Surface and interface study of SiO_{2-x} coated InP/InGaAs/InGaAsP semiconductor laser microstructures processed in the soft KrF laser irradiation regime

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ABSTRACT

The ability of a UV laser to modify surface properties of quantum well (QW) microstructures that would lead to formation of a “defective layer” is of particular interest to the process of QW intermixing (QWI). We discuss the results of surface and interface study of InGaAs/InGaAsP QW microstructures capped with InP and a 243-nm thick layer of SiO_{2-x} that were irradiated with a KrF excimer laser delivering up to 25 pulses at 124 mJ/cm^2. The optical quality of SiO_2 films remains relatively unaffected by the irradiation with the KrF laser operating in the investigated window of parameters. The x-ray photoelectron spectroscopy experiments point out the negligible role of SiO_{2-x} in out-diffusion of matrix atoms that would enhance the QWI process. However, the KrF laser was found to significantly modify the interface between UV transparent SiO_{2-x} and the InP layer. Our results suggest that the resulting layer of the altered material promotes out-diffusion of atoms and intermixing in the QW region.

Key words: KrF laser, SiO_2 layer, InP/InGaAs/InGaAsP microstructure, quantum well intermixing, Photonic devices surface and interface analysis, X-ray photoelectron spectroscopy, Atomic Force microscope, Scanning electron microscope

1. INTRODUCTION

Surface study of laser irradiated dielectric films on hard substrates, such as sapphire, fused silica or SiC, is important for the understanding and development of damage-resistant coatings, as illustrated by numerous investigations devoted to this problem.[1-2] However, relatively few results have been published that concern semiconductor substrates (wafers) coated with Si_3N_4 or SiO_2 films and irradiated with ultraviolet lasers at the material near-ablation threshold. For instance, KrF laser irradiation of SiO_2/Si sample at 724 mJ/cm^2 has been reported to melt the SiO_2 and create ripple structures with significant cracks at the interface between SiO_2 and Si.[3] We have been investigating the ability of UV lasers to modify surface properties of III-V quantum well (QW) microstructures and generate defects capable of promoting the process of QW intermixing (QWI).[4-6] Excimer lasers are known to modify the surface chemical composition of InP- and GaAs-based QW microstructures irradiated in air and lead to a significantly enhanced interdiffusion between the well and barrier materials during high temperature rapid thermal annealing (RTA).[6-8] X-ray photoelectron spectroscopy (XPS) measurements have demonstrated that the amount of oxides, e.g. indium oxides and gallium oxides, have significantly increased in the excimer laser irradiated sites.[6, 8] From secondary ion mass spectroscopy (SIMS) measurements, it has

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been reported that on InP based QW structures, In and Ga atoms are easier to outdiffuse to the SiO_{2-x} layer and create V interstitials that enhance interdiffusion between barrier and well materials during RTA.[9] XPS depth profiling results also demonstrated that the total concentration of In and Ga atoms outdiffusing to the SiO_2 layer could be at around 0.5 at. % while a negligible presence of P atoms has been found in SiO_2.[10] This results in the excess of P and As interstitials in the QW region and leads to an enhanced intermixing process.

We have recently found that the KrF laser enhances QWI in InP/InGaAs/InGaAsP microstructures coated with a 243 nm thick SiO_{2-x} layer, without causing obvious damage to that layer.[4] A moderate 67 nm net blueshift was observed for sites irradiated by the KrF laser delivering 25 pulses at 124mJ/cm^2. In this paper, we investigate the role of a laser-induced layer of the altered material in the QWI process and the participation of the SiO_{2-x} layer in that process.

