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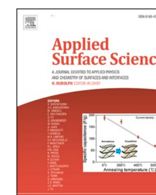

Full Length Article

Effect of H_2O_2 and H_2O immersions on epitaxial GaInP-GaAs interfaces: Photoluminescence and x-ray photoelectron study

Zahra Jahanshah Rad [a,*], Mikko Miettinen [a], Johanna Laaksonen [a], Perttu Piispanen [a], Marko Punkkinen [a], Kalevi Kokko [a], Pekka Laukkanen [a,*], Antti Tukiainen [b], Heidi Tuorila [b], Helmer Piirilä [b], Jukka Viheriälä [b], Mircea Guina [b]

[a] *Department of Physics and Astronomy, University of Turku FI-20014 Turku, Finland*
[b] *Optoelectronics Research Centre, Tampere University, FI-33720 Tampere, Finland*

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ABSTRACT

Epitaxially grown lattice-matched GaInP on a GaAs crystal is a common part in semiconductor devices such as bipolar junction transistors and space solar cells. Due to the larger band gap of GaInP, it provides also high-quality passivation for GaAs surfaces. Therefore, the photoluminescence (PL) intensity measured from GaInP-capped GaAs is among the strongest intensities obtained from GaAs crystals having different surface passivation layers. Here we demonstrate that a facile wet chemical treatment, including immersions in two solutions: first in hot hydrogen peroxide (H_2O_2) and then in hot water (H_2O), increases the PL intensity from epitaxial GaInP/GaAs. Concomitantly, the GaInP surface is further oxidized according to x-ray photoelectron spectroscopy results. Particularly, arsenic impurities and indium at the surface become oxidized. Finally, the $H_2O_2 \rightarrow H_2O$ treatment combination is used to modify the n-type GaAs contact layer (150 nm), deposited on the top of epitaxial GaInP/GaAs such that the PL intensity from GaAs increases by factor of two as compared to PL from the high-quality GaInP/GaAs reference. The $H_2O_2 \rightarrow H_2O$ treatment is discussed to transform an initial n-type GaAs contact layer, which degrades the PL intensity, to an antireflective and less absorptive layer which resembles a recently reported black GaAs surface.

1. Introduction

GaInP/GaAs-based heterojunction bipolar transistors are industrial semiconductor devices which have several application areas such as telecommunication, space, and defence technologies [e.g. Refs. [1–6]]. An epitaxially grown GaInP/GaAs junction is also the key part of high-efficiency and durable multijunction solar cells [e.g. [7–10]]. When a composition of the film is near $Ga_{0.5}In_{0.5}P$, its lattice constant is well matched with the GaAs substrate lattice, which enables the growth of a highly crystalline GaInP/GaAs junctions. A difference in the band gaps of GaAs (1.4 eV) and $Ga_{0.5}In_{0.5}P$ (1.9 eV) provides a barrier for the electric carriers in the GaAs side (Fig. 1), which decreases the carriers' density near the GaInP surface area that is often defect rich. In other words, epitaxial $Ga_{0.5}In_{0.5}P$ films improve the surface passivation of GaAs crystals significantly, even if $Ga_{0.5}In_{0.5}P$ does not provide an insulating barrier. The improved passivation level is expected to arise partly from specific surface properties of GaInP. Namely, it has been previously found that harmful non-radiative recombination at native-oxide covered InP surfaces is clearly smaller than at many other III–V surfaces (e.g. at oxidized GaAs surfaces) [11–14]. That difference has been observed for instance in photoluminescence (PL) measurements, where part of the photon-induced extra carriers can readily diffuse toward surface areas of a semiconductor crystal. When the surface is not well passivated, many carriers recombine in non-radiative manner via surface defects, decreasing the total PL intensity [15–20].

Indeed, strongest GaAs PL intensities have been measured from the epitaxial heterostructures where a GaAs surface is covered by so-called window layer like GaInP, AlInP or AlGaAs with a larger band gap than the GaAs one [e.g. [18–20]]. Because these lattice-matched junctions have a highly crystalline interface structure, a density of point defects and therefore also a density of defect-induced electron levels in the band gap are decreased. At many other GaAs interfaces, structural disorder is typically increased causing the formation of many point defects at GaAs surfaces, for example, due to uncontrolled oxidation of GaAs surfaces

* Corresponding authors.
E-mail addresses: zahra.s.jahanshahrad@utu.fi (Z.J. Rad), pekka.laukkanen@utu.fi (P. Laukkanen).

