Compressively-strained GaSb nanowires with core–shell heterostructures

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ABSTRACT

GaSb-based nanowires in a gate-all-around geometry are good candidates for binary p-type transistors, however they require the introduction of compressive strain to enhance the transport properties. Here, we for the first time demonstrate epitaxial GaSb–GaAsSb1−x core–shell nanowires with a compressively strained core. Both axial and hydrostatic strain in GaSb core have been measured by X-ray diffraction (XRD) and Raman scattering, respectively. The optimal sample, almost without plastic relaxation, has an axial strain of −0.88% and a hydrostatic strain of −1.46%, leading to a noticeable effect where the light hole band is calculated to be 33.4 meV above the heavy hole band at the Γ-point. This valence band feature offers more light holes to contribute the transport process, and thus may provide enhanced hole mobility by reducing both the interband scattering and the hole effective mass. Our results show that lattice-mismatched epitaxial core–shell heterostructures of high quality can also be realized in the promising yet demanding GaSb-based system.

KEYWORDS
compressive strain, GaSb-GaAsSb1−x, core–shell, nanowires, heterostructure, p-type transistors

1 Introduction

Metal-oxide-semiconductor field-effect transistors (MOSFETs) based on III-V semiconductor nanowires (NWs) with vertical architecture and gate-all-around geometry have sparked intensive interests [1–5] due to high mobilities and injection velocities in III-V materials [6] and good electrostatics enabling aggressive gate length scaling [7]. Among the III-V binary compounds suitable for MOSFETs, GaSb has been a promising candidate for p-type NW transistors as complementary metal-oxide-semiconductor (CMOS) devices [2, 4, 8] due to its high hole mobility [9–11]. Recently, many successful and important investigations, including growth direction control [12] and reduced doping level [13], have been made to enhance the hole mobility of GaSb NWs. The introduction of compressive strain in GaSb, however, is required to increase the hole transport properties further [14]. In the case of planar transistors, lattice-mismatched GaSb/AlAsSb heterostructures with a biaxial strain of −1% to −3% (negative values denote compressive strains while positive ones denote tensile strains) in GaSb layer have been already demonstrated to increase the mobility by over twice in contrast to that of the bulk [15, 16]. Therefore, epitaxial heterostructures can provide a considerable option to highly strain GaSb NWs as well. However, most reports of lattice-mismatched epitaxy related to GaSb NWs, such as InAs/GaSb [8, 17, 18], and GaAs/GaSb [19, 20], focused on axial heterostructures in which the strain is only localized near the interface due to the large aspect ratio of NWs [21].

A lattice-mismatched core–shell structure can accommodate uniform strain in the core along the growth direction, which has been suggested by both simulations [22, 23] and recent experiments of GaAs–InGaAs [24, 25] and InAsP–InP core–shell NWs [26, 27]. The strain effects in NWs also exhibited a larger axial strain than other strain components, indicating a near-uniaxial strain distribution in the core, which can not only lift the degeneracy of valence bands but provide a small effective mass along the transport direction due to a light-hole (LH) dispersion instead of heavy-hole (HH) dispersion adjacent the Γ-point [28, 29]. Thus, the mobility can be efficiently enhanced by reducing both the interband scattering and the hole effective mass [29]. In the case of GaSb NWs, however, there are only few reports on electrical properties of GaSb-InAs [30, 31] and GaSb-InAsSb1−xSbx [32] core–shell NWs where the strain in the core is limited by small lattice misfit and large core diameters. Although GaAs–GaSb core–shell NWs were demonstrated, the GaSb shell was plasticly relaxed due to unbearably high misfit between GaAs and GaSb [33]. To transfer more strain into GaSb core NWs with the same shell thickness and further to enhance the mobility as well as benefit the electrostatics of transistors by reducing the total core–shell NW diameter, a thin GaSb core is essential [34]. Nevertheless, differing from the mature control in epitaxy of III-As and III-P NWs, high
quality growth of III-Sb NWs still faces difficulties including the limitation in shrinking diameters [35] and sensitive nucleation condition because of Gibbs-Thompson effects in Sb precursors [36]. Therefore, the knowledge about strain engineering in GaSb-based NWs is very restricted in spite of its significant influence on potential improvement in electrical properties.

