Photocorrosion metrology of photoluminescence emitting GaAs/AlGaAs heterostructures

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Photocorrosion metrology of photoluminescence emitting GaAs/AlGaAs heterostructures

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Abstract
High sensitivity of the photoluminescence (PL) effect to surface states and chemical reactions on surfaces of PL emitting semiconductors has been attractive in monitoring photo-induced microstructuring of such materials. To address the etching at nano-scale removal rates, we have investigated mechanisms of photocorrosion of GaAs/Al$_{0.35}$Ga$_{0.65}$As heterostructures immersed either in deionized water or aqueous solution of NH$_4$OH and excited with above-bandgap radiation. The difference in photocorrosion rates of GaAs and Al$_{0.35}$Ga$_{0.65}$As appeared weakly dependent on the bandgap energy of these materials, and the intensity of an integrated PL signal from GaAs quantum wells or a buried GaAs epitaxial layer was found dominated by the surface states and chemical reactivity of heterostructure surfaces revealed during the photocorrosion process. Under optimized photocorrosion conditions, the method allowed resolving a 1 nm thick GaAs sandwiched between Al$_{0.35}$Ga$_{0.65}$As layers. We demonstrate that this approach can be used as an inexpensive, and simple room temperature tool for post-growth diagnostics of interface locations in PL emitting quantum wells and other nano-heterostructures.

Keywords: photocorrosion, GaAs/AlGaAs quantum wells, AFM, x-ray photoelectron spectroscopy, metrology of heterostructures

(Some figures may appear in colour only in the online journal)
et al 2011), monolayer chemical beam etching (Tsang et al 1993) and x-ray photoelectron spectroscopy (XPS) (Vilar et al 2005, Hinkle et al 2009). Furthermore, current–voltage ($I-V$) and capacitance–voltage ($C-V$) measurements (Fleetwood et al 1993) have been employed to investigate device band structure (Watanabe 1985) and provide information about carrier concentration and distribution by employing electrochemical (Kaniewska and Slomka 2001) and photo-electrochemical profiling (Blood 1986). The thickness of layers constituting heterostructure devices is one of the fundamental parameters determining functioning characteristics of quantum well (QW) and other quantum confined microstructure based devices. Typical methods for determining this parameter are based on cross-section SEM or TEM imaging (Perovic et al 1995), ellipsometric analysis (Erman et al 1983), depth profiling with Auger electron spectroscopy (Dubowski et al 1985), SIMS (Liu et al 2013) and x-ray diffraction techniques (Tapfer and Ploog 1986). Electrochemical and photo-electrochemical profiling techniques have been employed to study multi-junction devices due to the ease in revealing location of different interfaces. However, relatively high etch rates induced in such experiments prohibit their application in studying nano-scale interfaces, such as those involving QWs and quantum dots (Fink and Osgood 1993). Nevertheless, selective etching of cleaved wafers revealed interfaces between ~60 nm thick pairs of GaAs and AlGaAs layers using the AFM technique (Pakhomov et al 2002). Typical etch rates of GaAs and InP achieved with a laser-induced photoetching technique have been reported between 0.1–100 µm min$^{-1}$ (Ruberto et al 1991), while fabrication of relatively smooth surfaces of GaAs has been demonstrated with reactive ion etching in Cl$_2$/BCl$_3$/Ar plasma at rates exceeding 0.3 µm min$^{-1}$ (Nordheden et al 1999). Generally, surface morphology of etched or photo-etched semiconductors is characterized by average roughness exceeding 15 nm (Kirchner et al 2002) and, thus, such techniques cannot be readily employed to provide information about location of interfaces in QW or quantum dot heterostructures.

