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Passivation efficacy study of Al_2O_3 dielectric on self-catalyzed molecular beam epitaxially grown $\text{GaAs}_{1-x}\text{Sb}_x$ nanowires

Mehul Parakh^{1,3} , Priyanka Ramaswamy^{2,3} , Shisir Devkota¹, Hirandeep Kuchoor¹, Kendall Dawkins¹ and Shanthi Iyer¹ 

¹Nanoengineering, Joint School of Nanoscience and Nanoengineering, North Carolina A&T State University, Greensboro NC, 27401, United States of America

²Department of Electrical and Computer Engineering, North Carolina A&T State University, Greensboro NC, 27411, United States of America

E-mail: iyer@ncat.edu

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Abstract

This work evaluates the passivation efficacy of thermal atomic layer deposited (ALD) Al_2O_3 dielectric layer on self-catalyzed $\text{GaAs}_{1-x}\text{Sb}_x$ nanowires (NWs) grown using molecular beam epitaxy. A detailed assessment of surface chemical composition and optical properties of Al_2O_3 passivated NWs with and without prior sulfur treatment were studied and compared to as-grown samples using x-ray photoelectron spectroscopy (XPS), Raman spectroscopy, and low-temperature photoluminescence (PL) spectroscopy. The XPS measurements reveal that prior sulfur treatment followed by Al_2O_3 ALD deposition abates III–V native oxides from the NW surface. However, the degradation in 4K-PL intensity by an order of magnitude observed for NWs with Al_2O_3 shell layer compared to the as-grown NWs, irrespective of prior sulfur treatment, suggests the formation of defect states at the NW/dielectric interface contributing to non-radiative recombination centers. This is corroborated by the Raman spectral broadening of LO and TO Raman modes, increased background scattering, and redshift observed for Al_2O_3 deposited NWs relative to the as-grown. Thus, our work seems to indicate the unsuitability of ALD deposited Al_2O_3 as a passivation layer for GaAsSb NWs.

Supplementary material for this article is available [online](#)

Keywords: atomic layer deposition (ALD), Al_2O_3 passivation, $\text{GaAs}_{1-x}\text{Sb}_x$ nanowires (NWs), molecular beam epitaxy (MBE), dielectric/III–V nanowire interface

Introduction

III–V semiconductor nanowires (NWs) are being pursued as a potential candidate for next-generation electro-optical devices. They offer unique and superior properties stemming from their anisotropic geometry compared to their bulk counterparts [1–3]. The self-catalyzed vapor–liquid–solid growth mechanism of these nanowires enables high phase purity growth with excellent homogeneity amongst NW ensembles [4], thus eliminating foreign contamination and induced

defects arising from metal-catalyzed growth that has been commonly used until recently. However, the presence of a high density of surface states arising due to the anisotropic NW geometry is detrimental to the efficiency, sensitivity, and speed of these self-catalyzed III–V NW devices [5]. The severity of surface state formation depends on the un-passivated dangling bonds arising from the sharp termination of material growth radially and native oxide formation. These can act as non-radiative recombination centers or surface charge traps, thereby increasing surface scattering leading to decreased carrier mobility and increased density of electronic states within the bandgap, causing Fermi level pinning at the

³ Mehul Parakh and Priyanka Ramaswamy share the joint first authorship.

surface [6]. It is known [7] that the Fermi level pinning creates a depletion region on the surface, which can extend throughout the NW volume, rendering it entirely insulating or non-conducting, thereby hindering the use of NWs for practical applications. Hence, to reduce the effect of surface states, different passivation schemes have been investigated, namely *ex situ* treatment using wet chemical processes involving sulfur [8], hydrazine-sulfide [9], and *in situ* treatment involving shell growths of AlGaAs [10, 11], or InGaP [12]. Among these, treatment with sulfur and hydrazine-sulfide for long-term passivation is limited, inconvenient because of tedious chemical process control, and in the latter case hazardous due to its combustible and toxic nature. In contrast, the constraint of *in situ* passivation is the difficulty in obtaining shell growth uniformity for high-aspect-ratio NWs due to the shadowing effect [13]. Therefore, there is a need to explore suitable passivation techniques for III–V NWs that can provide long-term stability against any ambient variation and process convenient.

