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Ultralow Surface Recombination Velocity in Passivated InGaAs/InP Nanopillars

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Supporting Information

ABSTRACT: The III–V semiconductor InGaAs is a key material for photonics because it provides optical emission and absorption in the 1.55 μm telecommunication wavelength window. However, InGaAs suffers from pronounced nonradiative effects associated with its surface states, which affect the performance of nanophotonic devices for optical interconnects, namely nanolasers and nanodetectors. This work reports the strong suppression of surface recombination of undoped InGaAs/InP nanostructured semiconductor pillars using a combination of ammonium sulfide, (NH₄)₂S, chemical treatment and silicon oxide, SiOₓ, coating. An 80-fold enhancement in the photoluminescence (PL) intensity of submicrometer pillars at a wavelength of 1550 nm is observed as compared with the unpassivated nanopillars. The PL decay time of ~0.3 μm wide square nanopillars is dramatically increased from ~100 ps to ~25 ns after sulfur treatment and SiOₓ coating. The extremely long lifetimes reported here, to our knowledge the highest reported to date for undoped InGaAs nanostructures, are associated with a record-low surface recombination velocity of ~260 cm/s. We also conclusively show that the SiOₓ capping layer plays an active role in the passivation. These results are crucial for the future development of high-performance nanoscale optoelectronic devices for applications in energy-efficient data optical links, single-photon sensing, and photovoltaics.

KEYWORDS: InGaAs, nanopillars, surface passivation, surface recombination velocity

Scaling down optoelectronic devices, including light sources and detectors, to submicrometer sizes is required to achieve small footprint, low energy consumption and ultrahigh speed, as needed for example in energy efficient and low-cost optical interconnects. In the past few years, there has been an explosion of novel nanoscale light sources, such as photonic crystal lasers, metal-dielectric nanocavity lasers, and nanolight-emitting diodes (nanoLEDs), employing III–V compound semiconductor materials as the gain medium. Despite recent efforts, difficulties in achieving efficient nanoscale optoelectronic devices, specifically nanolasers with continuous wave (CW) operation at room temperature (RT) and high output optical power, considerably limits their use in optical communication systems and interconnects. Future on-chip optical interconnects require ultracompact light sources working efficiently (<10 fJ/bit) at Gb/s speeds. Among the many challenges to achieve such requirements, nonradiative processes, specifically large surface recombination rates, have been shown to have a detrimental effect on the efficiency of wavelength- and subwavelength-scale cavity nanolasers and nanoLEDs. At these small scales, surface properties become more important as the surface-to-volume ratio increases. The surface often contains electronically active states because of unsaturated surface bonds (or dangling bonds). As a result, the threshold current of a nanolaser increases because the injected carriers recombine faster, whereas the efficiency of nanoLEDs is compromised due to the predominance of nonradiative recombination. Therefore, methods such as...
passivation that aim at removing these dangling bonds are key approaches in enabling room-temperature and CW operation in nanoscale laser sources and high efficient nanoLEDs.

Among many methods, there has been intense research on core–shell structures to keep minority carriers far from the surface in integrated nanomaterials, such as AlGaAs, GaAsP, and InGaP semiconductor nanowires (NWs),\textsuperscript{15–18} GaAs/AlGaAs core–shell NWs,\textsuperscript{19–22} ultrathin GaP and InP shells on GaAs,\textsuperscript{23} and core–shell InGaAs/InGaP and InP micro/nanopillars.\textsuperscript{17,18,24,25} Such improvements were rapidly followed by the development of more efficient and low-threshold nanolaser devices.\textsuperscript{25,26} Despite the remarkable results, the aforementioned approaches typically require complex epitaxial growth techniques.