We have recently employed the photocorrosion effect of photoluminescence (PL) emitting GaAs/AlGaAs heterostructures for monitoring surface reactions involving electrically charged molecules that allowed for a rapid detection of bacteria in aqueous solutions (Nazemi et al 2015, Aziziyan et al 2016). This was possible thanks to creating conditions for uniform photocorrosion proceeding at rates typically not exceeding ~60 nm h$^{-1}$. The potential problem of As, Ga and Al ions released during photocorrosion that could interfere with the growth of bacteria has been discussed by us in a recently published paper (Nazemi et al 2016). In the current work, we investigate the mechanisms of GaAs and AlGaAs photocorrosion in water and aqueous environment of NH$_4$OH, and we examine the conditions leading to the high-resolution resolving of different material layers contributing to PL emission.

2. Experimental section

2.1. Materials and chemicals reagents

GaAs/AlGaAs heterostructures were grown on double side polished semi-insulating GaAs (100) by molecular beam epitaxy (Wafers 10-150 and v0803) and by metalorganic chemical vapor deposition (Wafer 10-413). A 200 nm thick epitaxial GaAs, as well as a superlattice with 2.4 nm thick AlAs and 2.4 nm thick GaAs were grown in each case on GaAs substrates as defect reduction buffer layers (Dawson and Woodbridge 1984, Noda et al 1990). A schematic cross-section of the Wafer 10-150 microstructure is shown in Figure 1. 

Figure 1. Schematic cross-sections of the investigated nano-heterostructures. Photoluminescence in the 10-150 sample originates from a 500 nm thick GaAs layer (a), and is centered at 869 nm (b). Photoluminescence in the 10-413 sample originates from a stack of 6 nm quantum wells (c), and is centered at 829 nm (d). Photoluminescence in the v0803 sample originates from a 500 nm thick GaAs layer (e), and is centered at 869 nm (f).
figure 1(a). It consists of a 500 nm thick GaAs epitaxial layer designed to provide PL emission at ~869 nm. Additionally, a stack of 100 nm thick Al_{0.35}Ga_{0.65}As, 3 nm thick GaAs and 10 nm thick Al_{0.35}Ga_{0.65}As, capped with an 8 nm thick GaAs layer, was grown on top of the PL emitting GaAs layer. The room-temperature PL emitted by this microstructure is shown in figure 1(b). Note that due to the quantum confinement effect, the 3 nm thick GaAs QW emits PL at low-temperatures as reported in Aziziyan et al (2016), but no such emission could be observed at room temperature. A cross-section of the Wafer 10-413 microstructure is illustrated schematically in figure 1(c). It comprises a stack of 29 GaAs QWs (6 nm thick) surrounded by 10 nm thick Al_{0.35}Ga_{0.65}As barriers grown on a 200 nm thick GaAs buffer layer. This microstructure is capped with a 10 nm thick layer of GaAs. The 829 nm PL emission of this microstructure, as illustrated in figure 1(d), originates from the stack of GaAs QWs, while a high-energy shoulder at ~820 nm, most likely, is due to a free-exciton recombination observed in high-quality GaAs/AlGaAs multi-QW microstructures (Dawson et al 1983, Chemla et al 1984). A cross section of the Wafer v803 microstructure is illustrated schematically in figure 1(e). A 500 nm Al_{0.35}Ga_{0.65}As, overlaid on a 500 nm thick PL emitting epitaxial GaAs layer, forms the base of the heterostructure. There are eleven units of GaAs and Al_{0.35}Ga_{0.65}As grown on the base. The thicknesses of GaAs of these units are 60, 50, 40, 30, 20, 10, 8, 6, 4, 2 and 1 nm for the top layer. The thickness of Al_{0.35}Ga_{0.65}As is 20 nm in each of the eleven layers and the whole structure is capped with a 20 nm thick GaAs layer. A plot of the PL emission at 869 nm that originates from the 500 nm thick GaAs layer in this microstructure is shown in figure 1(f). We have also employed bulk Si-doped \((n = 10^{17} \text{ cm}^{-3})\) GaAs (AXT, Inc., Fremont, USA) while investigating oxide formation on samples exposed to de-ionized (DI) water.

Semiconductor grade OptiClear (National Diagnostics), acetone (ACP Chemicals, Canada), isopropyl alcohol and 28% ammonium hydroxide (Anachemia, Canada) were used without further purification. A MilliQ system was used to obtain DI water with a resistivity of 18.2 MΩ·cm. The wafers were spin coated with photoresist (S1813, Shipley), mounted on a carrier tape and diced into 2 mm × 2 mm and 4 mm × 4 mm samples.