Recently, high k-dielectric materials such as Al₂O₃ and HfO₂ are emerging as promising candidates for III–V planar device passivation [14]. Among these, atomic layer deposited (ALD) Al₂O₃ layers has been attracting attention. This is due to the flexibility of *ex situ* deposition by ALD, enabling precise thickness control and unpinning of the Fermi level, owing to the inherent native-oxide cleaning property of Trimethylaluminum (TMA) precursor during the initial cycles of deposition [15, 16]. Further, low leakage current density (10^{-8} A cm⁻²) and low interfacial density ($D_{it} \sim 10^{12}$ eV⁻¹ cm⁻²) are reported on Al₂O₃ ALD deposited III–V planar devices [17–19]. In addition, its larger bandgap compared to HfO₂ and good environmental resistance are some key advantages cited for its potential use as a passivation layer [20]. In NWs, however, there have been mixed results. Enhanced PL characteristics and suppressed surface recombination velocity of $\sim 10^3$ cm s⁻¹ were observed in In-based III–V NWs with ALD Al₂O₃ deposited passivation layers [21, 22]. In contrast, in thin GaAs NWs of 40 nm diameter, the passivation effect of the Al₂O₃ layer on the NWs was absent, as evidenced by the lack of PL emission for both as-grown and Al₂O₃ passivated NWs [23]. It was attributed to strong Fermi level pinning by large surface state density of thin NWs, leading to complete depletion.

In the last few decades, arsenide–antimonide (As–Sb) based material systems, particularly mixed-anion GaAs_{1-x}Sb_x materials, are among the promising candidates for widespread device applications from photonics to electronics [24–26]. The ability of bandgap tuning by varying Sb composition in GaAsSb materials makes the system attractive for tunable bandgap lasers [27] and photodetectors [28–31] in the near-infrared spectrum range. Despite the broad range of applications, the high surface recombination velocity and complex nature of native oxides pose a challenge in finding a suitable passivation layer, limiting their potential use in device technologies.

This report on the *ex situ* passivation of MBE-grown Ga-based As–Sb NWs was motivated by the simplicity of Al₂O₃ *ex situ* deposition and its success in the past oxide passivation

studies on III–V thin films and NWs, including mixed As–Sb system [21–23, 32]. This work examines the efficacy of the Al₂O₃ passivation layer deposited by thermal ALD technique on self-catalyzed ensemble GaAsSb NWs using surface and optical characterization tools, namely XPS, PL spectroscopy, and Raman spectroscopy. A systematic 4K-PL study carried on Al₂O₃ passivated NW samples observed a severe optical degradation compared to as-grown samples. XPS analysis indicated that the precleaning with sulfur treatment before the deposition of the Al₂O₃ shell layer effectively removed III–V native oxides formed on the NW surface, thereby eliminating their role in optical degradation of the Al₂O₃ passivated NW samples. From Raman analysis, increased background scattering, spectral broadening of LO and TO Raman modes, and enhanced redshift compared to the as-grown NWs suggest increased density of point defects due to complex NW/dielectric interface reconstruction. These act as non-radiative recombination centers and can be the main contributors towards the optical degradation of GaAsSb NWs on Al₂O₃ shell layer deposition.

Results and discussions

The growth rate of Al₂O₃ deposition by thermal ALD was initially calibrated using 90 cycles of trimethylaluminum (TMA) and water vapor precursors on GaAs (100) substrates, which has undergone similar substrate preparations as SAO NWs mentioned in the Methods section. Statistical wafer grid scans with 49 average points using spectroscopic ellipsometry on Al₂O₃ deposited GaAs wafer was performed for refractive index versus thickness measurements as shown in Supplementary (figure S1 (available online at stacks.iop.org/NANO/33/315602/mmedia)), which yielded Al₂O₃ oxide layer thickness of $\sim 9.6 \pm 0.5$ nm. This corresponds to an average growth rate of ~ 1.01 – 1.12 Å/cycle and refractive index (n_f) $\sim 1.65 \pm 0.1$, as ascertained using a 633 nm wavelength light source. The slight deviation of the observed refractive index from ~ 1.63 corresponding to the stoichiometric Al₂O₃ layer grown on GaAs (100) substrate can be correlated to the complex interface layer formed between the dielectric and the GaAs surface due to the spurious oxidation effect of GaAs [33]. III–V semiconductors, in general, are known for their high surface reactivity, posing a challenge for ideal interface formation during the subsequent device processing steps. Hence, chemical passivation by sulfur was employed before Al₂O₃ ALD deposition on self-catalyzed GaAsSb NWs to minimize this effect. This study used Al₂O₃ deposition thickness of ~ 3 – 4 nm based on the past passivation reports [16, 18, 32].