2. EXPERIMENT

The investigated QW microstructure consists of five 5.5-nm-thick $In_{0.47}Ga_{0.53}As$ QWs separated by four 12-nm-thick $In_{0.74}Ga_{0.26}As_{0.57}P_{0.43}$ barriers. The microstructure is capped with a 400 nm thick InP layer, which compares to a 30-nm-thick InP layer used in our previous structure.[4] The investigated microstructure is a full semiconductor laser structure grown on n-doped InP substrate and is designed to emit at 1.540 µm at room temperature. A 243 nm thick SiO_{2-x} layer was deposited on top of the microstructure by a plasma enhanced chemical vapor deposition (PECVD) method. Samples, typically 8 mm x 8 mm, were first cleaved from a wafer and cleaned with standard solvents. The laser delivered 23 ns long pulses of fluence at 124 mJ/cm^2. A double micro-lens fly-eye-array homogenized laser beam was used to project a circular mask on the sample surface. A computer controlled X-Y-Z-Theta positioning of the sample allowed for the processing of the same sample at numerous sites. The irradiation was carried out in an ambient air environment. Following the irradiation, the sample was annealed in a commercial RTA processor (Jetfirst) in an atmosphere of mixed hydrogen and nitrogen (0.1: 0.9) for 2 minutes at 700°C. Thus, the annealing conditions for different sites on the same sample were nominally the same.

XPS was used to analyze the samples using an Al Kα as X-ray irradiation source and pass energy of 20 eV. High resolution XPS analysis was used to investigate the chemical composition of the SiO_{2-x} layer and the SiO_{2-x}/InP interface region before and after KrF laser irradiation. Curve fittings of experiment peaks were achieved with Lorentzian and Gaussian profiles and the background was removed using Shirley method. For Si 2p peak, the same full width at half maximum (FWHM) was used to curve fit different Si oxidized states. To correct charge effect, all the binding energies were referenced to adventitious C 1s at binding energy of 285.0 eV. The outdiffusion of different atoms from top InP layer to the SiO_{2-x} layer was analyzed by implementing an XPS depth profiling procedure that employed Ar ion sputtering. High-resolution analysis of In 3d, P 2p, Si 2p, O 1s and C 1s was also carried out for reference and sputtered surfaces. A scanning electron microscope (SEM) was used to examine the cross section of SiO_{2-x}/InP interface before and after KrF laser irradiation. The ellipsometry was used to estimate the thickness of the interface region. The SiO_{2-x} and InP surface morphology of samples after excimer laser irradiation and RTA was imaged with an AFM (Digital Instrument, Nanoscope III) operating in a tapping mode. To characterize sample surface morphology, the AFM images were collected over the 10 µm x 10 µm region with 512 points per line at scan rate of 1.97 Hz.

3. RESULTS AND DISCUSSIONS

The photoluminescence (PL) map of the InGaAs/InGaAsP/InP microstructure after KrF laser irradiation at 124 mJ/cm^2 and RTA at 700 °C for 2 minutes, and PL shift dependence on pulse number are shown in Figure 1 (a) and (b), respectively. The map shows an array of laser-irradiated sites that, depending on the number of irradiating pulses, emit at different wavelengths. The upper and bottom numbers in every site indicate the pulse number and achieved blueshift, respectively. Figure 1 (b) demonstrates that the blueshift increases with the pulse number and the maximum blueshift amplitude achieved in this case is about 80 nm. This result is obtained without removal of the PECVD deposited SiO_{2-x} layer, as illustrated by a set of insets in Figure 1 (b) showing microscopic images of the sites irradiated with 2, 10 and 25

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pulses. Note that the maximum blueshift in the SiO_{2-x} coated microstructure is almost the same as that in the uncoated sample that was irradiated in air and RTA.