In this work, we for the first time demonstrate compressively-strained GaSb NWs with a GaSb–GaAs0.33Sb0.67 core–shell structure epitaxially grown on pre-patterned Si substrates by metal-organic vapor phase epitaxy (MOVPE). Large arrays with an area of 1.5 cm × 3 cm were utilized to enable highly homogeneous sizes and good positioning in NWs. The GaSb core and GaAs0.33Sb0.67 shell were successively grown on an InAs stem whose growth was initialized using the Au-assisted vapor–liquid–solid process (see details in Methods). The misfit between GaSb and GaAs0.33Sb0.67 ranges 1.5%–3.9% while the highest axial strain reaches ~0.92% by reducing GaSb core diameter down to 42 nm.

2 Results and discussion

The morphology of InAs/GaSb as well as InAs/GaSb–GaAs0.33Sb0.67 core–shell NWs were examined by scanning electron microscopy (SEM) (Figs. 1(a) and 1(b)). By comparing the diameters of the different segments for increasing GaAs0.33Sb0.67 shell growth time (Fig. 1(c)), we conclude that a significant shell growth only occurs on the sidewalls of the zincblende GaSb segment and no measurable shell growth on the wurtzite InAs stem due to the lower surface energy [37]. The diameter of the core–shell NWs increases almost linearly with the shell growth time, with a stable growth rate of ~0.44 Å/s. During the radial shell growth, a short axial GaAsSb segment on top of the NW is also grown from the Au particle.

A transmission-electron microscope (TEM) image of a NW with core diameter \(d_{\text{core}} = 50\) nm and shell thickness \(t_{\text{shell}} = 8\) nm is shown in Fig. 1(d), combined with a corresponding diffraction pattern, which indicates [111] as the NW growth direction and [110] as the upper sidewall facet in agreement with our earlier studies [19]. Both axial and shell growth of GaAs0.33Sb0.67 were visible simultaneously in the TEM as shown in Fig. 1(d) and separated by the dashed lines, in agreement with the SEM inspection in Fig. 1(b). Scanning TEM (STEM) along with X-ray energy dispersive spectroscopy (XEDS) is used to determine the As composition in both the shell and the axial segment displayed in Fig. 1(e). The elemental distribution of Ga, Sb and As was obtained by XEDS inspection in Figs. 1(b) and separated by the dashed lines, in agreement with the axial GaAs0.33Sb0.67 segment in Fig. 1(d) (both the shell and axial segment) segments separated by dashed lines. The inset shows the corresponding diffraction pattern of the NW, indicating the NW growth direction and sidewall facet.

X-ray diffraction (XRD) measurements with \(111\) \(ω/2θ\)-scans were employed for probing the axial strain in the GaSb core. As sketched in Fig. 2(a), A large \(ω\)-offset \(Δω = 0.8^\circ\) was utilized for suppressing the diffraction signal from the bulk substrate and planar growth (see Fig. S4(a) in the ESM, Section S3) owing to the fact that NWs exhibit broad peaks along the \(ω\) axis [38]. Figure 2(b) presents the XRD curves of bare GaSb NWs and GaSb–GaAs0.33Sb0.67 core–shell NWs with \(t_{\text{shell}} = 9.2\) nm. For clarity, we set the peak positions of the InAs buffer layer (zincblende) \((2θ_{\omega\text{a}})\) to \(0^\circ\) to obtain the relative peak positions \((2θ – 2θ_{\omega\text{a}})\). The strongest peaks of both XRD spectra account for GaSb since it has a much larger volume as compared to the InAs and GaAsSb segments. The GaSb peak of the core–shell NW corresponds to an axial strain \(\varepsilon_{zz}\) of ~0.92% in the core. For the sample in Fig. 1(d), the axial strain in the GaSb core determined from XRD agrees well with the strain obtained from \(111\) planar distance measured in TEM by using selective fast-Fourier transformation of the GaSb core (see Fig. S1(c) in the ESM, Section S1). Thus, the \(\varepsilon_{zz}\) determined from XRD is reasonable. The shell As composition was determined by the