Scanning electron microscopy

The SEM images of R, AO, and SAO samples, as shown in figures 1(a)–(c), reveal high-density vertical NWs with an average length (measured for around 50 NWs) of $\sim 0.7 \pm 0.2$ μm. From the inset of SEM images, the observed

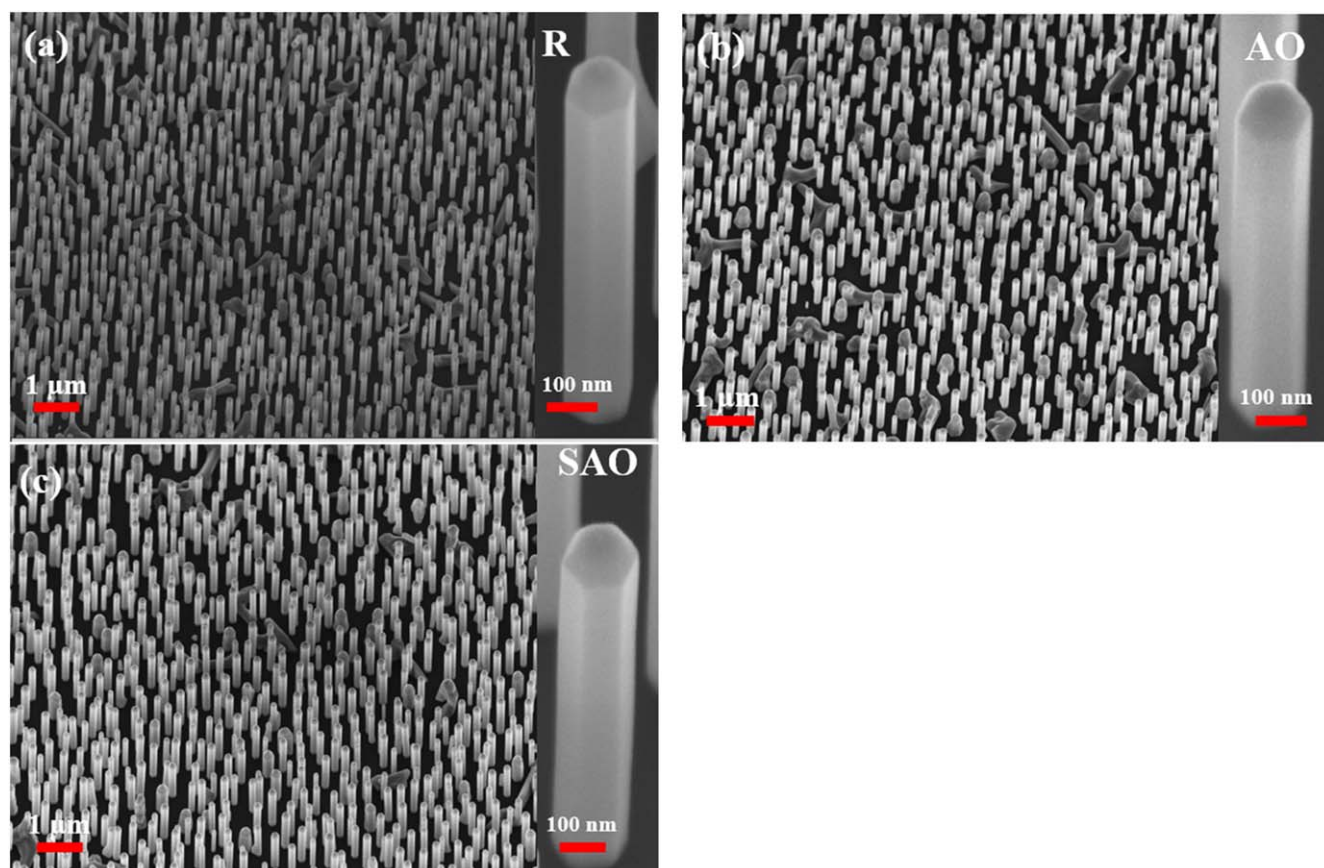


Figure 1. SEM images of self-catalyzed GaAsSb NW samples: (a) R, (b) AO, and (c) SAO (inset: SEM images of corresponding SNW).

average single nanowire (SNW) diameter for the R sample was $\sim 140 \pm 5$ nm, AO sample, and the SAO sample were $\sim 148 \pm 5$ nm, yielding a uniform Al_2O_3 layer deposition thickness of $\sim 3.5 \pm 0.5$ nm on the NW surface.

It is to be noted that all these samples are based on the growth conditions published elsewhere [28, 34], where the crystal structure was confirmed to exhibit zincblende structure with an Sb composition of 7%, as determined from the scanning transmission electron microscope and energy-dispersive x-ray spectroscopy, respectively.

