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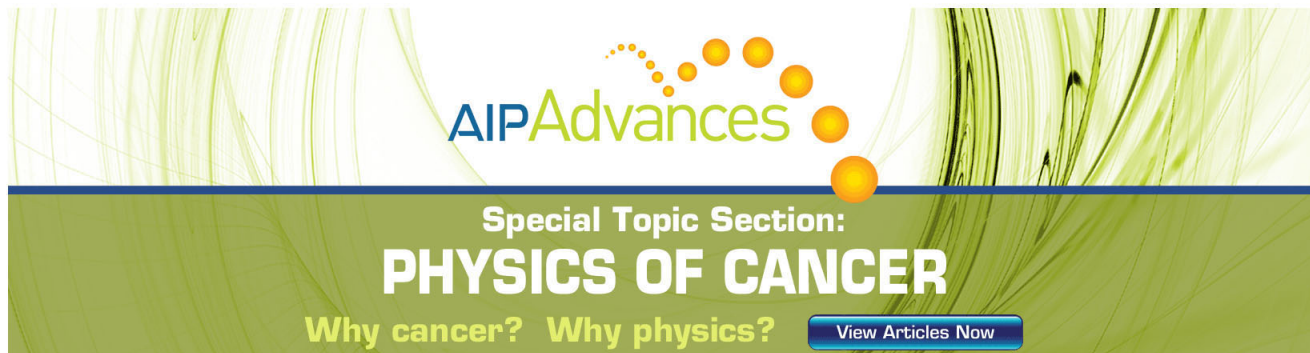
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## Relaxation dynamics and residual strain in metamorphic AlSb on GaAs

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We have observed the evolution of the accumulated stress during heteroepitaxial growth of highly lattice mismatched AlSb on GaAs by measuring the deformation of the substrate as a function of time. High resolution transmission electron microscopy images show almost all of the plastic relaxation is accommodated by an array of 90° misfit dislocations at the interface. The in-plane lattice parameter of the resulting metamorphic AlSb is slightly smaller (0.3%) than the bulk value and perfectly matches the lattice parameter of bulk GaSb. It is, therefore, possible to grow nearly stress-free GaSb on GaAs using a metamorphic AlSb buffer layer. © 2012 American Institute of Physics. [doi:10.1063/1.3674986]

The growth of metamorphic III-V semiconductors on substrates of dissimilar lattice parameter is of interest for the engineering of optoelectronic devices, particularly, for those in which substrate availability would otherwise limit the applications of the material. Such is the case of the antimonides, which are very appropriate for small band gap photovoltaics or thermophotovoltaics. To increase the efficiency of tandem solar cells (typically based on Ge or GaAs substrates), it is necessary to integrate semiconductors with band gap <1.4 eV. Metamorphic InGaAs has been integrated in GaAs based tandems, but requires the growth of thick step graded buffers of InGaP.<sup>1</sup> Here, we present a study on the nucleation and subsequent growth of AlSb on GaAs (001) and compare our results with the more extensively studied case of GaSb on GaAs (Refs. 2 and 3) and the case of AlSb grown on Si.<sup>4,5</sup> There is a twofold motivation to study the metamorphic growth of AlSb on GaAs rather than the more commonly studied case of GaSb on GaAs. First, the use of high band gap materials, such as AlSb, for the metamorphic interface is advantageous for integration in solar cells, where the separation of minority carriers away from defects is of utmost importance. Second, metamorphic layers are most often not 100% free of strain, but as AlSb has a slightly larger lattice parameter than GaSb (6.1355 Å vs 6.0959 Å, 0.6%), it would, in principle, be possible to grow strain free GaSb on top of metamorphic AlSb if a small compressive strain remains in the AlSb. Metamorphic AlSb is also being actively studied for the fabrication of high electron mobility transistors<sup>6–8</sup> and mid infrared laser sources.<sup>9</sup>

The sample was epitaxially grown on GaAs (001) by solid source molecular beam epitaxy. After oxide desorption and buffer layer growth, the sample was exposed to Sb at a substrate temperature  $T_s$  of 600 °C until a  $8 \times$  periodicity was observed in the reflection high energy electron diffraction (RHEED) pattern in the  $[1\bar{1}0]$  azimuth, this being indicative of a Sb terminated GaAs surface.<sup>2</sup> The substrate

temperature was then lowered to 500 °C and AlSb growth was started at 0.25 ML/s in Sb rich conditions ( $2 \times 10^{-6}$  mbar Sb beam equivalent pressure). The evolution of the accumulated stress during epitaxial growth was monitored *in situ* by a multi-beam optical stress sensor (MOSS).<sup>10</sup> The substrate dimensions were  $20 \times 4 \times 0.1$  mm to maximize the deflection of the substrate due to the accumulated stress, with the longest side along the  $[110]$  direction. The deflection is measured by comparing the angle of reflection of two laser beams: one of them incident on the end that is firmly held to the substrate holder and the other incident on the free standing end of the substrate.