2.2. PL measurements

A custom designed quantum semiconductor photonic biosensor (QSPB) reader or hyperspectral imaging photoluminescence mapper (HIPLM) described by Nazemi et al (2015) and Aziziyan et al (2016), and Kim et al (2009), respectively, were used to collect PL data. The above bandgap excitation in QSPB was provided either by a 625 nm LED source or by a halogen lamp. An 812 nm cut-off long pass filter (Thorlabs FELH800) was applied in the QSPB reader to prevent the excitation photons from reaching the CMOS detector employed for detecting PL emission. The HIPLM is equipped with a laser emitting at 532 nm, computer controlled volume Bragg gratings, beam homogenizing microlens array and a Peltier cooled high sensitivity CCD camera. The HIPLM allows measurement of spatial-temporal variation of the PL emission from GaAs/AlGaAs heterostructures (Kim et al 2009). The PL measurements were carried out for samples installed in a flow cell as illustrated schematically in figure 2.

The flow cell allows a continuous exposure of samples to the photocorrosion supporting solutions that could be refreshed continuously. A dedicated glass window facilitates excitation of samples and collection of PL data. The excitation power density measured at the sample surface in both QSPB and HIPLM systems ranged between 20–150 mW cm\(^{-2}\). The power emitted by LED was continuously monitored with a Si photodiode. Typically, during a 4 h run, the LED emitted power was stable to within ±1%. In all the measurements, the samples were excited with intermittent pulses to allow (a) formation and dissolution of a sufficient thickness surface oxides, and (b) diffusion of the photocorrosion products and disperse into the solution between consecutive pulses. The intermittency was defined by a ‘duty cycle’ (DC) parameter: \(T_{\text{ON}}/(T_{\text{ON}} + T_{\text{OFF}})\). The experiments were performed in an open circuit condition. While photocorrosion in an aqueous NH\(_2\)OH environment is expected to result in a continuous formation and removal of GaAs and AlGaAs oxides, the photocorrosion in DI water could lead to accumulation of some oxides at the surface of the irradiated GaAs/AlGaAs samples. This effect, however, seems negligible in view of the well-resolved PL runs observed in DI water that we report in section 3.2.

2.3. AFM measurements

A Nanoscope IIIa (Digital Instruments, Inc.) was used to carry out AFM study of surface morphology of the investigated samples. Scans of 5 \(\mu\text{m} \times 5 \mu\text{m}\) were collected in a tapping mode at ambient conditions. The photocorrosion rates were determined by measuring depths of photocorroded craters. For this, we used 4 mm × 4 mm samples of the 10-413 heterostructure, partially covered with a positive photoresist. The samples were briefly cleaned with isopropanol, placed in the flow cell with DI water and photocorrosion performed for different periods of time. The AFM measurements were performed for samples with removed photoresist, in the areas covering both non-photocorroded and photocorroded surfaces.
mounted in a chamber with the base pressure of 1 Torr. Survey and high-resolution scans were performed for samples peak on the photocorroded surfaces. Both surface the Al 2p region to investigate chemical composition of photocorroded microstructures. The measurements were carried out with a Kratos Analytical AXIS Ultra DLD XPS spectrometer equipped with Al Kα source operating at 150 W. Following the photocorrosion experiments, the samples were de-oxidized for 5 min in a 28% aqueous solution of NH₄OH, dried with gas N₂ and transported in the N₂ ambient to the XPS chamber. The de-oxidization procedure was necessary to remove the surface accumulated oxide layer whose thickness exceeded ~10 nm as evidenced by the absence of the Al 2p peak on the photocorroded surfaces. Both surface survey and high-resolution scans were performed for samples mounted in a chamber with the base pressure of 1 × 10⁻⁹ Torr. The size of an analyzed area on the investigated samples was set at 220 μm × 220 μm and data were collected at a takeoff angle of 60° from the surface normal. All XPS results, referenced to the adventitious C 1s peak at the binding energy (BE) of 285.0 eV, were processed using the Casa XPS 2.3.15 software.