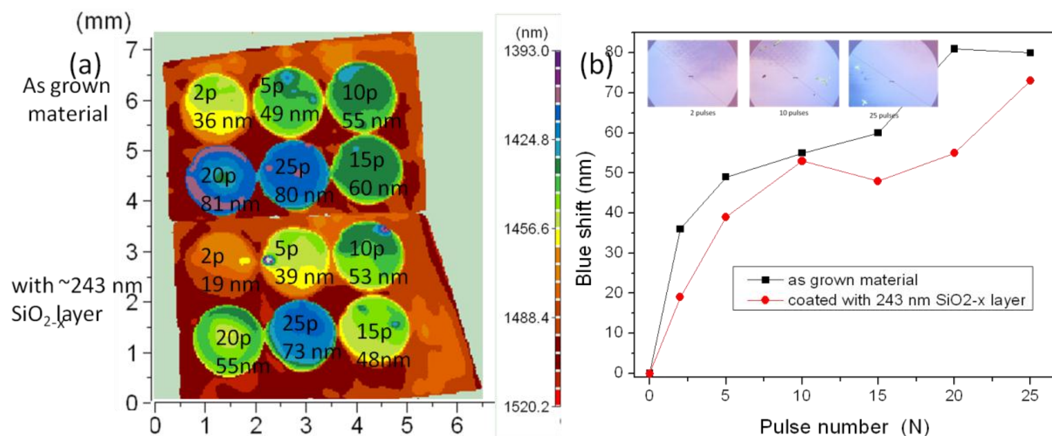

Figure 1. (a) PL map of InGaAs/InGaAsP/InP microstructure after KrF laser irradiation at 124mJ/cm^2 and RTA at 700°C for 2 minutes, and (b) PL shift dependence on pulse number. The insets show microscopic images of the irradiated sites.

Table 1 shows the atomic percentage of Si, O and C in as-grown SiO_{2-x} and in 25-pulse irradiated sites of the SiO_{2-x} layer. The O/Si ratio in both cases is less than 2, which suggests that the film is a mixture of different Si-oxides. It can also be seen that the O/Si ratio (1.52) increases, although minimally, in the laser irradiated site (1.54), which is an expected result given that the irradiation was carried out in an atmospheric environment. A decrease of C on the surface of laser irradiated SiO_{2-x} suggests that some laser cleaning of the adventitious C took place, similarly to the reported KrF laser dry cleaning of C absorbates.[11] The detailed Si 2p, O 1s and C 1s XPS spectra for this sample are shown in Figure 2. Following the typical order binding energy of Si oxides, the Si 2p peak can be deconvoluted into SiO_2 (BE=104.0±0.1 eV, FWHM=1.28 eV) and SiO_x (BE=103.5±0.1eV, FWHM=1.28 eV)[12-13]. On the site irradiated with 25 pulses, an additional peak is found at a lower binding energy (BE=101.9 eV) in the Si 2p XPS spectrum shown in Figure 2 (d). This peak is accompanied by a peak at a lower binding energy (BE=282.8 eV) in the C 1s spectrum in Figure 2 (f). The 282.8 eV peak in the C 1s spectrum corresponds to carbon in carbides or carbon-oxygen-metal complexes.[14] Therefore, according to the peak position of both C 1s and Si 2p spectra, this peak was assigned to Si-C created by the KrF laser.[15] All the Si oxides are referenced to the same binding energy (533.0 eV) in the O 1s spectra shown in Figure 2 (b) and (e) as the position of this peak is less sensitive to the presence of all oxides in contrast to the Si 2p peak.[16] There is also a weak O 1s peak at the lower binding energy (BE=531.116 eV) that could be ascribed to O 1s in non-stoichiometric Si oxides. On the irradiated site, there is another Si-O peak observed at 533.7 eV, which is similar to the reported results.[17,18] Besides the Si-O peak, there is an H_2O related peak (BE=534.2±0.2 eV), whose amount has been reduced after laser irradiation due to KrF laser heating as shown in Figure 2 (b) and (e). Also, in C 1s spectra in Figure 1 (c) and (f), the quantities of –COH (BE=286.6±0.2 eV), -CO (BE=287.8±0.2 eV), -COO-(BE=289.2±0.2 eV) absorbates have decreased following the laser irradiation, i.e., the carbon contained absorbates have been partly eliminated by the KrF laser surface cleaning.[11]

Atomic %	Si	O	C
Non irradiated	35.92	54.50	9.58
Irradiated with 25 pulses	35.74	55.14	9.12

Table 1. Atomic percentage of elements in as-grown SiO_{1-x} and in sites irradiated with 25 pulses of the KrF laser.