![Figure 1](image-url)
 decrease the hole effective mass down to 0.05m₀ along [111] in GaSb [40]. This value is 8 times smaller than the unstrained HH mass of 0.4m₀, and is thus expected to lead to a mobility improvement [9]. However, εzz drops when incorporating more As in the GaAsₐSb₁₋ₐ shell, suggesting that an optimal As composition close to x = 0.33 gives the highest strain for t_shell = 8.3–13.3 nm, which is similar to results reported for GaAs–InGaAs core–shell NWs [24]. The reason for this result can be interpreted as different plastic relaxation in the samples with different As compositions, which will be discussed later. Surprisingly, the sample with 4.4 nm GaAs₀.₃₃Sb₀.₆₇ shell exhibits a high axial strain of −0.88%, which is very close to the highest strain (−0.92%) in the sample with 9.2 nm GaAsₓSb₁₋ₓ shell. A significant suppression of plastic relaxation along [111] direction was found in the sample with 4.4 nm GaAsₓSb₁₋ₓ shell, accounting for the reason of this phenomenon, which will be discussed in detail later. The shell thickness of samples in this series varies due to that different shell As compositions give different growth rates (see Section S2 in the ESM). Although a different strain distribution as a single function of the As composition is expected, the conclusion that GaSb core reaches the maximum axial strain with a GaAsₓSb₁₋ₓ shell is still valid since the strain decreases when increasing both the shell thickness and the As composition.

High-resolution TEM (HRTEM) was used to estimate the density of edge dislocation loops (loop dislocations) originating from the large misfit of the core and shell, which can introduce the plastic relaxation along [111] in the core. Figure 3(a) reveals three local dislocations (black arrows) near the interface of the core and shell. One additional atomic plane is observed in the shell (Fig. 3(b)), leading to a reduced lattice mismatch along the z-axis between the GaSb core and GaAsₓSb₁₋ₓ shell. No matter the direction of the loop dislocation, there is always a z-component of Burgers vector involved [41]. Hence, we can attribute the local dislocations seen in TEM to loop dislocations perpendicular to the NW growth axis, sketched in Fig. 3(c). The simulated axial strain field in a core–shell NW with the same geometry and shell As composition as shown in Fig. 3(a) is presented in Fig. 3(d), showing εzz = −0.86% without any dislocations (only elastic relaxation occurs), whereas the experimental value measured in XRD gives εzz = −0.11%. Here we define the axial strain reduction in percentage as the dislocation-induced plastic relaxation Rp = (εzz − ε′zz)/εzz, resulting in 87% plastic relaxation for this sample. This gives a density of 7.7 × 10⁵ cm⁻² assuming a uniform loop dislocation distribution (Fig. 3(a)) (see details in the ESM, Section S4). To reach the experimentally measured strain of εzz = −0.11%, loop dislocations with a density of 7.9 × 10⁵ cm⁻² had to be included in the simulation (Fig. 3(e)) (see Method). This density is in good agreement with that observed in TEM. Therefore, we can confirm that the axial strain reduction is mainly due to plastic relaxation caused by loop dislocations.

Figures 3(f) and 3(g) present the simulation results of Rp as a function of shell thickness and As composition, respectively. The axial strain εzz can be written as εzz = ε′zz (1 − Rp). For a GaAsₓSb₁₋ₓ shell with t_shell > 3.4 nm, a similar Rp (~ 70%) was calculated, so εzz is only determined by the elastic strain ε′zz which here mainly depends on t_shell. Thus, εzz increases and eventually saturates, the same as reported cases [25, 26]. For t_shell < 3.4 nm, Rp gradually increases from 20% to a high level (Fig. 3(f)) that effectively limits the axial strain increasing so that εzz increases fast first and slowly afterwards (Fig. 2(c)). In Fig. 3(g), the plastic relaxation Rp increases with the shell As composition. Additionally, both ε′zz and Rp increase when increasing the shell As composition with a similar t_shell, resulting in
in a tradeoff in $\varepsilon_{zz}$ and $R_p$ to emerge a maximum value of $\varepsilon_{xx}$ with an optimal $x = 0.33$ in GaAs$_{0.33}$Sb$_{0.67}$. When shrinking $t_{\text{shell}}$ from 9.2 to 4.4 nm but remaining the same GaAs$_{0.33}$Sb$_{0.67}$ shell, the drop of $R_p$ from 34% to 2% (the purple star in Fig. 3(g)) balances the elastic strain $\varepsilon_{zz}$ reduction, which leads to a similar $\varepsilon_{xx}$ in the core. The significant suppression of plastic relaxation in the sample with 4.4 nm GaAs$_{0.33}$Sb$_{0.67}$ shell can be explained by the critical shell thickness in the lattice-mismatched core–shell structure [42]. For $d_{\text{core}} = 42$ nm and a GaAs$_{0.33}$Sb$_{0.67}$ shell, we speculate that the critical shell thickness should be slightly smaller than 4.4 nm, indicating almost no presence of plastic relaxation. However, increasing $t_{\text{shell}}$ to 9.2 nm, which is far beyond the critical shell thickness, introduces plastic relaxation to the axial strain, thus no evident strain increase.

