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Outermost AlGaO_x native oxide as a protection layer for GaAs/AlGaAs core-multishell nanowires

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Abstract

We propose native oxide AlGaO_x outer protective layer for GaAs/AlGaAs core-multishell nanowires to provide yearly stable stronger optical and electrical confinement within the nanowire core. We prepared core-multishell NWs consisting of GaAs core, Al_{0.2}Ga_{0.8}As multi-layered barrier layer, and amorphous Al_{0.9}Ga_{0.1}O_x outer shell, which was obtained simply by growing Al-rich AlGaAs and exposing the NWs to the ambient air. Photoluminescence from the NWs reveals that the Al_{0.9}Ga_{0.1}O_x outer shell provides efficient optical confinement and creates a compressive strain in the interior of the NW that enhances and blueshifts the photoluminescence of the GaAs core.

Hybrid nanostructures combining III–V compound semiconductors and oxides form dielectric materials with large bandgaps and small dielectric constants. The strong optical confinement of these structures¹ and their enhanced excitonic properties due to the modified Coulomb interaction^{2,3} make them promising for various optoelectronic applications.^{1–3} In nanowires (NWs), such dielectric confinement can, for instance, improve the room temperature radiative efficiency⁴ and modify carrier transport properties.^{5–7} The complex configurations of NWs, their large surface-to-volume ratio, and the possible integration of III–V compound semiconductors onto Si platforms has attracted significant research interest due to the potential for applications in nanoscale quantum, photonic, electronic, energy-converting, and biological devices.^{8–14}

GaAs, which is one of the most widely used semiconductors in fast electronics and optical devices in the near-infrared, is also of interest as a NW material. However, GaAs suffers from surface effects that are several orders of magnitude greater than those of most other III–V semiconductors,^{15,16} so surface passivation is one of the most critical issues to overcome before GaAs NWs may be used in practical applications. Often, an AlGaAs barrier is used to circumvent the undesired surface effects of GaAs NWs, which allows GaAs/AlGaAs core-multishell NW systems to be used in optical and electronic devices, where their tunable optical characteristics stemming from carrier confinement may be exploited.^{17–26}

In previous work, we developed a method to fabricate (Al,Ga)Ox oxide shells around GaAs nanowires that involves wet oxidation of Al-rich AlGaAs^{27,28} based on a mature oxidation technique for III–V laser processing.^{29,30} However, the heat treatment involved

in this approach may damage the inner structure and thereby degrade the optical properties of the original GaAs NWs^{27,28} (note that this damage occurs before oxidation of the NWs). To overcome this problem, one proposal is to introduce oxides into NWs by native oxidation of AlP segments.³¹ In the present work, we propose a method involving native oxidation by exposure to the ambient atmospheric to fabricate (Al,Ga)Ox outer shells around GaAs/AlGaAs axial core-multishell NWs on Si substrates. The goal of such encapsulation by the outer shell is to provide stronger electrical and optical confinement in the NW core and to exploit the stability of oxides.

To test this approach of native oxidation, GaAs-related NWs were grown on Si (111) substrates by using molecular beam epitaxy (MBE) in vapor-liquid-solid growth mode at 560 °C with Ga as self-catalyst.^{27,32,33} The Ga flux was set to match GaAs 1 monolayer/s planar growth on a GaAs (001) substrate. First, the GaAs core was grown for 15 minutes, following which the growth was interrupted to crystallize the Ga catalyst, which promotes lateral growth to form the shell layers. Next, we grew GaAs/Al_{0.2}Ga_{0.8}As-multiple quantum well/GaAs cap/Al_{0.9}Ga_{0.1}As layers to form core-multishell structures (total growth time was 18 minutes); this approach was expected to enlarge the NW diameter to about 60 nm.³³ The composition-controlled AlGaAs multi-layered structure was formed by using the standard MBE procedure involving shutter control and temperature adjustment of the Al and Ga cells while rotating the substrate.³⁴

The diameter of the GaAs core was about 100 nm. The Al_{0.2}Ga_{0.8}As layer was introduced to serve as a passivation layer and a carrier confinement barrier around the

GaAs core.^{17–26} The outer Al-rich $\text{Al}_{0.9}\text{Ga}_{0.1}\text{As}$ was fabricated so that it could be transformed into an oxide layer by native oxidation.^{27,29,30} The native oxide was formed simply by leaving the NWs exposed to ambient air for a certain time, typically several days. Note that the present investigation was done on a sample exposed to the ambient air for a year, which demonstrates the stability of the sample.