X-ray photoelectron spectroscopy

XPS spectra of binding energy between 0 and 150 eV of self-catalyzed GaAsSb NW samples R, AO, and SAO are shown in figure 2. The peaks of O 2s, Al 2p, and Al 2s at 23.3 eV, 74.6 eV, and 118.9 eV, respectively, present only in AO and SAO NW samples attest to the formation of the Al_2O_3 passivation layer on the surface [35].

The core-level spectra of Ga 3d and As 3d of as-grown R sample are depicted in figure 3, whereas figure 4 represents Al 2s, O 1s, Ga 3d, and As 3d spectra of AO and SAO samples. The peaks observed at 21 eV and 44.5 eV (figures 3(a) and (b)) are assigned to Ga and As complex native oxide-related peaks, respectively [32]. The atomic ratio of O/Al is 1.7 and 1.3 for AO (figures 4(a) and (b)) and SAO (figures 4(c) and (d)) samples, respectively. The appearance of Ga–O

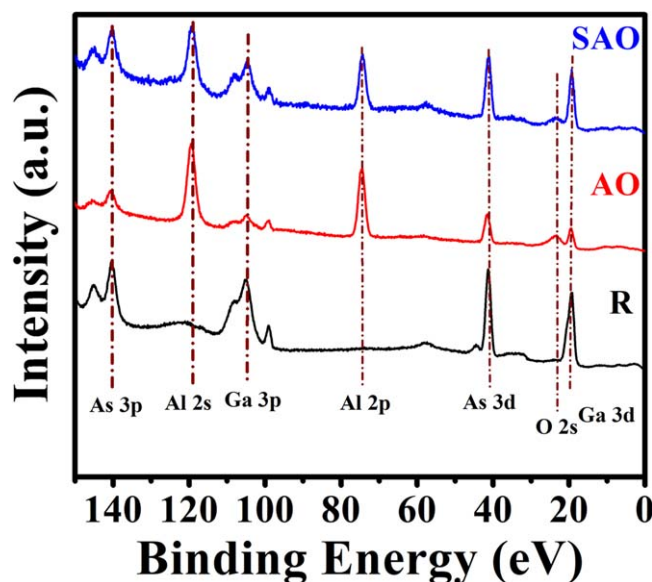


Figure 2. XPS spectra of R, AO, and SAO samples.

(figure 4(e)) only in the AO sample corroborates the observed increase in O: Al stoichiometry in this sample. It is speculated that the interdiffusion of oxygen during ALD deposition from native oxides to the Al_2O_3 passivation layer has contributed to the higher O/Al ratio in the AO sample. The absence of satellite peaks (figures 4(a)–(d)) rules out the formation of any other sub-oxide species of Al during the ALD deposition of

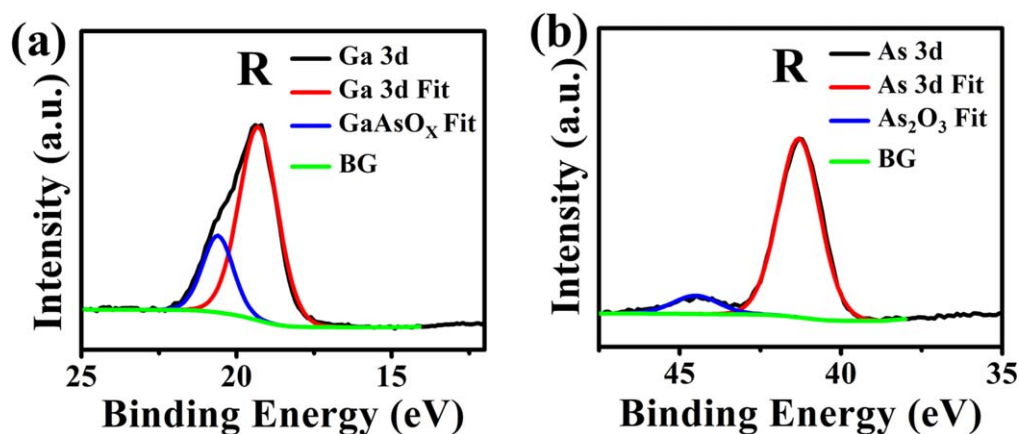


Figure 3. XPS spectra of Ga 3d and As 3d of R sample.

the Al_2O_3 passivation layer on NWs [33]. A lack of As–O peak (figures 4(f) and (h)) on Al_2O_3 deposited NWs in both non-sulfur and sulfur treated samples can be viewed as evidence of the self-cleaning process of TMA [16]. Therefore, sulfur treatment before the deposition of 3 nm of the Al_2O_3 passivation layer effectively removes the native oxides of Ga in NW samples. Further, Sb 3d signal was not registered in any of the GaAsSb NWs, which we attribute to a strong O 1s signal due to the overlapping binding energies of O 1s and Sb 3d peaks [36]. The other contributing factors could also arise from radial variation in Sb composition due to the As–Sb exchange mechanism at the NW surface [37], manifesting in Sb composition reduction at the surface as the XPS probing depth is ~ 10 nm.