Finally, Raman microscopy was performed on individual NWs to enable evaluation of the hydrostatic strain ($\varepsilon_{xx}+\varepsilon_{yy}+\varepsilon_{zz}$) of the core–shell NWs, thus calculating the valence band shifts at the $\Gamma$-point. Figure 4(a) presents Raman spectra of a bare GaSb NW and a core–shell NW from the same sample as Fig. 2(b). In the case of the bare GaSb NW, both transverse optical (TO) and longitudinal optical (LO) phonons are clearly observed, with peak positions at 224.8 and 233.3 cm$^{-1}$, respectively, in good agreement with reported values [43]. For the core–shell NW, however, the LO mode was dramatically suppressed even with a shell as thin as 0.8 nm (see all Raman spectra in the ESM, Section S5), while the TO mode still has a high intensity in the sample with 9.2 nm GaAs$_{0.33}$Sb$_{0.67}$ shell. The same conclusion can be drawn when considering the shell thickness variation.

Combining the axial strain with the hydrostatic strain in the core, we are able to deduce the sum of the radial strain $\varepsilon_{xx}+\varepsilon_{yy}$ and thus estimate the band edge of the strained GaSb core. The band edge depends on both the hydrostatic strain $\varepsilon_{\text{hydro}}$ and the strain component $\varepsilon_{\text{val}} = \varepsilon_{xx} - 0.5 \cdot (\varepsilon_{xx}+\varepsilon_{yy})$ (here, the subscript “vbs” denotes the valence band splitting since this strain component only affects the LH maximum shift) [46]. Table 1 lists the calculated results of the difference between the
interband scattering is expected for this sample. As a consequence, suppression of interband scattering, a process usually accompanied by absorption or emission of optical phonons [47]. This means the interband scattering rate for holes at the Γ-point at the valence band maximum can be drastically lowered as long as the separated energy \( E_{\text{LH}} - E_{\text{HH}} \) is greater than the optical phonon energy \( h\nu_0 \) [29]. Thanks to the nearly absent of plastic relaxation, the sample with \( t_{\text{shell}} = 4.4 \text{ nm} \) has similarly high axial strain as the sample with \( t_{\text{shell}} = 9.2 \text{ nm} \) while a manifest reduction in \( \varepsilon_{\text{hyd}} \), thus providing a more significant valence band splitting of 33.4 meV which is greater than the optical phonon energy \( h\nu_0 \approx 28 \text{ meV} \) with the optical phonon frequency \( \nu_0 \approx 230 \text{ cm}^{-1} \) extracted from Raman. As a consequence, suppression of interband scattering is expected for this sample.

