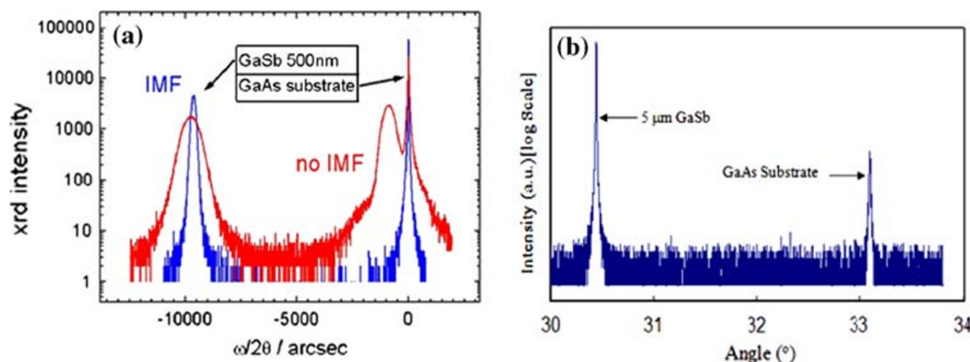

Fig. 3 XRD (004) scan of **a** 0.5 μm GaSb on GaAs substrate grown using IMF and non-IMF growth mode, illustrating highly relaxed GaSb for the IMF growth, and **b** 5 μm GaSb on GaAs substrate showing a narrow FWHM of ~20 arcsecs for the GaSb epitaxial layer

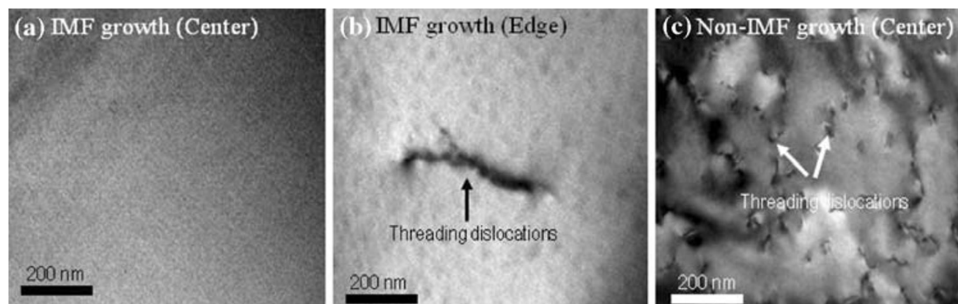

Fig. 4 Plan-view TEM showing TDs from **a** center, **b** edge of the IMF sample, and **c** center of the non-IMF sample for a 5 μm GaSb epilayer on a GaAs substrate

has been calculated based on the number of dislocations within the unit area from several wafer surfaces. In the non-IMF grown GaSb layers, TD density is measured to be $\sim 10^{9}$ cm^{-2} as shown in bright-field plan-view TEM shown in Fig. 4c, which is imaged along zone axis [001]. This confirms the fact that the TD density is reduced in the IMF growth compared to the non-IMF growth due to spontaneous strain relaxation. Also no 60° dislocations were observed in IMF-grown GaSb, which indicates that the IMF dislocations are non-interacting and pure-edge (90°) 2D arrays. Since the 90° dislocations can relieve strain almost completely at the interface, high quality "buffer-free" GaSb epilayers can be deposited monolithically on GaAs substrates in the IMF growth.

Figure 5a, b shows the two-beam bright-field plan-view TEM **g**.3**g** [**g** = (220) and (2-20)] obtained from GaSb epitaxial layers deposited on GaAs substrates using the IMF growth. These TEMs show moiré fringe patterns, which are the interference patterns that are formed when two crystals with different orientations or lattice constants overlap, thus providing an excellent indication of whether the epitaxial layer is strained. Moiré fringes image the projection of dislocations instead of the dislocations themselves. The moiré fringes shown here are translational moiré fringes as the planes and thereby **g** vectors are parallel to each other. Moiré fringe spacing, which is defined as the spacing between two consecutive white or dark lines is measured to be 2.8 nm from Fig. 5a, b. The theoretical spacing for translational moiré fringes is given by: $D_{\mathrm{tm}} = \left(\frac{1}{d_{\mathrm{GaSb}}} - \frac{1}{d_{\mathrm{GaAs}}}\right)^{-1}$, where d is the inter-planar spacing assuming that $d_{\mathrm{GaSb}} = 2.155$ nm and $d_{\mathrm{GaAs}} = 0.1999$ nm for {220} reflections and is calculated to be 2.75 nm. The measured value of 2.8 nm is in good agreement with the theoretical spacing, which again indicates that the film is fully relaxed.