2.4. X-ray photoelectron spectroscopy measurements

X-ray photoelectron spectroscopy (XPS) measurements were conducted in the Al 2p region to investigate chemical composition of photocorroded microstructures. The measurements were carried out with a Kratos Analytical AXIS Ultra DLD XPS spectrometer equipped with Al Kα source operating at 150 W. Following the photocorrosion experiments, the samples were de-oxidized for 5 min in a 28% aqueous solution of NH₄OH, dried with gas N₂ and transported in the N₂ ambient to the XPS chamber. The de-oxidization procedure was necessary to remove the surface accumulated oxide layer whose thickness exceeded ~10 nm as evidenced by the absence of the Al 2p peak on the photocorroded surfaces. Both surface survey and high-resolution scans were performed for samples mounted in a chamber with the base pressure of 1 × 10⁻⁹ Torr. The size of an analyzed area on the investigated samples was set at 220 μm × 220 μm and data were collected at a takeoff angle of 60° from the surface normal. All XPS results, referenced to the adventitious C 1s peak at the binding energy (BE) of 285.0 eV, were processed using the Casa XPS 2.3.15 software.

3. Results and discussion

3.1. PL monitored photocorrosion of bulk n-GaAs

A 2 mm × 2 mm bulk n-GaAs sample, after cleaning with OptiClear, acetone and isopropanol, and deoxidizing with NH₄OH was quickly transferred to the flow cell with DI water. The sample was irradiated with the broadband halogen lamp and its PL emission was measured with the QSPB reader. The excitation intensity was 43 mW cm⁻², and the sample was continuously exposed to the radiation (DC = 1, T₀FF = 0). A temporal evolution of the PL signal measured at 869 nm is shown in figure 3.

It can be seen that PL intensity increases monotonically with time, exhibiting a 10% increase after 18 h and a tendency towards asymptotic saturation. The origin of this behaviour, referred to as photowashing (Wilmsen et al 1988, Choi et al 2002), can be traced down to oxidation of the GaAs surface and photocorrosion (Hideki et al 1988, Geisz et al 1995).

The PL emission from a photoexcited semiconductor is a result of the recombination of excited charge carriers, and its efficiency is an index of the lifetime of the minority carriers (holes for an n-type semiconductor): the increase of their lifetime results in the increased PL emission. Minority carrier lifetime, τ, could be described by the following equation (Ahrenkiel et al 1989):

\[
\frac{1}{\tau} = \frac{1}{\tau_R} + \frac{1}{\tau_{SRV}} + \frac{2SRV}{d}
\]  

where \( \tau_R \) is the radiative recombination lifetime, \( \tau_{SRV} \) the non-radiative recombination lifetime, SRV denotes the surface recombination velocity, and \( d \) is the depletion width. With the exception of SRV and \( d \), the other components in this equation are bulk parameters of the semiconductor. The SRV represents the non-radiative recombination component due to the break in the translational symmetry of the semiconductor at its surface, which results in the formation of surface states with energy levels in the band gap, and formation of the depletion width region in the semiconductor. Decreased SRV increases the minority carriers lifetime, and hence the efficiency of PL emission. Furthermore, it is known that the oxidation and photo-decomposition of n-type GaAs could be described by the following equations (Ruberto et al 1991):

\[
\text{GaAs} + \text{H}_2\text{O} + 6h^+ \rightarrow \text{Ga}^{3+} + \text{HAsO}_2^- + 3\text{H}^+
\]  

(2)

\[
\text{GaAs} + 3e \rightarrow \text{Ga}^0 + \text{As}
\]  