In addition, the LH band appears at the top of the valence band \( (E_{\text{LH}} - E_{\text{HH}} > 0) \), allowing more light holes to contribute to the electrical transport. In the other words, the hole effective mass should be much reduced near the Γ-point. Also, highly compressive strain along the transport direction [111] can also increase the curvature in the \( E-k \) dispersion (band warping), leading to a further reduction in the effective mass of holes along [111]. Both the suppression of the interband scattering and the effective mass lowering along transport axis are therefore expected to improve the hole transport properties in compressively strained GaSb core NWs. However, in the case of biaxial compressive strain in planar GaSb films, although the degeneracy of valence bands is lifted the HH maximum stays above the LH maximum, resulting in the mobility enhancement mainly due to the valence band splitting that reduces the interband scattering between the valence bands, rather than slight reduction in the effective mass due to band warping [48]. Consequently, a very high strain is required to split the valence bands sufficiently to enhance the mobility for biaxial compression. In contrast, the core–shell structure with high axial strain and small hydrostatic strain may more efficiently enhance the mobility than that with biaxial strain in the film. This agrees well with simulation results from Nainani et al. [49] as well as experimental data from Thompson et al. [50] which indicated that the same strain level result in higher mobility in the uniaxial case than in the biaxial one. On the other hand, the bandgap widening due to the compressive strain may benefit the off-state performance of a transistor. More axial compressive strain in GaSb core is likely to further increase the hole mobility. The best option is considered to reduce the core diameter even more, which can significantly increase the critical shell thickness as well as the tolerance of lattice misfit for the same critical shell thickness [42, 51]. However, it is currently challenging to reduce the diameter of

![Figure 4](image)

**Figure 4** (a) Raman spectrum of a core–shell NW (bottom) from the same sample as in Fig. 2(b) and a spectrum of a bare GaSb NW (top). (b) and (c) show the TO mode shift of the GaSb core with varying the shell thickness \( t_{\text{shell}} \) and shell As composition \( x \), respectively. (d) and (e) show the corresponding extracted hydrostatic strain. Red dots in ((b) and (c)) or ((d) and (e)) correspond to the same sample. Lorentzian fitting was used for Raman curves to interpret the different scattering modes. The growth rate of the shell was found to be slightly different when varying the As composition but remaining the same growth time of 220 s in (e).

| Shell type | \( t_{\text{shell}} \) (nm) | \( \varepsilon_{\text{xx}} \) | \( \varepsilon_{\text{yy}} \) | \( \varepsilon_{\text{hyd}} \) | \( E_{\text{LH}} - E_{\text{HH}} \) (meV) | \( E_g \) (eV) |
|------------|------------------|--------|--------|--------|----------------|--------|
| No shell   | 0                | 0      | 0      | 0      | 0              | 0.72   |
| GaAs\(_{0.67}\)Sb\(_{0.33}\) shell | 9.2        | -0.92% | -1.91% | -0.43% | 23.9           | 0.85   |
| GaAs\(_{0.67}\)Sb\(_{0.33}\) shell | 4.4        | -0.88% | -1.46% | -0.59% | 33.4           | 0.81   |

LH and HH maxima \( (E_{\text{LH}} - E_{\text{HH}}) \) and the bandgap \( (E_g) \) of the strained GaSb core (see Methods), resulting from different strain components. By shrinking \( t_{\text{shell}} \) from 9.2 to 4.4 nm, \( \varepsilon_{\text{xx}} \) keeps similar while \( \varepsilon_{\text{hyd}} \) drops much, leading to an increasing value of \( E_{\text{LH}} - E_{\text{HH}} \) up to 33.4 meV.
<111>-oriented GaSb NWs grown by MOVPE below 32 nm [35]. Thus, for a NW with \( d_{\text{core}} = 32 \text{ nm} \), based on simulation, 1.08% axial strain can be achieved (increasing 23% compared to the case of \( d_{\text{core}} = 42 \text{ nm} \) in the GaSb core without any plastic relaxation by selecting GaAsSb_{0.33}Sb_{0.67} shell and \( t_{\text{shell}} = 4.4 \text{ nm} \). This structure is expected to further enlarge \( \omega \)-scattering and the hole effective mass. Besides, external stress further by reducing both the interband coupling band edge at the Γ-point of GaSb core was calculated by band-edge deformation potential theory [46]. GaSb band parameters along <111> were extracted from Ref. [46].