In another approach, sulfur treatments were implemented to passivate planar surfaces of III–V compound semiconductors,\textsuperscript{27} namely in GaAs-based devices, revealing a potential to deliver high-performance heterojunction bipolar transistors,\textsuperscript{28} and p-i-n photodetectors.\textsuperscript{29} Following these excellent results, several research groups implemented similar recipes in micro- and nanoscale optoelectronic devices, namely in nanostructure light-emitting diodes,\textsuperscript{30} and photonic crystal nanocavity lasers.\textsuperscript{26,31} The best reported results so far on surface recombination velocity (SRV), $v_s$ include $v_s = 1.5 \times 10^4$ cm/s for nanostructured InGaAs LEDs,\textsuperscript{30} $7 \times 10^3$ cm/s for InGaAsP microcolumns,\textsuperscript{32} and $4 \times 10^3$ cm/s for a InGaAsP photonic crystal nanolaser.\textsuperscript{33} Despite these achievements, the SRV values reported are still too large to achieve efficient and high-power nanoscale light sources, resulting in subnanosecond nonradiative lifetimes when the devices are scaled down to the deep submicrometer size ($< 1 \mu$m).

In this work, we report extremely low surface recombination velocities for submicrometer and micrometer size III–V undoped InGaAs/InP nanopillars. The surface passivation treatment comprises a gentle wet etching of the active material in an ammonium sulfide, (NH$_4$)$_2$S, chemical solution followed by silicon oxide, SiO$_x$, encapsulation by plasma-enhanced chemical vapor deposition (PECVD). In our studies using InGaAs/InP nanopillars, a reproducible, robust, and long-term passivated surface is observed, exhibiting an 80-fold enhancement of the photoluminescence (PL) intensity at a wavelength of 1550 nm on pillars with dimensions ranging from 275 nm to 2.86 $\mu$m. Interestingly, we find that the SiO$_x$ capping layer deposited by PECVD plays a key role in the passivation besides providing protection to oxidation. Time-resolved $\mu$-PL (TRPL) using time-correlated single-photon counting (TCSPC) measurements of the emission dynamics further confirms the substantial improvements, showing up to a 2 orders of magnitude increase of the carrier lifetime, allowing us to estimate an extremely low surface recombination velocity of $\sim 260$ cm/s for the best sample treatments that to our knowledge is a record low value for undoped InGaAs/InP compound semiconductors. Impressively, the achieved values are of the same order of magnitude of semiconductor materials that are well-known to be relatively insensitive to surface states, such as InP.\textsuperscript{33} This is a clear indication of a drastic reduction of nonradiative centers on the sidewalls of the nanopillars investigated here. Most importantly, this remarkable reduction of the surface recombination velocity could be a key enabler in the future development of room-temperature low-threshold nanolasers, more efficient nanoLEDs, and highly sensitive nanoscale detectors.

A systematic experimental study was performed to investigate the effect of ammonium sulfide [(NH$_4$)$_2$S] treatment and of a coating layer on undoped InP/InGaAs/InP nanopillars. The layer stack, shown schematically in Figure 1a, is composed from top to bottom by 350 nm of InP, 350 nm of InGaAs, and 1000 nm of InP, all not intentionally doped, grown on an InP substrate by metalorganic vapor phase epitaxy (MOVPE). The samples are comprised of pillar structures organized in arrays,
Figure 1b, each array with square pillars with dimensions ranging from 275 nm to 2.86 μm width. Figure 1c shows the scanning electron microscope (SEM) picture of a 275 nm wide nanopillar and Figure 1d displays an example of a SEM picture of a micropillar of 1.1 μm width.

The fabrication of the micro- and nanopillars was performed using electron beam lithography (EBL) and dry etching (Supporting Information 1). After etching, one cycle of 10 min oxygen plasma treatment was employed to remove any remaining organic material formed during dry etching followed by a cleaning step in diluted phosphoric acid (Supporting Information 1). For the passivation, the samples were first subjected to a gentle etch in H2SO4, then submerged in a solution of H2O/(NH4)2S (100:10) for 5 min. Finally, for the dielectric coating step a thin capping layer of 50 nm thick SiO2 was deposited by PECVD covering the walls and the top of the pillars (see Supporting Information 2 for full details on the surface passivation and encapsulation steps). In order to investigate the influence of the capping layer thickness, deposition of either a thin (∼5 nm) or a thick (140 nm) layer of SiO2 was also investigated. In all aforementioned cases, the SiO2 deposition was realized using PECVD at 300 °C and was performed either immediately after the ammonium sulfide treatment or 24 h later to check the stability of the sulfur treatment and the potential role of the oxidation of the sulfide layer formed on the sidewalls of the pillars.