(3)

where \( h^+ \) represents the holes in the semiconductor and \( H^+ \) is the proton in the solution, \( e \) represents the electron and \( \text{Ga}^0 \) denotes a reduced gallium atom. The water environment and excited holes arriving at the semiconductor surface lead to oxidation, and the reactions represented by equations (2) and (3) describe oxidation and dissolution of GaAs upon photoexcitation. As As-oxides are preferentially removed in an aqueous solution (Schwartz et al 1979), the increased presence of Ga-oxides (Ga₂O₃) develops at the GaAs-solution interface. It has been demonstrated that capping of GaAs with a Ga₂O₃ film results in the reduction of the SRV of minority carriers and, consequently, in the increased PL emission intensity (Passlack et al 1995, Priyantha et al 2011). In agreement with these results, and with our XPS data, we argue that it is the predominant formation of a Ga₂O₃ layer and partial dissolution of As-oxides that are responsible for the increasing PL signal observed in figure 3. The results reported in that figure were obtained with a white light source, but the same behaviour is expected to take place as long as GaAs is irradiated with photons of energy exceeding the bandgap energy of this material.
3.2. PL monitored photocorrosion of nano-heterostructures

Examples of temporal plots of PL emission from GaAs/AlGaAs samples (Wafer 10-150) exposed in the flow cell to continuously flowing DI water and irradiated in the HIPLM system with a CW 532 nm laser at 70 mW cm\(^{-2}\) are shown in figure 4. These experiments were designed to investigate the influence of the irradiation conditions on the average photocorrosion rate of studied microstructures. It can be seen that 1 s irradiation in each 100 s period (DC\(_{100}=0.01\)) allows observing the formation of two PL maxima at 80 and 275 min, followed by a slowly decaying signal.

Similar maxima are observed for the samples irradiated for 1 s in a 10 s period (DC\(_{10}=0.1\)). But, in this case, the maxima occur at 15 and 50 min, i.e. they form significantly sooner and they are separated by only 35 min, in comparison to the 195 min separation observed for the sample processed with DC\(_{100}=0.01\). The initial increase of the PL signal observed in these experiments suggests a similar mechanism of photocorrosion as in the case of bulk GaAs discussed in figure 3. The number of PL maxima observed for low duty cycle experiments (DC\(_{10}=0.1\) and 0.01) coincide with the number of GaAs–Al\(_{0.35}\)Ga\(_{0.65}\)As interfaces in this nano-heterostructure. Furthermore, the region of a slowed down PL decay is observed for DC = 0.01 near 125–140 min, and for DC = 0.1 near 25–40 min. We link this behaviour with a reduced rate of photocorrosion of the 10 nm thick Al\(_{0.35}\)Ga\(_{0.65}\)As layer. For DC = 0.5, the first maximum occurs around 12 min, which suggests an accelerated rate of photocorrosion. Consistent with this was the inability to observe the formation of a second PL maximum.

In figure 5, we plot temporal positions of the first maximum observed for the sample 10-150 as a function of the excitation power density, \(P\), for DC = 0.01.

It can be seen that the position of this maximum occurs more rapidly with increasing excitation power density, and it depends linearly on \(P\). Thus, the related photocorrosion rate is consistent with the kinetics of a process driven entirely by the availability of photo-excited holes. At \(P > 105\) mW cm\(^{-2}\), a departure from the linear dependence took place (data not shown), showing significantly delayed positions of the investigated PL maxima. This seems to suggest the onset of a mechanism affected by a greater rate of oxide formation than dissolution.

In figure 6(a), we present a temporal plot of PL signal measured at 869 nm from the v0803 sample that undergoes photocorrosion in a 28% NH\(_4\)OH aqueous solution. The sample was irradiated in the HIPLM system with a 532 nm laser delivering 25 mW cm\(^{-2}\) irradiance at DC = 0.5 (30 s/60 s). In this experiment, we replaced DI water with an aqueous solution of NH\(_4\)OH to provide a more efficient means of removing As- and Ga-oxides from photocorroding surface. Such treatment has been reported to leave only a small amount of Ga-suboxides on the photocorroding surface of GaAs (Lebedev et al. 2004).