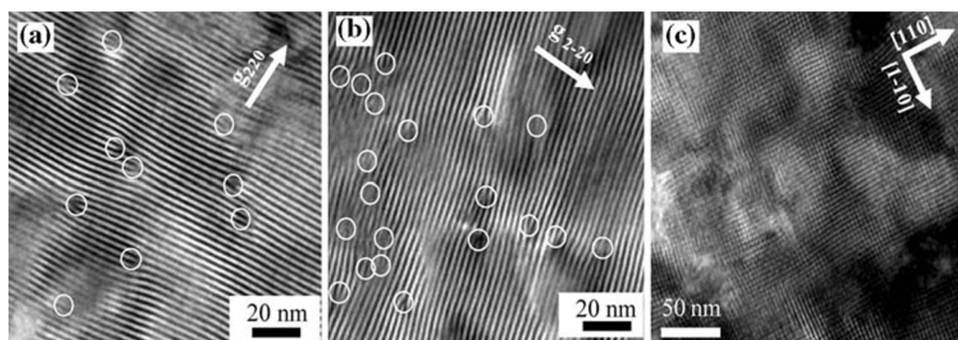

Fig. 5 Plan-view TEMs showing moiré fringes of 2D IMF arrays along **a** [110] **b** [1-10], and **c** 2D Lomer dislocation network along both [110] and [1-10] measured using diffraction vectors (220), (2-20), and both (220) and (2-20), respectively. Consecutive *white* and *dark lines* represent moiré fringes, and the *white circles* represent the edge dislocations

Moiré fringes are often used to identify dislocations in semiconductors [12–14] as well as metals [15]. The terminating half lines (THLs) shown in Fig. 5a, b, indicated by white circles illustrate the projection of pure-edge dislocations and are similar to the observations made by other groups in various material systems [13, 15]. The pure-edge dislocation density from various areas of the moiré fringes averages to 6.62×10^{10} cm^{-2}. The THLs in the moiré fringes might also represent TDs as shown in Ref. [16]. The TDs revealed in this way are attributed to the half-period shifts in the moiré fringes, which are produced as a result of the interaction between 60° and 90° dislocations. However, no half-period shifts are observed in the moiré fringes of IMF-grown GaSb samples as shown in Fig. 5a, b. Moreover, no 60° dislocations are observed in the IMF sample, which are considered to be the main source for the formation of TD when the former interacts with the 90° dislocations. Generally, distortions local to the interface, such as stacking faults are revealed as displacements in moiré fringes. In this study, displacement of the moiré fringes is not observed in the IMF samples, hence stacking faults or partial dislocations are not ascribed to the IMF growth. The moiré fringes are imaged along both [110] and [1-10] using (220) and (2-20) **g** vectors as shown in Fig. 5c. The projection of 2D Lomer dislocation network is observed to be uniform over a large area that was imaged (0.72 μm^2).

Conclusions

In conclusion, high quality "buffer-free" GaSb is grown on GaAs substrates with very low TD densities ($\sim 10^5$ cm^{-2}) despite the high (7.8%) lattice mismatch. The strain due to lattice mismatch is relieved immediately at the GaSb/GaAs heterointerface with the help of periodic, pure-edge misfit (IMF) arrays of dislocations along both [110] and [1-10] in the IMF-grown GaSb. Instead, if the GaSb is deposited using a non-IMF growth, the resulting epitaxial layer has very high TD density (10^9 cm^{-2}) due to buildup of strain in tetragonal distortion. Comparing the IMF and non-IMF samples using XRD and XTEM analyses have shown that the strain is completely (98.5%) relieved in IMF sample, whereas it is not the case for non-IMF sample. The plan-view TEM analysis for both samples also confirmed similar results, where the TD density is very low for IMF sample ($\sim 10^5$ cm^{-2}) compared to non-IMF sample ($\sim 10^9$ cm^{-2}). The long-range uniformity and the strain relief of the IMF-grown GaSb epitaxial layer measured using the moiré fringe patterns have shown a uniform 2D Lomer dislocation network over the entire scan area. The moiré fringe spacing of 2.8 nm agrees well with the theoretical spacing of 2.75 nm, which proves that the GaSb layer is completely relaxed. Further proof of strain is also achieved from SAED measurements, which shows that GaSb and GaAs has lattice constants almost similar to the expected lattice constants of the corresponding relaxed materials. We believe that this approach is useful for the deposition of "buffer-free" high quality GaSb on well-studied GaAs substrates in complex device structures.

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