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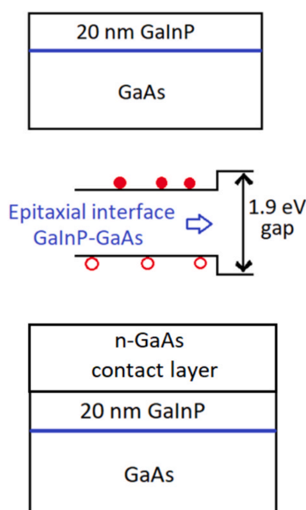

Fig. 1. Schematic surface structures for the samples studied in this work. The lattice-matched GaInP cap layer (top panel) provides an energy barrier for the electron and hole carriers (middle panel). GaAs band gap is 1.4 eV approximately. So-called n-GaAs contact layer (bottom panel) is used in many devices to manufacture the Ohmic metal contacts. This GaAs contact film is often etched away selectively between the metal contacts because it degrades the optical properties.

during the interface manufacturing processes [21]. Of course, the heteroepitaxial junctions like GaInP/GaAs contain also the outermost surface region which is defect rich as compared to the bulk crystal region. Moreover, part of the electric carriers overcomes the interface energy barrier, and some light absorption occurs also in the window layer. Therefore, non-radiative surface recombination happens also in the epitaxial junctions. It is still unclear why the III–V phosphide surfaces are not so prone to the non-radiative recombination [14].

To elucidate this issue, in this work we have studied effects of hot hydrogen peroxide (H_2O_2) and water (H_2O) immersions on the properties of epitaxial $Ga_{0.5}In_{0.5}P$/GaAs (hereafter only GaInP/GaAs) by photoluminescence (PL) and x-ray photoelectron spectroscopy (XPS) measurements. We have used similar treatments to that reported recently to cause an antireflective black GaAs surface [22]. Here we do not find any significant change in the surface reflectivity of GaInP, but still the GaAs PL intensity is increased due to proper treatments. Concomitantly, XPS spectra reveal changes in oxidation states which are discussed together with PL results. Finally, we present PL observations for the GaInP/GaAs junction, that is still covered by n-type GaAs, after hot H_2O_2 and H_2O immersions, which clarify also non-radiative recombination changes found recently for the black GaAs surface.

2. Experiments

Epitaxial GaAs-based semiconductors were grown by molecular beam epitaxy. Fig. 1 presents a basic structure for the materials treated and measured. Detailed layer structures characterized are presented below. All samples measured in this work included initially n-type GaAs contact layer, which was etched away wet chemically ($NH_4OH + H_2O_2$ solution) before the treatments and measurements of some samples. The material was cut into square pieces with 6 mm length, and then samples were inserted into a beaker of hot hydrogen peroxide followed by hot deionized water (resistivity of 21 MΩcm) on top of a hot plate. The water temperature was set and kept constant at 80 °C. The immersion time varied between 5 and 60 min in this work.

XPS was measured with Thermo Scientific Nexsa system equipped with monochromatized Al Kα radiation source and dual mode charge compensation. The wide energy-range survey spectra and high-resolution core-level spectra were collected with pass energies of 200 eV and 50 eV, respectively. The X-ray spot size was 200 μm. XPS peak fitting was performed with CasaXPS [23]. The components in the core level spectra were fitted with a Voigt-type LA(m) lineshape, where m is a parameter which corresponds to the amount of Gaussian character. The line shape was kept the same for the same component across different samples. The parameter m was chosen for each component by performing a test fitting with a minimum number of components and optimizing the residual standard deviation of the fit. Photoluminescence (PL) spectra were measured with Accent RPM2000 PL instrument at room temperature.

3. Results and discussion

Fig. 2 shows that the PL peak intensity from the GaInP/GaAs sample increases from a value of 2.3 V to 2.8 V when the sample is immersed first in hot H_2O_2 (5 min) and then in hot H_2O (30 min). Mere hot water H_2O (30 min) immersion also increases the peak value to 2.6 V (Supplementary Fig. S1). The maximum error in the PL intensity, arising from a variation in laser excitation power, is estimated to be ±0.1 V.

These results are surprising because firstly, the PL intensity measured from a native GaInP/GaAs is among the strongest GaAs emissions, as described in the introduction section above. Secondly, the H_2O_2 and H_2O immersions are expected to increase the surface oxidation that is commonly considered to degrade III-V surface properties [21]. The found PL intensity increase is not due to a decrease in the surface reflectivity because the treatment does not change significantly the reflectance curve (Fig. 2). Thus, we have used XPS to probe physicochemical properties of the GaInP surface, which might be linked to the PL results. It is worth noting that the link between XPS and PL results is indirect here because it is difficult to resolve and identify an XPS signal contribution(s), which arises directly from the local atomic-level defects that can still change PL intensity significantly. Thus, the presented XPS results rather describe changes in average bonding environment around local defects in long range.