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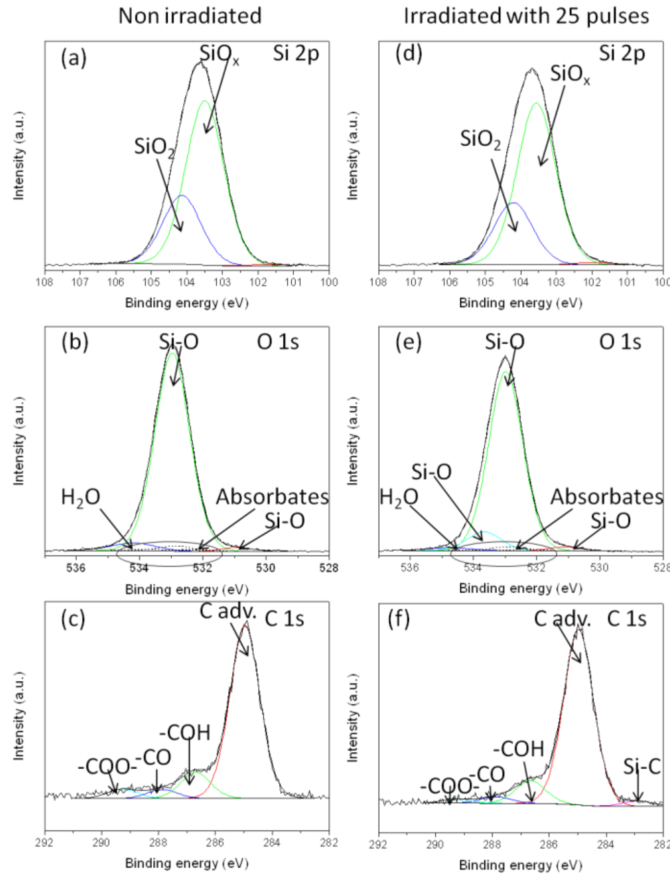

Figure 2. Si 2p (a), (d), O 1s (b), (e) and C 1s (c), (f) XPS spectra of no irradiated site and site irradiated with 25 pulses by KrF laser at $124 mJ/cm^2$.

Figure 3 shows an XPS survey of the SiO_{2-x} surface (a), the SiO_{2-x}/InP interface 1 (b), the SiO_{2-x}/InP interface 2 (c) and the InP cap material (d) after KrF laser irradiation and RTA. The SiO_{2-x}/InP interface 2 is the surface after interface 1 sputtered for another 60 seconds and the average sputtering speed in InP is estimated to be 0.5 nm/second. In contrast to earlier reported results,[10, 19] no In 3d (445 eV, red line) and P 2s (187 eV, green line) are found in the SiO_{2-x} layer. Furthermore, a weak In 3d (0.35 %) but no P 2s peaks are found at the SiO_{2-x}/InP interface 1. After ion sputtering for another 60 seconds, the In 3d and P 2s peaks have been observed in the interface 2 at 2.67% and 1.88%, respectively. No Si 2p is found in the InP cap material, as shown in Figure 3 (d). In view of the comparable blueshifting amplitudes observed for both as-grown and SiO_{2-x} coated samples, the negligible role of Si impurities (if any) is suggested by this result. Figure 4 shows high resolution XPS scans in the In 3d and P 2s spectral regions recorded from the SiO_{2-x} layer (a, e), SiO_{2-x}/InP interface 1(b, f), SiO_{2-x}/InP interface 2 and InP layer (c, g). No In 3d (445 eV) and P 2s (187 eV) peaks are found in the SiO_{2-x} layer. No P 2s peak is found at the SiO_2/InP interface 1. However, the presence of In 3d 5/2 peak is clearly observed in the interface 1 region. It has been reported that the charging effect caused by X-ray irradiation changes as sputtering time changes in depth profiling of SiO_{2-x} film coated semiconductor system.[20, 21] Here we find that both C1s and Si 2p core level have shifted 0.7eV from surface to interface 1. The peak at 445.1eV in the In spectrum in Figure 4 (b) is related to the C peak at 285 eV that after correcting the charging effect can be assigned to an In-Oxide peak.[22, 23] This indium oxide layer may contain plasma oxides grown during exposure to an oxygen plasma in the PECVD reaction chamber that has been verified by in-depth XPS analysis on PECVD deposited SiO_{2-x}/InP interface and it is always close to SiO_{2-x} layer.[24] At the SiO_{2-x}/InP interface 2, there are two additional peaks in the P 2s spectra. The peak at lower binding energy (186.3 eV) is fixed to InP or elemental P and the peak at higher binding energy (191.3 eV)