Low-temperature photoluminescence spectroscopy (4K-PL)

Next, the optical properties of the passivation effects of sulfur treatment and Al_2O_3 deposition on self-catalyzed Te-doped GaAsSb NWs compared to as-grown were assessed by 4K-PL spectroscopy characteristics of samples R with AO and SAO shown in figure 5.

The lack of sharp low energy onset in all the samples suggests the presence of defects attributed to acceptor vacancies in GaAsSb NWs. A drastic reduction in 4K-PL intensity by a factor of ~ 10 accompanied by increased FWHM was observed for AO and SAO samples, indicating the dominance of non-radiative recombination centers. Also, a small blue shift of 15–18 meV in the PL peak energy for these samples is attributed to the strain-induced due to the Al_2O_3 layer lattice mismatch with the NW surface. From SEM images (figure 1), the density of the NWs is found to be nearly uniform, hence ruling out the effect of variation in the NW density within the sample.

Recently, Ren *et al* [21] hypothesized that Al–O participates in interfacial bonding by substituting sulfur terminated bonds during Al_2O_3 deposition on InAsSb NWs. These probably elucidate why AO and SAO samples in this study observe similar PL results, with no observable effect of prior

sulfur treatment on NW passivation. Dhaka *et al* [23] has already shown a lack of improvement of the GaAs NW optical quality with the Al_2O_3 passivation layer. However, it is essential to note that no PL emission was observed in their both as-grown and Al_2O_3 deposited GaAs NWs. They attributed this effect to the thin diameter of as-grown GaAs NWs (~ 40 nm diameter) pinning the Fermi level. However, as GaAsSb NWs in this work are much thicker ~ 140 nm in diameter, PL deterioration due to the Fermi level pinning effect can be ruled out. Therefore, the reason for deterioration is different and probably more complex. Further, similar degradation in optical PL intensity has been observed for InP NWs after ALD deposition of HfO_2 [38]. It is to be noted that though data on only three representative samples are shown here, due to the conflicting reports of Al_2O_3 passivation effects on III–V NWs [21–23], the PL measurements were carried out on three sets of samples from different growth runs of both intrinsic and Te-doped NWs to ensure the reproducibility of observed results (shown in supplementary figure S2).

The optical degradation of NWs after Al_2O_3 deposition, irrespective of sulfur treatment or lack of it, indicates the presence of defect states contributing to non-radiative recombination centers. These defect states can be speculated to arise from (i) gap states or antibonding levels created on burying of As and Sb group V dimers formed during surface reconstruction under the Al_2O_3 layer, and/or (ii) complex interface reconstruction at the NW/dielectric interface due to lower symmetry of the surface than the bulk and presence of different surfaces [39, 40]. This complex NW/dielectric interface reconstruction can give rise to the large density of point defects because well-ordered interface layers are known to exhibit fewer point defects due to lower interface state density [41]. Further, the different surfaces of the one-dimensional configuration of the NWs may promote complex NW/dielectric interface reconstruction, enhancing interface state density. GaAsSb NWs are rich in exhibiting well-defined facets, as is the case in our grown NWs as well (SNW images in figures 1(a) and (b)). Besides these, the contribution from defect states on the semiconductor side, the presence of trap states, and native point defects in the Al_2O_3 layer itself