The samples were investigated by scanning electron microscopy (SEM) and cross-sectional scanning transmission electron microscopy (STEM) with energy dispersive x-ray spectroscopy (EDS) on axially sliced single-NW samples prepared by focused-ion-beam processing. Photoluminescence (PL) was measured on the as-grown NW ensembles by using a confocal Horiba Jobin-Yvon HR800 system with 660 nm excitation. The PL of the oxide-coated sample was measured between 77 K and room temperature (RT) and compared with that of the same sample after removal of the oxide shell by immersion in hydrofluoric (HF) acid solution. The excitation power was ~ 1.8 mW. The PL signal was collected by using a $50\times$ objective ($\text{NA} = 0.5$) in a backscattering configuration, dispersed by an 800-mm focal-length monochromator and detected with a thermoelectrically cooled CCD detector. During the performed confocal PL measurements, the detection plane was fixed on the sample surface. Considering the rather high density of the nanowires and the fact that a penetration depth of the 660 nm excitation light (about $0.3\ \mu\text{m}$) is much less than an average NW length ($\sim 2\ \mu\text{m}$), the detected PL signal from the parasitic layer on the substrate should be negligible. To ensure native oxide formation and sample stability, all investigations were done after leaving the sample exposed to the ambient air for over one year.

Figure 1 shows a cross-sectional SEM image of the NW sample that views the edge of the Si substrate. The NWs are preferably aligned in the vertical direction (i.e., normal to the Si substrate). The wire lengths vary significantly, presumably because of the different nucleation time during growth. The NWs are typically 150 nm in diameter and 2 μm long and have straight sidewalls and a faceted tip structure, as commonly observed for GaAs-related NWs.²⁷

FIG. 1. Cross-sectional SEM image of NWs as seen by looking at the edge of the Si substrate.

Figure 2(a) shows axial cross-sectional STEM images of a typical NW acquired in high angular dark-field (HAADF) mode. Figure 2(b) shows the elemental EDS intensity distributions along the line through the NW in Fig. 2(a). The presence of C is due to the supporting film that held the sample during the STEM investigations. Figure 2(c) shows the EDS elemental mappings for Ga, As, Al, and O (left to right). The wire has a hexagonal cross section about 150 nm in diameter. The structural observation and the

EDS elemental distributions suggest the formation of a layered core-multishell structure.^{32,35}

The NW consists of a regular hexagonal GaAs core (90 nm diameter) enclosed by an $\text{Al}_{0.2}\text{Ga}_{0.8}\text{As}$ shell (10 nm thick), a GaAs cap (10 nm thick), and an $\text{Al}_{0.1}\text{Ga}_{0.9}\text{O}$ outer shell (10 nm thick), as targeted by the growth protocol. The outer shell contains low As concentration but a significant concentration of Al and O, which indicates that leaving the sample exposed to the ambient air suffices to oxidize the outer Al-rich $\text{Al}_{0.9}\text{Ga}_{0.1}\text{As}$ shell.^{29,31} The outer shell also shows characteristic structural deformations that tend to extend its vertices, possibly due to the deliquescence of AlGaAs concomitantly with the progression of native oxidation.^{29,36} The inner multishell structure shows negligible oxide concentration, which indicates that the III-As compounds are not affected by the oxidation of the outer shell. The EDS elemental mapping reveals an approximately 10-nm-thick $\text{Al}_{0.2}\text{Ga}_{0.8}\text{As}$ shell around the perimeter of the GaAs core and enclosed by the outer oxide shell [see Fig. 2(b)]. Figure 2(c) also shows Al concentration in this zone. These results are further discussed below based on high magnification cross-sectional HAADF-STEM images.

FIG. 2. (a) Axial cross-sectional HAADF-STEM image of a typical NW. The dashed rectangle shows the area enlarged in Fig. 3. (b) Elemental EDS elemental distributions along the line through the NW in panel (a). (c) EDS elemental mapping for Ga, As, Al, and O (left to right).

Figure 3(a) shows enlarged cross-sectional bright-field (BF) images of the NW vertex shown in the red rectangle in Fig. 2(a), and Fig. 3(b) shows a slightly enlarged HAADF-STEM image of the same area. These images show multi-layered five-period $\text{Al}_{0.2}\text{Ga}_{0.8}\text{As}/\text{GaAs}$ barrier shells, both about 1 nm thick. The lower spatial resolution of the HAADF-STEM image in Fig. 2(a) and of the Al elemental mapping in Fig. 2(c) precludes the detection of the multilayer in these images. In Fig. 3(a), the bright areas in the AlGaAs multilayer image correspond to the dark areas in the HAADF image in Fig. 3(c). The structural modification produces the brighter areas in the BF image, and the light elements produce the darker areas in the HAADF image, which combine to indicate the formation of an $\text{Al}_{0.2}\text{Ga}_{0.8}\text{As}$ layer.

The GaAs/Al_{0.2}Ga_{0.8}As multilayer shows regular lattice fringes with no defects, disorder, or dislocations. Thus, the multilayer structure preserves single-crystalline structural perfection. The lattice fringes disappear outside the multilayer, which indicates an amorphous material in this area. As seen in the STEM and EDS results in Figs. 2(a)–2(c), this amorphous layer is the oxide Al_{0.9}Ga_{0.1}O_x that results from native oxidation of Al_{0.9}Ga_{0.1}As. These results show that the outer native oxide Al_{0.9}Ga_{0.1}O_x is fabricated without degrading the interior single-crystalline GaAs/Al_{0.2}Ga_{0.8}As core-multishell structure.