We performed CW μ-PL measurements at room-temperature (Supporting Information 3) on single pillars aiming at a comprehensive study of the quality of the surface passivation. Figure 2 presents a typical example of a μ-PL measurement showing the spectral intensity collected on a single nanopillar with 275 nm width for unpassivated, sulfur treatment, and silicon oxide coating passivation steps.

Figure 2. Continuous-wave photoluminescence measurement results at room-temperature displaying a typical μ-PL spectra (log scale in the vertical axis) from a single nanopillar with 275 nm width for unpassivated, sulfur treatment, and silicon oxide coating passivation steps.

dimensions. This poor performance can be attributed mainly to the strong nonradiative surface recombination due to the large surface-to-volume ratios of our nanopillars. Although some improvements were observed in samples using the ammonium sulfide treatment alone (red curve in Figure 2), a remarkable improvement was only achieved using ammonium sulfide treatment followed by encapsulation of the pillars with a 50 nm thick dielectric, SiO2 layer (blue curve in Figure 2). In this case, an 80-fold increase of the intensity spectrum at 1550 nm was measured as compared to the unpassivated pillars. The remarkable boost of the emission was measured in all pillars with dimensions ranging from 275 nm to 2.86 μm side width. The results clearly indicate a drastic reduction of nonradiative recombination centers in the sidewalls of both micro- and nanopillars.

Previous studies of sulfur passivation35 revealed that for the case of the InGaAs material system, the air-exposed surface can be covered by a layer of mixed As, Ga, and In oxides. These native oxides associated with nonradiative recombination defect centers are removed by the sulfur treatment, reducing considerably the surface defect density in the semiconductor and resulting in an enhancement of the PL intensity.35 It was previously suggested that a SiO2 coating layer can protect the passivated surface against subsequent oxidation.31 However, our results indicate that the capping layer also actively contributes to the passivation. Indeed, dielectric deposition performed either immediately after (∼1 min) the ammonium sulfide treatment or after waiting 24 h provides nearly identical PL intensity, similar to that shown in Figure 2 (blue curve). The radiative properties could be better quantified by TRPL measurements (discussed below and shown in Supporting Information Figure S2) revealing that the samples encapsulated after waiting 24 h exhibit only a small reduction in lifetime. Therefore, while exposure to oxygen presumably produces a small degradation of the samples treated with ammonium sulfide in the absence of capping, this effect (at least within 24 h for samples stored in the cleanroom) was not sufficient to substantially impact the passivation process reported here. In contrast, PL measurements without the SiO2 layer show poor PL properties [identical to Figure 2 (red curve)] even when they are carried out immediately after the sulfurization. These results conclusively show that the SiO2 layer not only protects the sulfurized surface from oxidation as reported in ref 31 but actively takes part in the passivation process. It is important to mention that no remarkable improvement was observed after sulfur treatment followed by thermal annealing performed at 300 °C (realized in the same conditions as PECVD was performed), ruling out the possibility that the thermal process is responsible for the PL improvement.

We also tested different deposition conditions and alternative capping layers on identical nanopillar structures. Neither SiO2 deposited by atomic layer deposition (ALD) (H4Si[N-(C2H5)2]3 and O2 plasma34 at 150 °C, 200 °C, and 300 °C, 20 nm thickness) nor Al2O3 by ALD (Al(CH3)3 and H2O, 150 °C, 10 nm thickness), nor SiNx deposited by PECVD (Supporting Information 2) with all films deposited after sulfurization35 provided improvements comparable to those of Figure 2 (blue curve). Possible explanations for the worse performance of ALD SiO2 films include the different chemical precursor used and the longer high-temperature exposure due to the significantly slower ALD deposition process. While it is known that the ALD process produces films with excellent bulk properties, surface passivation is highly sensitive to the
structure and composition of the semiconductor–dielectric interface, and the interface formation process may depend sensitively on hydrogen content, stoichiometry, and density of the PECVD and ALD films. It is possible that the passivation of the ALD SiO$_x$ layers would be improved by the application of a postdeposition annealing treatment. Such an anneal (typically in forming gas) is known to be necessary for achieving passivation in the case of ALD SiO$_2$ on Si. We have not examined the effects of such anneal in the present case. Lastly, no improvements were achieved using SiO$_2$ deposited by PECVD directly after the cleaning treatment without the sulfurization. These systematic investigations confirm that the sulfur treatment combined with SiO$_2$ deposition using PECVD are key steps to achieve a successful passivation.