Fig. 3 presents In 3d spectra from three different GaInP/GaAs samples: native-oxide covered reference, after hot H_2O (30 min) immersion, and after hot H_2O_2 (5 min) + hot H_2O (30 min) immersions. These three samples include a similar additional component at high binding-energy side with the shift of 1.1 eV, in relation to the bulk component. The intensity of this 1.1-eV shifted component increases with the immersions and is highest for the sample after hot H_2O_2 (5 min) + hot H_2O (30 min). Table 1 summarizes the relative component intensities. The extra component in In 3d can be associated with a surface oxide, most likely $InPO_4$ type and/or OH-containing ones [24–26]. According to the previous study [24] the oxide is not In_2O_3 type because In_2O_3 causes a very small shift or even decreases the binding energy as compared to InP. Because the In-oxide related component however increases more than the P-oxide related component (below), we conclude that the In-oxide component includes also another bonding environment without P atoms, in addition to the possible $InPO_4$ type structure. It is also possible that In-oxides are enriched at the topmost surface. The In 3d spectra also reveal that the 20-nm thick GaInP layer is not fully oxidized because the bulk-like GaInP crystal component remains.

The P 2p spectra from the same samples in Fig. 3 do not show a similar systematic increase in the intensity of the P-oxide related component, but a binding energy shift of the P-oxide component increases systematically from 4.3 eV to 4.55 eV with the treatments, being largest after hot H_2O_2 (5 min) + hot H_2O (30 min). Intuitively, this indicates that the average number of oxygen bonds per P atom increases, but the previous calculations have shown that the formation of different $InPO_4$ building blocks with PO_4 tetrahedra units can also cause the binding energy change of 4.3 eV–4.55 eV [24]. Furthermore, the calculations indicate that thickness of $InPO_4$ layer affects the P 2p core-level energy [24]. Another factor behind the binding-energy shift is still possible incorporation of hydrogen into the treated surfaces. However, this is consistent with the previous findings [27–29] have shown

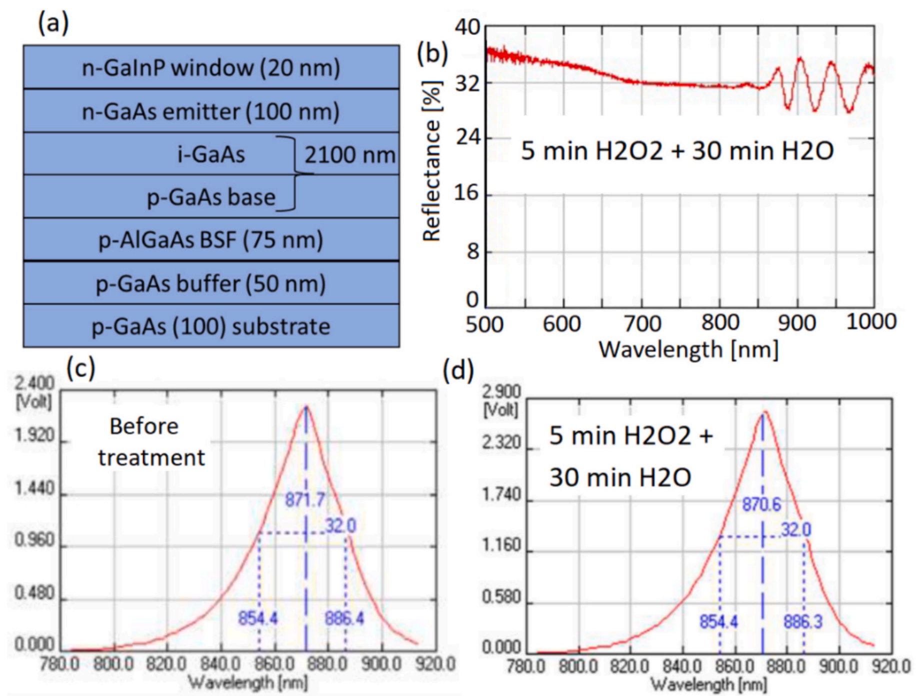

Fig. 2. (a) Epitaxial film structure studied. (b) GaInP surface reflectivity did not change significantly after the treatment of 5 min hydrogen peroxide immersion + 30 min water immersion both at 80 °C. (c) PL spectrum measured before the treatment. (d) PL spectrum measured after the treatment.