Transmission electron microscopy (TEM) characterization was carried out on JEOL 2100 and 2000FX microscopes, both operating at 200 kV.

The evolution of the RHEED pattern in the  $[1\bar{1}0]$  azimuth during AlSb epitaxy on GaAs (001) is shown in Fig. 1. The  $(2 \times 8)$  reconstruction used as the initial surface for AlSb nucleation is shown in Fig. 1(a). The RHEED pattern after deposition of 0.11 ML of AlSb on GaAs shows a spot pattern characteristic of transmission electron diffraction through the tip of 3D islands. Hao *et al.* have shown TEM evidence of metamorphic AlSb islands on GaAs in agreement with our observation of a RHEED pattern typical of island growth mode.<sup>11</sup> RHEED measurements indicate a change to a lattice parameter close to that of bulk AlSb within the first ML of AlSb deposition. Such rapid change is due to the grazing incidence of the electron beam, thus the measured lattice parameter is representative of the tips of the AlSb islands which are not laterally constrained until after island coalescence. In the related case of GaSb on GaAs, Huang *et al.* have observed the coexistence of a pseudomorphic (elastically strained) wetting layer and plastically relaxed metamorphic quantum dots.<sup>2</sup>

A TEM overview of the AlSb/GaAs interface is shown in Fig. 2. The image was acquired in 220 bright field conditions, and the incident electron beam is at a 30° angle relative to the plane of the interface. Most of the lattice

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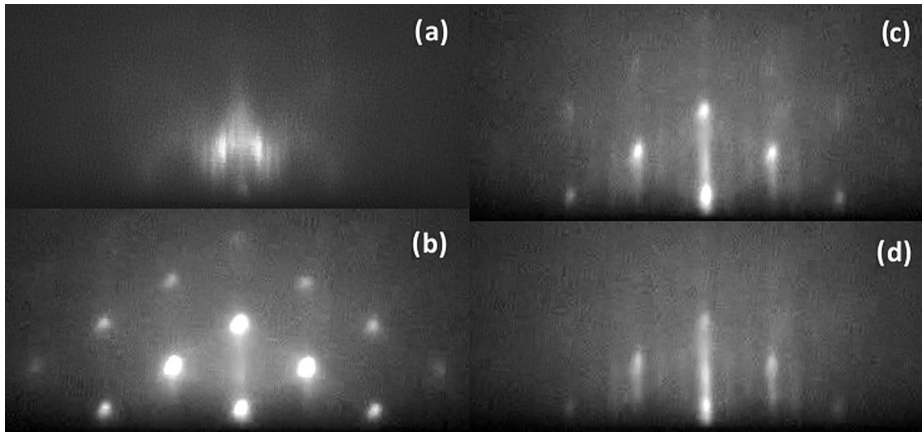


FIG. 1. RHEED pattern in the  $[1\bar{1}0]$  azimuth at various stages of AlSb on GaAs (001) epitaxy. (a) Exposure of GaAs to antimony at  $600^\circ\text{C}$  resulting in a  $(2 \times 8)$  reconstruction. (b) Spot pattern indicative of 3D island nucleation. (c) Initial stage of island coalescence after deposition of 20 ML. (d) Pattern indicative of island coalescence and progress towards surface planarization after 40 ML of AlSb on GaAs.

mismatch is relaxed by  $90^\circ$  dislocations at the interface. The presence of planar defects and  $60^\circ$  dislocations is also apparent in the TEM images. These can be a significant obstacle for device performance, but it has been shown that planar defects in metamorphic AlSb on GaAs can be suppressed with AlSb/GaSb super lattices.<sup>11</sup>

High resolution TEM (HRTEM) micrographs taken in a  $[110]$  projection also show a high density of interfacial misfit (IMF) dislocations (Fig. 3). Extra half planes can easily be observed at the AlSb/GaAs interface. A Burgers circuit analysis revealed that the lattice mismatch is relaxed mainly by  $90^\circ$  dislocations with a small fraction of  $60^\circ$  misfit dislocations. Figure 3(b) shows a map of the measured lattice parameter parallel to the interface, calculated from the lattice distortion using geometric phase methodology.<sup>12</sup> The plot in Fig. 3(c) shows the average in-plane lattice parameter along the direction perpendicular to the interface in the rectangular area indicated in Fig. 4(b). The average in-plane AlSb lattice parameter was found to be  $6.08 \pm 0.03 \text{ \AA}$ , slightly smaller than the lattice parameter of the bulk AlSb ( $6.1355 \text{ \AA}$ ). Thus, only  $89\% \pm 5\%$  of the lattice mismatch is plastically relaxed, and the growing AlSb is under compressive stress.