One possible mechanism for the passivation improvement following SiO$_2$ deposition is hydrogenation of residual interface defects by hydrogen contained in the SiO$_2$ film. This is well-known to contribute to passivation of Si surfaces by, for instance, thermal SiO$_2$ (where in this case the hydrogen is supplied externally$^{36}$). Such a mechanism has also previously been argued to be responsible for improved passivation observed following deposition of PECVD SiN$_x$ on sulfurized GaAs surfaces.$^{37}$ It is likely that it plays a role in the present case as well. However, it is also possible that the SiO$_2$ film additionally participates directly in the formation of interfacial bonding at the InGaAs surface, either supplementing or substituting the existing S-terminated bonds, in such a way as to reduce the density of defect sites. Previous studies of dielectric interface formation on sulfurized GaAs surfaces suggest that such a substitution of S-terminated bonds, possibly involving partial S-desorption, can indeed occur during dielectric deposition at elevated temperatures.$^{38}$ The fact that deposition of other dielectrics known to contain significant hydrogen was not found to result in similar passivation improvements strongly suggests that more than simply hydrogenation is occurring. However, more detailed microscopic investigations will be needed to further test this hypothesis. Besides the hydrogenation of residual interface defects, SiO$_2$ deposited by PECVD is well-known to add strain$^{39}$ and also to be charged,$^{40}$ which can play additional roles in the passivation properties. Regarding the presence of a fixed charge, SiO$_2$ films can contain charge density that may also play a role in the present case to reduce surface recombination, in addition to the reduction of interface states. Concerning to the strain effect, this has been shown to be mainly relevant for the spectral shift of the emission (exhibited for example in ref$^{39}$ for the case of quantum dots). However, no shift in the PL emission peak following SiO$_2$ deposition was observed in our case and therefore the sensitivity to strain in encapsulated nanopillars seems to be not very relevant.

To further characterize the substantial improvements in the surface quality of the nanopillars displayed in the μ-PL results of Figure 2, TRPL measurements (Supporting Information 3 and Figure S1) were performed by employing TCSPC to investigate the carrier dynamics in InGaAs/InP micro- and nanopillars. In the experiments, the pillars were optically pumped using a pulsed laser at 965 nm and the PL, filtered by a bandpass filter of 50 nm full width at half-maximum (fwhm) centered at 1550 nm, was measured by a superconducting single photon detector (SSPD) with a temporal resolution of ∼100 ps (instrument response function in the inset of Figure 3a).

Figure 3. Experimental TRPL decay curves of InGaAs/InP measured at room temperature for (a) 275 nm wide nanopillars and (b) 2.86 μm wide micropillars. For each pillar, their corresponding TRPL intensity curves before passivation (black color) and after sulfur treatment (red color) and silicon oxide coating (blue color) passivation steps are shown. The respective carrier recombination lifetimes are extracted from the measured TRPL curves fitted with a single exponential decay function (red solid curves). The IRF is shown in (a) in green color.