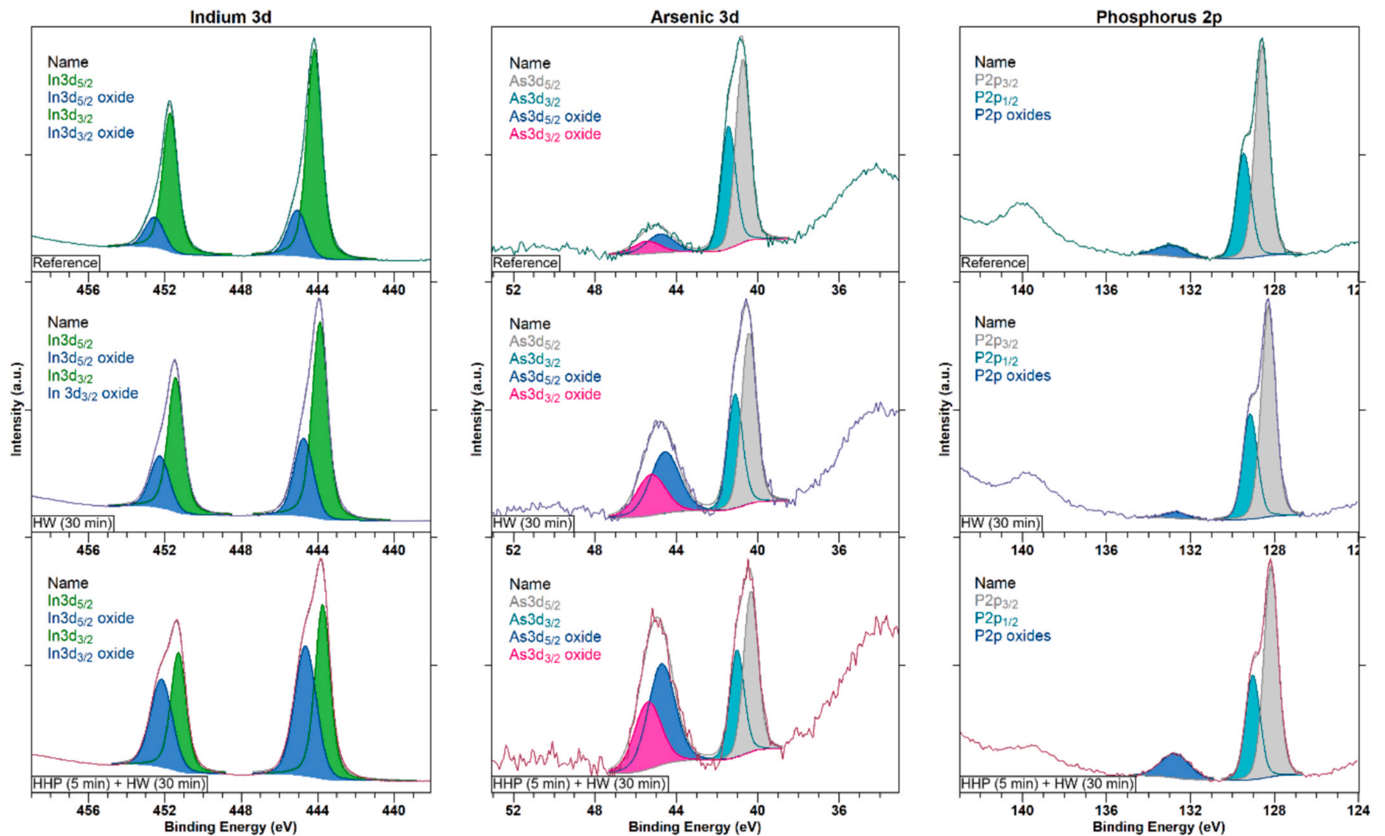

Fig. 3. XPS spectra of In 3d (left column), As 3d (middle column), and P 2p (right column) from three different samples: (i) reference GaInP with a native oxide (top row); (ii) after 30 min immersion in hot water (middle row); (iii) after 5 min immersion in hot hydrogen peroxide + 30 min hot water immersion (bottom row).

Table 1
Fitting results for In 3d, As 3d and P 2p spectra measured from three samples: reference InGaP sample with native oxide, after hot water (HW) immersion, and after hot hydrogen peroxide (HHP) + hot water immersions. Intensity means a relative intensity of component to the total intensity. Details of the fitting are described in the experiments section.