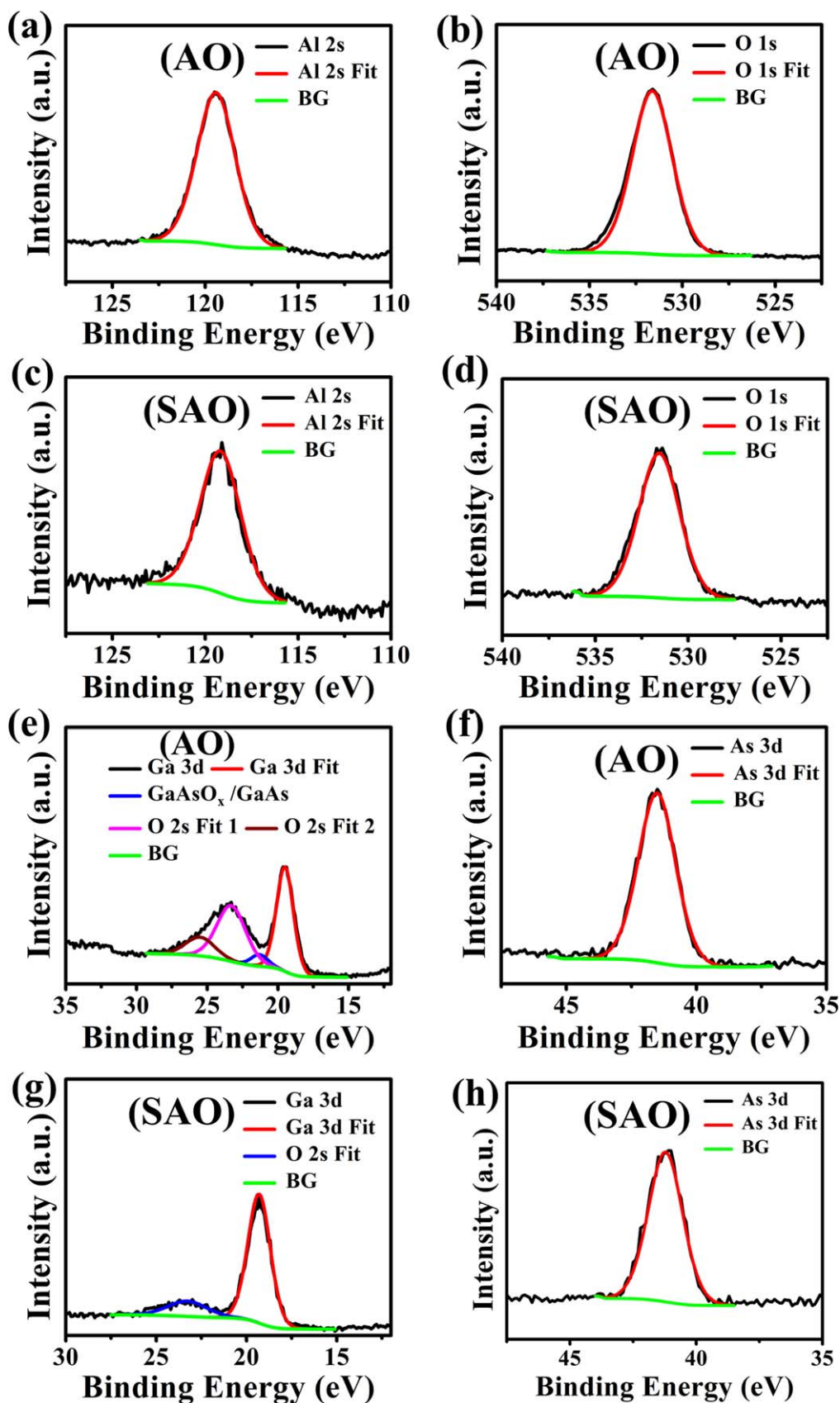


Figure 4. XPS spectra for (a), (c) Al 2s and (b), (d) O 1s of (1st row) AO and (2nd row) SAO NW samples, respectively. XPS spectra for (e), (g) Ga 3d and (f), (h) As 3d of (3rd row) AO and (4th row) SAO NW samples, respectively.

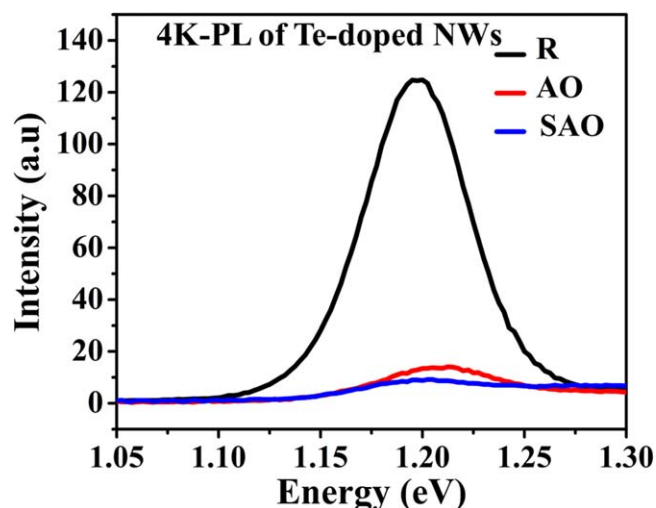


Figure 5. 4K-PL comparison of Te-doped GaAsSb: R, AO, and SAO samples.

cannot be ruled out [42, 43]. Generally, higher bandgap (bandgap higher than incoming radiation) passivation layers are preferred due to their transparency to incoming radiation. Al_2O_3 being a high-bandgap material ~ 7 eV with a lower refractive index than GaAsSb, promotes waveguiding effect in the NWs. Therefore, the contribution from absorption and scattering in the oxide can be neglected.