The accumulated stress in the epitaxial layer has been obtained from the measured substrate curvature using the following relation:

$$\frac{-Mh^2}{6R} = \Sigma\sigma + \Delta\tau, \quad (1)$$

where  $R$  is the curvature radius of the substrate,  $h$  is the substrate thickness,  $\Sigma\sigma$  is the accumulated stress in the epitaxial layer,  $M$  is the biaxial Young's modulus of the substrate, and  $\Delta\tau$  is the surface tension of the substrate back surface minus the surface tension of the epitaxial front surface. In the case

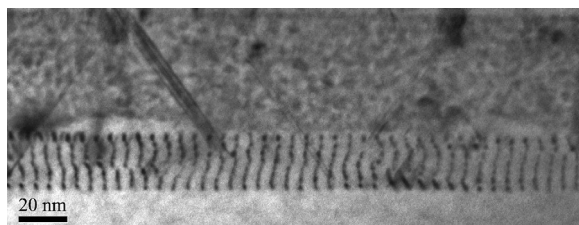


FIG. 2. Bright field 220 TEM image of the misfit dislocation array at the AlSb/GaAs interface.

of (001) substrates,  $M = Y/(1 - \nu)$ , where  $Y$  is Young's modulus and  $\nu$  is the Poisson ratio of the substrate.

The evolution of the accumulated stress during epitaxial growth is shown in Fig. 4 in comparison with the expected accumulated stress curves for pseudomorphic growth and for metamorphic growth with 94.1% plastic relaxation after 2 ML of pseudomorphic growth, the latter resulting from a linear fit to the experimental data for coverages larger than 40 ML.

The plastic/elastic relaxation ratio as determined from the MOSS stress sensor is much higher during the first ML of antimonide growth on GaAs. This is in contrast to the RHEED measurements, which seem to indicate a nearly instantaneous and complete relaxation of the lattice parameter. This apparent discrepancy is due to the fact that the RHEED measurement is predominantly probing the relaxed islands sticking out of the surface, whereas the stress measurements represent an average of the whole grown layer. This is consistent with the observation by Huang *et al.* of coexisting regions with and without the IMF array during the initial stages of antimonide growth.<sup>3</sup> The TEM shows clear evidence of the IMF both in planar view and cross section, thus the results presented here are probably also relevant to other

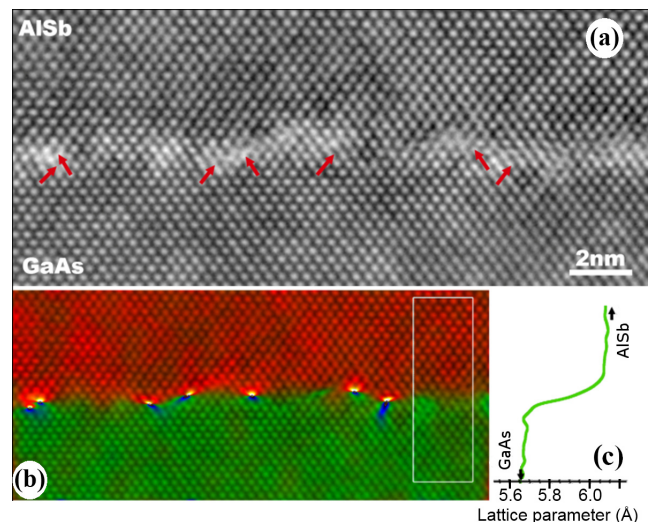


FIG. 3. (Color online) (a) High resolution TEM detail of the IMF array at the AlSb/GaAs interface. (b) Map of the lattice distortion using geometric phase methodology. (c) Average in plane lattice parameter for the rectangular region highlighted in (b).