		In 3d substrate	In 3d oxides	As 3d substrate	As 3d oxides	P 2p substrate	P 2p oxides
	Lineshape	LA(78)	LA(78)	LA(50)	LA(50)	LA(45)	LA(42)
Reference sample	Intensity (%)	79.6	20.4	83.4	16.6	93.4	6.6
HW (30 min)	Intensity (%)	68.6	31.4	58.6	41.4	97.1	2.9
HHP (5 min) + HW (30 min)	Intensity (%)	50.5	49.5	45.8	54.2	86.7	13.4

that a high oxidation state for P atoms improves the passivation, which supports our observation for the increased PL intensity (Fig. 2). The GaInP film also includes some As impurities which have been found in GaInP heterostructures earlier also [30]. The As 3d spectra indicate that the As-oxide related intensity increases with the immersion treatments. A shift of the As-oxide component also increases from 4.0 eV to 4.4 eV due to the treatments similarly to the P-oxide shift.

C 1s spectra do not show any significant change due to the treatments (Supplementary Fig. S2). O1s spectra (Supplementary Fig. S3) support that amount of oxygen increases at the surface due to the treatments, as expected, but the O1s line shapes do not reveal clear change(s) in a detailed bonding structure. However, we can see a systematic energy shift of about 0.3 eV toward higher binding energy for all peaks of the reference sample with a native oxide, as compared to the two other samples. This suggests a change in the band bending because the binding energies are measured in relation to the Fermi level. The possible change is such that the conduction (and valence) bands bend upward at the surface after both the treatments, as compared to the flat-band situation in Fig. 1, which can affect the PL intensity. The increased upward band bending repels the electron carriers from the surface while the hole carriers experience an attractive field toward the outermost surface. This leads to the situation where less electron carriers reach the defect-rich outermost surface, suppressing non-radiative recombination at the surface. Simultaneously, the increased oxidation of the surfaces, deduced from the XPS results, is expected to increase the surface band gap, reducing further the carriers' density at the surfaces.

Finally, we have tested (Fig. 4) whether it is possible to modify the highly n-type GaAs film (150 nm) that is often deposited on the top of GaInP to make an Ohmic metal contact with the strongly doped n-GaAs. The disadvantage of the n-GaAs cap is that it severely degrades the PL intensity, as discussed in the introduction section. Therefore, this n-GaAs film is typically removed in selective way from the areas between the metal contacts in optoelectronic devices. Also, in this work the as-grown sample with the highly n-type GaAs cap shows a clear decrease in the PL intensity about by factor of 16 (Supplementary S4), as compared to the intensities measured from the GaInP/GaAs samples in Fig. 2 without the top n-GaAs. Therefore, it is interesting that the PL intensity increases by factor of two approximately (Fig. 4), as compared to the values in Fig. 2, when the topmost n-GaAs film is treated first in hot H_2O_2 (30 min) and then in hot H_2O (30 min). This PL intensity change can be explained by a decrease in the surface reflectivity by the same factor of two (Fig. 4). Therefore, we expect here that the decreased reflectivity is the main factor behind the increased PL intensity in the n-GaAs containing sample. Then this correlation is connected to the previous findings from black GaAs surfaces [22]. Namely, the found correlation between the reflectivity change and the PL intensity change indicates that the recently found higher PL intensity increase, as compared to the reflectivity change, arises from an improved passivation level after the hot