4 Conclusion

We have introduced compressive strain in GaSb NWs along [111]-direction by epitaxially growing GaSb-GaAsSb\( \text{_{0.33}} \text{Sb}_{0.67} \) core−shell heterostructures integrated on Si substrates. Various shell As compositions and core−shell dimensions have been investigated to establish the growth conditions to accommodate the highest axial compressive strain in the GaSb core. The axial strain is obtained and its relaxation mechanism is described by a theoretical model for loop dislocations, also offering a guideline to achieve high strain in the GaSb core without dislocation-induced plastic relaxation. The hydrostatic strain is further determined and the strain effects on the band structure are evaluated, showing more benefits than existing biaxially strained GaSb films for hole mobility enhancement. Our results unlock the strain engineering in GaSb-based NWs and suggest that high compressive strain can also be introduced into GaSb NWs with high quality epitaxy, which provides an attractive option to further improve the electronic properties, in particular the hole mobility, of p-type MOSFETs to be used in for instance all-III-V CMOS digital applications.

4 Methods

4.1 Nanowire growth

Prior to the growth, large arrays with a large area of 1.5 mm × 3 mm consisting of Au discs with a pitch of 1 μm and diameters of 20–36 nm were patterned by electron-beam lithography on Si (111) substrates with a 260-nm-thick InAs layer on top. All the NWs were grown by MOVPE in a close-coupled showerhead system 18313 at a pressure of 100 mbar and a total flow of 8,000 sccm. The growth is initialized with an InAs stem at 448 °C for 32 or 36 nm Au particles while 465 °C for 20 nm ones, using trimethylindium (TMIn) and arsine with a molar fraction of \( X_{\text{TMIn}} = 6.25 \times 10^{-4} \) and \( X_{\text{arsine}} = 1.25 \times 10^{-4} \), respectively. Next, GaSb was grown while heating up and mainly at 505 °C with trimethylgallium (TMGa) and TMSb with a molar fraction of \( X_{\text{TMGa}} = 4.9 \times 10^{-4} \) and \( X_{\text{TMSb}} = 2.1 \times 10^{-4} \), respectively. At the same temperature, GaAsSb shells were finally grown with the same \( X_{\text{TMGa}} \) and a V/III ratio of 2.

4.2 TEM characterization

The structural and compositional analysis using TEM consisted of HRTEM imaging in conventional TEM mode and spectroscopic acquisition using XEDS, the latter in STEM mode. The TEM used was a JEM-3000F, operated at 300 kV. NWs were transferred to lacy carbon covered Cu-grids by pressing the grid against the substrate, hence breaking off wires. HRTEM micrographs were acquired and used to analyze the local lattice and defects. Compositional mapping was performed in STEM mode using an XEDS-detector (Oxford Instruments) and quantified using the corresponding software Aztec.

4.3 XRD measurements

Lab-setup Bruker D8 system equipped with a 40 kV Cu X-ray source (\( \lambda = 1.541 \text{ Å} \)) was used for all XRD measurements. All the NW samples with the large array of 1.5 mm × 3 mm were directly measured in XRD setup. (111) \( \omega/2\theta \) scans with large \( \omega \)-offset \( \Delta \omega = 0.8° \) were carried out for probing the axial strain along NW growth direction.

4.4 Raman measurements

Renishaw inVia Raman microscopy equipped with Leica N Plan (100/0.9) objective was used for strain study of individual NWs which were first transferred onto a pre-patterned SiO\(_2\) substrate coated with Au. All Raman spectra were measured with an excitation laser with a wavelength of 785 nm. In all cases, the laser power and spot size were ~0.1 mW and ~2 μm, respectively.

4.5 Theoretical modeling

The finite-element continuum elasticity model included in COMSOL Multiphysics was used for strain field simulations of core−shell NWs. The lattice mismatch between GaSb and the GaAsSb\( \text{_{0.33}} \text{Sb}_{0.67} \) shell was selected as the initial strain. A 3D model of NWs with finite length (500 nm) was built and the elastic constant matrix of materials were converted from <001> to <111> [22, 54]. Edge dislocation loops at the interface of the core and shell were intentionally taken into account to explain the mechanism of strain relaxation [42, 55]. The Burgers vector \( b = a/3 \) [111] in the loop results in a reduced initial mismatch along the NW growth direction [42, 56]. Strain-coupling band edge at the Γ-point of GaSb core was calculated by band-edge deformation potential theory [46]. GaSb band parameters along <111> were extracted from Ref. [46].

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