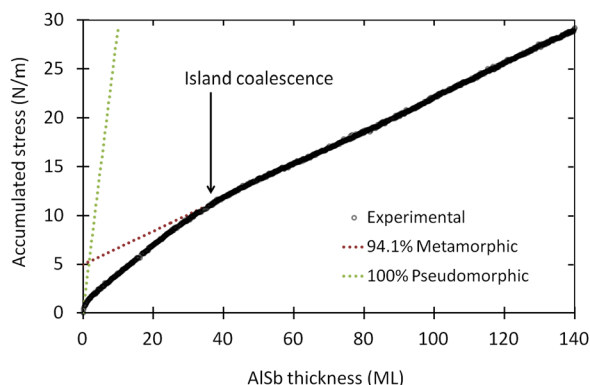


FIG. 4. (Color online) Accumulated stress as measured from the substrate deflection during growth of AISb on GaAs.

cases of IMF metamorphic growth, such as GaAs on Si,<sup>13</sup> GaSb on GaAs,<sup>3</sup> and AISb on Si.<sup>4</sup>

During the first 0.76 ML of AISb on GaAs growth, the accumulated stress slope ( $0.59 \pm 0.03$  N/m in the first 0.76 ML) corresponds approximately to the value expected for pseudomorphic growth. But changes in surface composition and reconstruction induced by Sb adsorption on GaAs may also contribute to changes in the measured substrate curvature due a reduction in surface tension, as can be seen in Eq. (1). Such changes of surface reconstruction and composition have been shown to induce surface tension changes of up to 1 N/m.<sup>14</sup>

After the first 0.8 ML, the slope rapidly decreases, suggesting the onset of stress relaxation (in this case the nucleation of the IMF dislocation array). The slope of the stress accumulation curve for coverages ranging from 10 to 20 ML is  $0.29 \pm 0.02$  N/m/per ML, approximately 10% of the value expected for pseudomorphic growth. The two contributions reducing the elastic strain are plastic relaxation and surface roughness.

A roughening of the surface can lead to a significant reduction of the slope of the accumulated stress curve, as in the case of quantum dot nucleation.<sup>10</sup> Once the growth front reaches a planar, stationary state, the changes in the slope of the accumulated stress curve are due to plastic relaxation processes. The accumulated stress corresponding to the elastic strain deduced from HRTEM measurements 10 ML above the interface is  $6 \pm 0.3$  N/m, while the accumulated stress measured in real time during growth after 10 ML of AISb deposition on GaAs is  $4.0 \pm 0.2$  N/m. This apparent discrepancy is in fact to be expected, as after 10 ML of AISb deposition island coalescence is still incomplete, and the growth front is still not planar, with the AISb at the tip of the islands barely contributing to the accumulated stress. As the islands grow, these become laterally constrained as they coalesce, increasing their contribution to the accumulated stress. An example of this phenomenon was reported by Massies and Grandjean, who observed, by RHEED, oscillations in the lattice parameter of InGaAs/GaAs 2D islands corresponding to the period of monolayer completion.<sup>15</sup> As the islands coalesce, their lattice parameter decreases, increasing the accumulated stress. Thus, there are two contributions to the slope of the accumulated stress curve: the one due to the lattice mismatch between substrate and epi-layer, and the

one related to island coalescence, which is in effect due to changes of surface morphology changing the stress.

From 20 to 40 ML, there is a gradual change of slope, coinciding with the observation of island coalescence in the RHEED pattern. This change of slope in the accumulated stress curve is thus interpreted as a signature of the end of the island coalescence process. From 40 to 120 ML, the curve is linear with a slope of 0.173 N/m per ML. This is indicative of a metamorphic AISb lattice parameter of  $6.11 \text{ \AA}$ , below the bulk value of  $6.1355 \text{ \AA}$  but in good agreement with our HRTEM measurement of the metamorphic lattice parameter for AISb on GaAs ( $6.08 \pm 0.03 \text{ \AA}$ ). This lattice parameter is very nearly the same as that of bulk GaSb ( $6.096 \text{ \AA}$ ). It is therefore possible to grow nearly stress free GaSb on GaAs using a metamorphic AISb buffer layer. In fact, Gotoh *et al.* have reported higher hole mobilities for GaSb grown on AISb/GaAs than for GaSb grown directly on GaAs.<sup>16</sup> The AISb layer, having a wider band gap than GaSb, should also act as a barrier for minority carrier recombination at the interface with the substrate.

In summary, the lattice mismatch during heteroepitaxial growth of AISb on GaAs is mostly accommodated by a misfit dislocation array confined to the interface. After deposition of 40 ML of AISb on GaAs, there is a change in the slope of the accumulated stress curve, which we interpret as an indication of the end of the island coalescence process and the consequent lateral constraining of elastically relaxed material at the tip of the islands. This hypothesis is consistent with the changes observed in the RHEED pattern. The slope of the accumulated stress curve after island coalescence is consistent with the in plane lattice parameter of metamorphic AISb as measured by HRTEM ( $6.08 \pm 0.03 \text{ \AA}$ ). This lateral parameter is suitable for the strain free growth of GaSb on GaAs substrates. Our accumulated stress measurements suggest that for this purpose, an AISb buffer layer of at least 40 ML is required to ensure island coalescence, but no more than 140 ML should be used to avoid the accumulation of excessive compressive stress.

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