Our observations are contrary to those observed in InAsSb NWs [21] and InP NWs [22], where Al_2O_3 layer passivation resulted in enhanced PL intensity. This can be attributed to a higher atomic radius of In when compared to Ga, thereby promoting long-range ordering at the interface [41]. This can increase the number of chemical bonds formed with the dielectric, providing better surface ordering, resulting in comparatively less defect state density. Hence, both factors contribute to an overall reduction in the interface state density [44] in In-based compounds. Thus, the Al_2O_3 passivation layer for GaAsSb NWs is inefficient due to the combination of NW anisotropy involving different facets/surface steps and a small atomic radius of Ga atoms promoting short-range surface ordering, thereby creating complex NW/dielectric reconstruction, resulting in a high density of defect states when compared to planar III–V's and In-based III–V NWs. However, 4K-PL spectra of R sample exhibited increased PL intensity with *in situ* grown AlGaAs passivating shell (shown in supplementary figure S3) [45, 46], thus demonstrating why AlGaAs is commonly preferred *in situ* passivation layer for Ga-based III–V NWs.

Raman spectroscopy

Raman spectroscopy was carried out as the shape of the Raman peak and spectral shift direction can reveal further insight into the nature of the defects. It has been reported that point defects induce spectral shift but do not give rise to asymmetry and vice versa for planar defects [31, 47–49]. Figure 6 shows the RT Raman spectra comparison of R, AO,

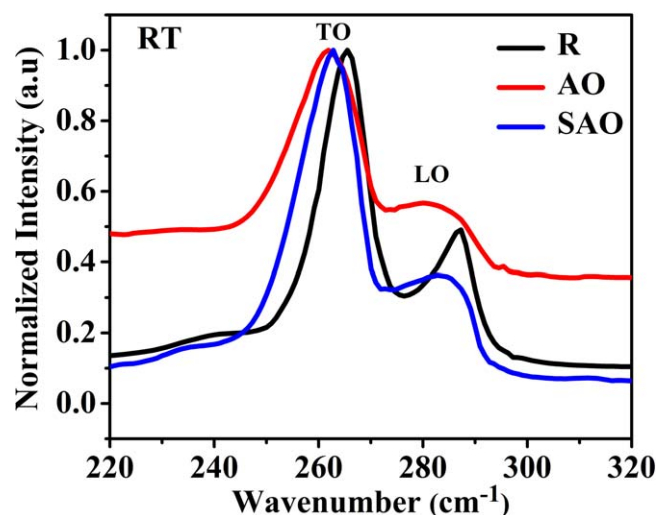


Figure 6. Normalized RT Raman spectra comparison of R, AO, and SAO samples.

and SAO samples. GaAs TO and LO vibrational mode peaks at 265 cm^{-1} and 285 cm^{-1} , respectively, were observed in the R sample [31]. The AO sample demonstrates a significant redshift of 3.5 cm^{-1} and a broad LO and TO mode spectra relative to the R sample, attributed to the induced strain on the NW surface during oxide deposition and phonon localization at the defects [25, 31, 49–51]. Further, the broad asymmetry is consistent with our conjecture of defect enhancement arising from complex NW/dielectric interface reconstruction, as explained in the PL section. Similar features are observed in the SAO sample with reduced peak broadness, suppressed background scattering intensity, and a comparatively smaller redshift of 2.5 cm^{-1} . These are possibly related to the more effective removal of III–V native oxides formed on the NW surface by sulfur treatment in SAO samples compared to AO, as observed from XPS measurements, aiding in reducing defects [29]. A nearly symmetric LO and TO peaks and redshifts in both the AO and SAO samples indicate the nature of defects being predominantly point defects, as the symmetric line shape of Raman suggest point defects while asymmetric line shape shows the presence of planar defects [25, 51, 52]. Raman characterization thus further strengthens the conjecture of defect creation at NW/dielectric interface on Al_2O_3 deposition and ascertains the positive effect of pre-sulfur treatment on NWs before oxide deposition. It is to be noted that an in-depth structural analysis is required to further strengthen these claims.

Conclusion

The effect of sulfur treatment and Al_2O_3 passivation using thermal ALD *ex situ* deposition on self-catalyzed GaAsSb NWs using XPS, PL spectroscopy, and Raman spectroscopy were successfully studied. The 4K-PL spectra revealed a significant drop in PL intensity (~ 10 -fold) for Al_2O_3 passivated NWs compared to as-grown irrespective of prior sulfur treatment. The XPS spectral analysis indicated that

Table 1. Sample preparation for as-grown GaAsSb NWs.