A series of PL maxima, denoted as 1–11, can be distinguished in that figure. Initially, after reaching the first maximum, the PL intensity drops by about 60%. This is followed by the PL signal modulating between less than 5% for the second maximum (2), to about 30% for the ninth maximum (9). The oxidation and subsequent removal of oxides lead to a continuous thinning of the semiconductor microstructure and switching in situ between interfaces involving GaAs-solution and Al\(_{0.35}\)Ga\(_{0.65}\)As-solution. Figure 6(b) demonstrates a relationship between the thickness of this microstructure and temporal positions of PL maxima. The thickness was extracted from the known growth parameters of this microstructure (see figure 1(e)), and an assumption was made that each maximum coincides with a completely etched GaAs layer and the photocorrosion onset of an AlGaAs layer. It can be seen that there is a reasonable linear correlation between the cumulative microstructure thickness and the number of a consecutive PL maximum. Based on this model, we estimated that the average photocorrosion rate of the microstructure was
We note that the ability to resolve a 1 nm thick GaAs layer in this experiment was a consequence of the proper adjustment of the duty cycle and the power of LED employed for e–h+ excitation. Generally, the resolution of the process is expected to also depend on the corroding strength of an employed electrolyte and the physical properties of investigated nano-heterostructures.

3.3. AFM depth measurements

AFM measurements were performed for samples 10-413 that photocorroded in DI water. The samples were irradiated with a 625 nm LED at 20 mW cm\(^{-2}\) and DC = 3 s/40 s. The PL runs of these samples are shown in figure 7. Similarly to the results reported in figure 4, the initial increase of a PL signal and formation of the 1st PL maximum are related to GaAs oxidation, dominated by formation of Ga2O3 and, ultimately, dissolution of a 10 nm thick GaAs cap at the S1 point. This behaviour is reproduced during dissolution of a 6 nm thick GaAs and formation of the second PL maximum expected near the S3 point. We note that the initial attempts to AFM analyse photocorroded craters were unsuccessful due to the presence of a thick oxide layer covering the irradiated surface. It was only after subjecting the samples to ammonium hydroxide etching that we were able to observe clear contrast between non-irradiated (photoresist protected) and photocorroded surface. The results of these measurements are summarized in table 1.

The estimated depth for the end-point S1 is 9.8 nm, hence the photocorrosion has consumed most of the 10 nm thick GaAs cap layer of that sample. For the end-point S2, the estimated depth is 15.7 nm, hence the 10 nm GaAs cap layer and 5.7 nm of the first 10 nm AlGaAs layer have photocorroded. For the end-point S3, the estimated depth is 26.7 nm, which indicates that in addition to the entire GaAs cap and the first AlGaAs layer, the second GaAs (QW) layer have photocorroded. These results suggest that the initial PL increase, until the end-point S1 is reached, coincides with the dissolution of a GaAs cap layer. The decrease of a PL signal leading to the end-point S2, coincides with dissolution of the first AlGaAs layer, while the subsequent increase of the PL signal leading to the end-point S3, coincides with the second GaAs layer dissolving entirely. It has been demonstrated that capping of GaAs with a Ga2O3 film results in the reduction of the SRV of minority carriers and, consequently, in the increased PL emission intensity (Passlack et al 1995, Priyantha et al 2011). In agreement with these results, and with our XPS data, we argue that it is the predominant formation of a Ga2O3 layer and partial dissolution of As-oxides that are responsible for the increasing PL signal observed in figure 7.
3.4. XPS measurements

High resolution XPS scans in the Al 2p peak region for a reference (non-photocorroded) sample 10-413, and for a series of 10-413 samples photocorroded for different period of time, together with the associated PL plots collected in the QSPB reader during their photocorrosion are shown in figures 8(a) and (b), respectively. As the photocorrosion proceeds through the GaAs/Al0.35Ga0.65As interface, the XPS determined concentration of Al aluminium increases. Hence, evolution of the Al 2p peak could be used to investigate depth of the photocorroded material, keeping in mind that a typical XPS depth resolution approaches 10 nm (Dallera et al 2004).