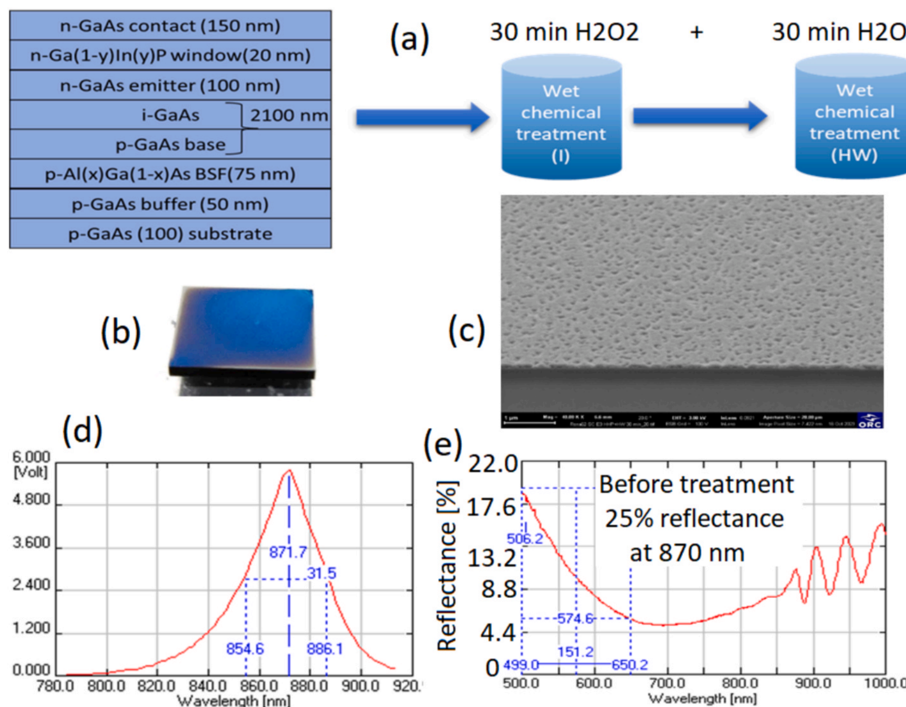

Fig. 4. (a) Studied film structure includes also n-type GaAs cap layer. The sample treatment was 30 min hydrogen peroxide immersion + 30 min water immersion both at 80 °C. (b) Photo after the sample treatment shows a reflectivity change. (c) Scanning electron microscopy image after the treatment. (d) PL spectrum after the treatment. Before the treatment, the PL intensity was weak with a peak value of 0.15 V (Supplementary Fig. S4) because the n-GaAs layer degraded the intensity. (e) Reflectivity curve shows that the n-GaAs surface reflectance decreases by factor of 2.5 approximately due to the treatment.

water treatment of GaAs surfaces [22]. Thus, the treatment based on the immersions in hot H_2O_2 and in hot H_2O has potential in the optical and electrical passivation. It is worth noting that the presented results were obtained after the first optimization series and that a parameter space is large. Future studies are also needed to clarify other electrical properties of the surface oxides as well as their durability. Here we note that our samples were, at least, for one day in atmospheric conditions after the treatments before the PL measurements. Some samples were re-measured after two months without significant changes in the PL intensity.

4. Conclusions

We have studied effects of the recently reported surface treatment, which includes immersions in hot H_2O_2 and hot H_2O, on properties of epitaxial GaInP/GaAs junctions. In contrast to a clear reflectivity decrease at GaAs surfaces [22], this treatment does not change the reflectivity significantly at GaInP surfaces, according to our measurements. However, the presented results demonstrate that the proper combination of the hot (80 °C) H_2O_2 and H_2O immersion increases about 20 % the PL intensity from GaInP/GaAs, which is a very high-quality reference sample due to well-crystalline GaAs interface and specific properties of GaInP native oxides. XPS measurements show that the found PL-intensity increase can be linked indirectly to (i) increased amount of In oxides or/and (ii) increased oxidation state of P atoms. Finally, we have shown that the highly n-type GaAs contact layer (150 nm), which degrades the PL intensity initially, can be modified by the hot H_2O_2 and H_2O immersions into a form which increases the PL intensity of epitaxial GaInP/GaAs about 200 %. This increase is suggested to arise from the decreased surface reflectivity. The presented results support that the surface treatment, based on the hot H_2O_2 and H_2O immersions has the potential in the surface passivation for III-V device components.

CRediT authorship contribution statement

Zahra Jahanshah Rad: Writing – original draft, Validation, Methodology, Investigation, Conceptualization. **Mikko Miettinen:** Writing – review & editing, Methodology, Investigation. **Johanna Laaksonen:** Writing – review & editing, Validation, Methodology, Investigation. **Perttu Piispanen:** Writing – review & editing, Software, Investigation. **Marko Punkkinen:** Writing – review & editing, Supervision. **Kalevi Kokko:** Writing – review & editing, Validation, Supervision. **Pekka Laukkanen:** Writing – original draft, Supervision. **Antti Tukiainen:** Writing – review & editing, Supervision, Methodology. **Heidi Tuorila:** Writing – review & editing, Methodology, Investigation. **Helmer Piirilä:** Writing – review & editing, Methodology, Investigation. **Jukka Viheriälä:** Writing – review & editing, Supervision, Resources. **Mircea Guina:** Writing – review & editing, Supervision, Funding acquisition.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.apsusc.2025.165009.

Data availability

Data will be made available on request.

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