In Figure 3, the measured carrier dynamics are displayed for a 275 nm wide nanopillar, panel a, and for a 2.86 μm wide micropillar, panel b. The TRPL decay data is fitted using a single exponential decay function (solid red curves) to obtain the values of the carrier recombination lifetime. Outstanding improvements in the lifetimes are observed. The 0.275 μm wide nanopillars reveal a carrier lifetime increase by 2 orders of magnitude from 131 ps (black curve in the inset of Figure 2a) before surface passivation to ∼23 ns (blue curve of Figure 2a) after sulfur treatment and dielectric encapsulation, while just small improvements are observed if only sulfur treatment is employed (lifetime of ∼271 ps). The 2.86 μm wide micropillar exhibits an extremely long lifetime after passivation: ∼117 ns.
(blue curve in Figure 3b) as compared to the ~5 ns for the unpassivated sample (black curve in the inset of Figure 3b). Again, the lifetime improvements (~8 ns shown inset of Figure 3b) in the samples using sulfur treatment only are not substantial. Our studies also reveal that just a thin layer of SiOx (nominal thickness of tSiOx ~ 5 nm) deposited using PECVD after the sulfur treatment already provides a substantial improvement of the lifetime, whereas increasing the thickness of the capping beyond 50 nm (we tested thicknesses up to 140 nm) does not alter the already impressive properties of the surface, as shown in Figure 4 where the TRPL for different capping conditions (tSiOx ~5, 50, and 140 nm) in small nanopillars are displayed. We note that the lifetimes in bulk undoped InGaAs can reach values ranging from 100 ns to 1 μs under low excitation conditions due to the bimolecular nature of the radiative recombination.41−43 Hence, very long lifetimes such as the ones reported here can be expected if nonradiative recombination is strongly suppressed. To the best of our knowledge, the lifetimes achieved here are the highest reported to date for undoped InGaAs in both micro- and nanoscale structures.

The implication of such long lifetimes is a drastic reduction of the surface recombination rates in both submicrometer and micrometer size pillars. The measured PL decay time, τPL, is related to the radiative, τr, and nonradiative, τnr, carrier lifetimes by the expression \( \tau_{PL} = \tau_{r}^{-1} + \tau_{nr}^{-1} \), where the nonradiative rate may have a bulk and a surface contribution. Assuming that the surface-related nonradiative recombination rate scales as \( 4 \eta_d/d \), where \( \eta \) is the surface recombination velocity and \( d \) is the width of the pillar with square cross-section, the surface recombination velocity can be estimated directly from the size-dependent measured carrier lifetimes in the low injection regime46

\[
\frac{1}{\tau_{PL}} = \frac{1}{\tau_0} + \frac{4 \eta}{d}
\]

(1)

where \( \tau_0 \) is the carrier lifetime in the bulk material. Figure 5 shows the inverse carrier lifetime estimated from the TRPL measurements versus the inverse pillar width, before and after passivation. The corresponding linear fit (solid red curves) of the experimental data using eq 1 allows us to estimate the corresponding surface recombination velocity, \( \eta_0 \) values.

![Figure 4. Experimental TRPL decay curves measured at room temperature for nanopillars with 275 nm width using different deposited capping thicknesses, tSiOx. The respective carrier recombination lifetimes are extracted from the measured TRPL curves fitted with a single exponential decay function (red solid curves).](image)

![Figure 5. Inverse carrier lifetime, \( \tau_{PL} \), estimated from the TRPL measurements versus the inverse pillar width, \( d^{-1} \), before (black dots, left axis) and after passivation and SiOx (blue triangles, right axis). Also shown is the corresponding linear fit (dashed red curves) of the experimental data using eq 1 allowing us to estimate the corresponding surface recombination velocity, \( \eta_0 \) values.](image)
specifically hydrogenation of residual interface defects and the possible presence of a fixed charge, further detailed microscopic investigations are needed to confirm these hypotheses. The ultralow surface recombination rates achieved here are a key enabler for the future development of room-temperature nanoscale optoelectronic devices for applications in nanophotonic integrated circuits, energy-efficient data optical links, single-photon sensing, and photovoltaics.

**ASSOCIATED CONTENT**

1. Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.nanolett.7b00430.

Fabrication of the nanopillars. Surface passivation and dielectric deposition. Optical characterization setup. Schematic of the time-correlated single-photon counting experimental setup. Experimental time-resolved PL decay curves. Internal quantum efficiency (PDF)

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**Notes**

The authors declare no competing financial interest.

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