Nomenclature	NWs samples	Preclean & wet passivation	Al ₂ O ₃ ALD cycles
R	GaAsSb core	—	—
AO	GaAsSb core	—	30
SAO	GaAsSb core	Yes	30

precleaning with sulfur treatment before Al₂O₃ ALD deposition effectively removed III–V native oxides formed on the NW surface, thus ruling out the effect of native oxides on optical degradation. Further, the Raman characterization observed significant redshifts, peak broadening, and asymmetry for Al₂O₃ passivated NWs when compared to as-grown NWs, suggesting the presence of point defects on Al₂O₃ ALD deposition. Hence, the 4K-PL optical degradation observed in both sulfur treated and untreated Al₂O₃ passivated NWs samples suggests non-radiative recombination centers arising from complex NW/dielectric interface formation to be the main contributing factor. Reporting of similar PL degradation in GaAs NWs, while enhanced PL intensity in In-based NWs [23] allows us to speculate that non-radiative recombination centers arising from the Al₂O₃/NW interface states are peculiar to Ga-based III–V NWs only, and surface faceting in GaAsSb NWs may also enhance this contribution. Thus, ALD deposited Al₂O₃ layer is not a good choice for the passivation layer for GaAsSb NWs.

Materials and methods

Material synthesis

The growth of Ga-catalyzed axial GaAsSb NWs on Si (111) substrate was carried out via a vapor–liquid–solid growth mechanism using EPI 930 solid source MBE system. The Sb₂ and As₄ flux were provided by valved cracker sources operating at 900 °C and 600 °C, respectively. Axial n-GaAsSb NWs (Sb flux ~17% and GaTe source temperature ~530 °C) with group V/III flux ratio ~5.4 at a substrate temperature of 620 °C was preset to yield a nominal growth rate of 1.1 nm s^{−1}. Ga-catalyzed epitaxial growth was initiated with Ga opening a few seconds before opening the group V fluxes. Later, *ex situ* Al₂O₃ passivation layer thickness of ~3–4 nm at 200 °C was deposited on NW samples using trimethylaluminum (TMA) and water vapor as precursors in Savannah S200 thermal ALD system, operating at 0.5 mTorr base pressure.

Material characterization

Surface analysis was performed on a Thermo ESCALAB 250 Xi with the base chamber pressure of 10^{−10} Torr, equipped with a monochromated Al K α x-rays source of $h\nu = 1486.6$ eV for XPS measurements, at room temperature. Micro-PL spectroscopy system at 4 K temperature was used to measure the optical characteristics of NWs, which employ

a 633 nm He-Ne laser as the source excitation, 0.32 m double grating monochromator, and InGaAs detector with conventional lock-in techniques. A closed-cycle optical cryostat from Montana cryostation was used for cooling the samples. Raman spectroscopy was performed in a Horiba Jobin Yvon ARAMIS Raman microscope with a He–Ne laser of 633 nm as the excitation source at room temperature. It is to be noted that all the measurements were performed on the NW ensemble. We have studied three sets each of n-type doped and intrinsic NW samples, where each set corresponds to all three R, AO, and SAO type samples. The ensemble NW sample was cut into four quadrants. Each quadrant was used for different characterization; hence the density variation is minimum among these different quadrants from the same ensemble NW sample. Further, for PL analysis, multiple spots (five on one quadrant) were performed for uniformity in the observed peak position and intensity.

Methods

In this work, three axial self-catalyzed GaAsSb NW samples were grown to investigate the effect of preclean and surface passivation technique in conjunction with *ex situ* thermal ALD deposited Al₂O₃ passivation layer. They include three types of GaAsSb NW samples. The two grown samples are (R) as-grown reference NW and (AO) as-grown with *ex situ* Al₂O₃ passivation layer. The third sample, referred to as SAO, is as-grown GaAsSb NWs submerged initially in HCl aqueous solution (for native oxide removal) followed by 5 min in 1:10 (NH₄)₂S/H₂O diluted solution (for sulfur passivation), immediately blown dried by nitrogen (without rinsing with deionized water to minimize any loss of sulfur from the surface) and quickly transferred to a thermal ALD system for depositing Al₂O₃ passivation layer. All the samples used in this study are listed below in table 1.

Acknowledgments

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

ORCID iDs

Mehul Parakh  <https://orcid.org/0000-0003-0014-7443>

Priyanka Ramaswamy  <https://orcid.org/0000-0002-7692-3907>

Shanthi Iyer  <https://orcid.org/0000-0002-8163-9943>

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