Sample 1 that photocorroded for 9 min shows a small intensity peak in the region of the elemental Al 2p, suggesting that it originated from the GaAs capped AlGaAs layer. Sample 2 that photocorroded for 25 min, and revealed the first PL maximum, shows an increased concentration of the elemental Al 2p. In addition, the presence of some Al 2p oxides in this case suggests a contribution from the AlGaAs layer exposed by photocorrosion. Samples 3 and 4 that photocorroded for 40 and 65 min show decreasing concentration of the elemental Al at the expense of Al-oxides, which is consistent with the increased contribution from the photocorroding AlGaAs layer. Finally, sample 5 that photocorroded until it revealed the second PL maximum, shows an increased concentration of Al originating from the second AlGaAs layer.

The XPS and AFM data confirmed the origin of PL features revealed during photocorrosion of the GaAs/AlGaAs nano-heterostructures. The formation of the first PL maximum is consistent with the photocorrosion of GaAs through the formation and dissolution of Ga-oxides, similarly to the photocorrosion of n-GaAs discussed in section 3.1. Furthermore, the revealing of the AlGaAs surface is associated with the formation of Al-, As- and Ga-oxides. This affects the intensity of PL signal emitted by the microstructure. The SRV of minority carriers at the Ga-oxide covered GaAs surface is of the order of 4000–5000 cm s\(^{-1}\), whereas that at the Al-oxide covered AlGaAs surface increases to \(\sim 10^7\) cm s\(^{-1}\) (Passlack et al 1996). Hence the formation of PL maxima in these experiments is consistent with the onset of the photocorrosion of AlGaAs, each time the GaAs layer is dissolved, while a slowed down decay of the PL signal (slower photocorrosion rate) is associated with a reduced concentration of holes available at the surface of AlGaAs due to the significantly increased SRV of minority carriers.

### Table 1. AFM determined depths of the craters fabricated by photocorrosion of GaAs/AlGaAs QWs (Wafer 10-413) illustrated in figure 7.

<table>
<thead>
<tr>
<th>End point</th>
<th>Duration of photocorrosion (min)</th>
<th>Depth (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>S1</td>
<td>12</td>
<td>9.8</td>
</tr>
<tr>
<td>S2</td>
<td>107</td>
<td>15.7</td>
</tr>
<tr>
<td>S3</td>
<td>124</td>
<td>26.7</td>
</tr>
</tbody>
</table>

*Note: the samples were irradiated with a 625 nm LED at 20 mW cm\(^{-2}\) and DC = 0.075 (3 s/40 s).*

4. Conclusions

We have investigated photocorrosion of GaAs/Al\(_{0.35}\)Ga\(_{0.65}\)As nano-heterostructures immersed in DI water and aqueous solution of NH\(_2\)OH. The pulse excitation of such samples with the above bandgap radiation produces photoluminescence of intensity that could be correlated with the sequential formation in situ of different electrochemical junctions. For high DC, defined as the ratio of \(T_{ON}/(T_{ON} + T_{OFF})\), and for the excitation power densities exceeding 105 mW cm\(^{-2}\), the photocorrosion
rates are relatively large, resulting in a poor depth resolution of the process. Under optimized irradiation conditions, described by $P \leq 105 \text{ mW cm}^{-2}$ and $D < 0.5$, the photocorrosion process proceeds at rates of less than 0.2–0.4 nm min$^{-1}$. Such conditions enabled us to observe dissolution of a 1 nm thick layer of GaAs. A linear correlation between photon flux and corrosion rates observed under these conditions is consistent with a pure photo-induced corrosion process. Due to different surface recombination velocities of minority carriers, oxides at the GaAs surface tend to increase PL emission from the GaAs/AlGaAs microstructures, while oxides at the AlGaAs surface reduce that emission. We have demonstrated that PL monitored photocorrosion of the GaAs/AlGaAs heterostructure could be used for post-growth metrology of such architectures with at least a 1 nm depth resolution. The ability to control photodecomposition of PL emitting III–V nano-heterostructures has the potential application for the fabrication of multibandgap devices requiring the regrowth on precisely revealed surfaces prepared, e.g. for the realization of so-called butt joint integration architectures.

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