FIG. 3. (a) Cross-sectional HAADF-STEM image of the area enclosed in the red rectangle of Fig. 2(a). (b) Enlarged HAADF image of panel (a) showing constituent layers. (c) Enlarged BF-STEM image of panel (a) showing constituent layers.

Figure 4(a) shows the RT PL of the as-synthesized NWs with the oxide shell and of the same NWs after etching away the oxide shell by immersion in HF. Both RT PL spectra indicate that the sample is of good optical quality. The PL peak of the as-synthesized (HF-etched) sample is at 858 (873) nm. Notably, the GaAs-related PL peak redshifts when the oxide is removed, which we attribute to elimination of the compressive strain in the GaAs NW core induced by the outer native oxide shell.³⁷ Because oxygen is smaller than arsenic,

the transformation from $\text{Al}_{0.9}\text{Ga}_{0.1}\text{As}$ to $\text{Al}_{0.9}\text{Ga}_{0.1}\text{O}_x$ shrinks the lattice, inducing a compressive strain on the adjacent layers.³⁸ Thus, the wavelength of the PL peak might be tunable by adjusting the strain due to the outer oxide shell. The emission peak is about four times as intense for the as-synthesized sample (i.e., with oxide) than for the HF-etched sample (i.e., without oxide), which suggests that the $\text{Al}_{0.9}\text{Ga}_{0.1}\text{O}_x$ oxide shell improves the optical confinement and thereby enhances the PL efficiency.²⁹

Figure 4(b) shows the 77 K PL for the sample with and without the outer oxide shell. Similar to RT measurements, the PL spectra of both structures contain an intense band caused by the near-band-edge emission from GaAs. The peak position of this emission shifts from 815 nm to 822 nm upon etching away the oxide shell, similar to what occurs at RT [cf. Fig. 4(a)]. Thus, this result is also attributed to effects of strain induced by the outer oxide shell, as discussed above. In addition to the GaAs emission, the spectra also contain a highly structured PL band within the 650 - 800 nm spectral range. In our previous study of oxide NWs made by wet steam oxidation, we observed similar emission from oxide nanoparticles.²⁷ We note that the sharp lines emission at low temperatures can also arise from the AlGaAs shell, due to alloy fluctuations.^{39, 40} The highly structured PL that remains after HF etching (i.e., with no oxide shell) is considered to result from some residual oxide nanoparticles that remain after etching.

The greater PL intensity from the oxide-coated sample suggests that the oxide shell provides optical confinement or carrier protection within the nanowire, which is preferable for its application in optical devices. Figure 4(c) shows the integrated PL intensity as a function of inverse temperature for the sample with the outer oxide shell.

The PL intensity decreases less than 50-fold upon increasing the temperature from 77 K to RT, which is indicative of good PL efficiency from these samples.^{41,42} Over the entire temperature range investigated, the PL intensity from the NW sample with the $\text{Al}_{0.9}\text{Ga}_{0.1}\text{O}_x$ shell is roughly four times that from the NW sample without the oxide shell, which suggests that the $\text{Al}_{0.9}\text{Ga}_{0.1}\text{O}_x$ shell enhances the PL efficiency of the NW core.

FIG. 4. PL spectra at (a) RT and (b) 77 K from the NW sample with AlGaO_x oxide shell and after removal of AlGaO_x oxide shell by HF etching. (c) Integrated near-GaAs-band-edge PL intensity as a function of inverse temperature with the oxide layer.

We propose herein the use of the native oxide AlGaO_x as a protective layer around GaAs/AlGaAs core-multishell NWs to obtain stronger optical and electrical confinement. To test this concept, we prepared core-multishell NWs consisting of the GaAs core, Al_{0.2}Ga_{0.8}As multi-layered barrier layer, and the Al_{0.9}Ga_{0.1}O_x outer shell. The outer oxide shell is obtained simply by growing Al-rich Al_{0.9}Ga_{0.1}As as the outer shell and exposing it to the ambient air. STEM and EDS analyses show that the resulting Al_{0.9}Ga_{0.1}O_x outer shell is amorphous and that the inner Al_{0.2}Ga_{0.8}As multi-layered barrier layer and GaAs core retain a highly regular single-crystalline structure. The NWs with the oxide outer shell preserve their structural and optical properties for over a year, which testifies to their stability. An analysis of the PL spectra reveals that the Al_{0.9}Ga_{0.1}O_x shell provides an effective optical confinement and also creates a compressive strain in the interior of the NW. This allows controllable tuning of the spectral position of the GaAs core emission by changing the thickness of the outer shell.

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