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is fixed to be P oxides [25]. This P-oxide layer is created by the reaction between the outdiffused P atom from InP land the SiO_{2-x} layer.[26] In our case, it is possible that the KrF laser irradiation modifies the interfacial oxides as suggested by our SEM and elliposometry measurements that are discussed in the following section. There are obvious In 3d (444.5 eV) and P 2s (186.3eV) peaks in the InP layer spectra in Figure 4 (d) and (h).[25] However, the peaks of In-oxide at 445.1 eV in the InP spectrum and P-oxide at 191.3eV in the P 2s spectrum have disappeared.

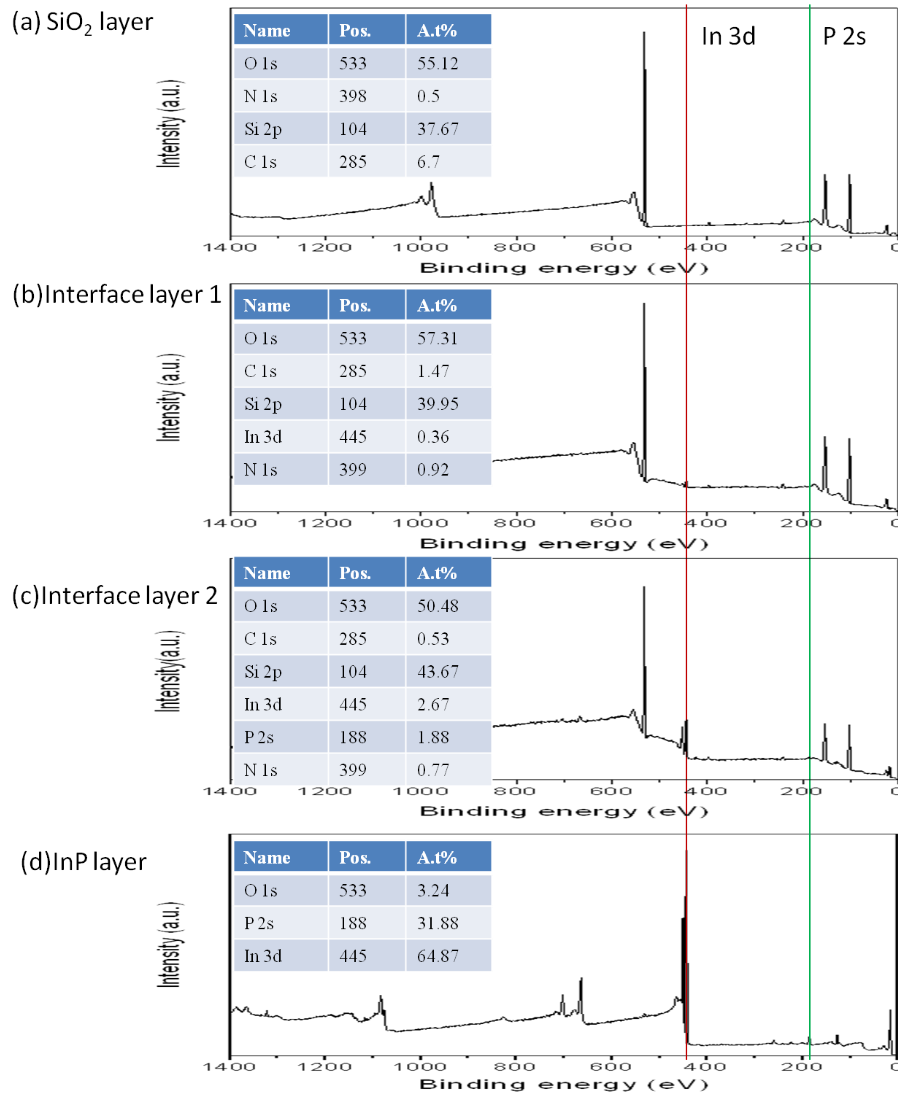

Figure 3. XPS survey of (a) SiO_2 layer, (b) SiO_{2-x}/InP interface 1, (c) SiO_{2-x}/InP interface 2 and (d) InP layer after KrF laser irradiation and RTA.

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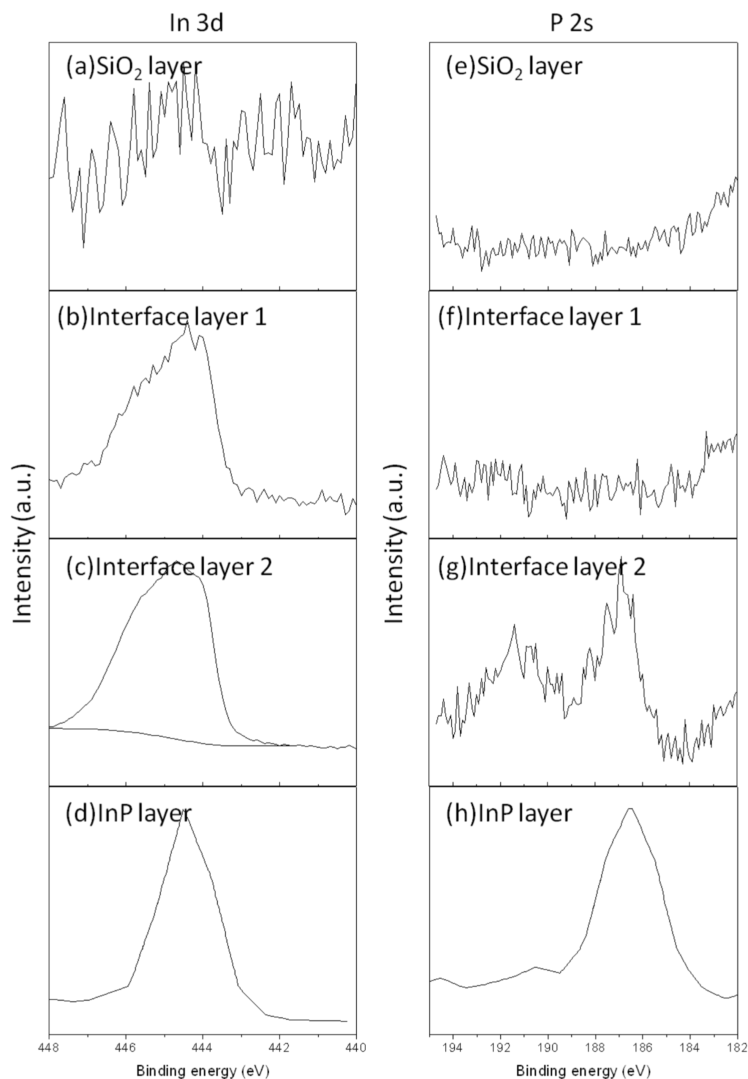

Figure 4. In 3d 5/2 (a, b, c, d) and P 2s (e, f, g, h) XPS spectra of the SiO_2 layer, SiO_{2-x}/InP interface 1, SiO_{2-x}/InP interface 2 and the InP layer following KrF laser irradiation and RTA.

Figure 5 shows XPS depth profiles of O, Si, C, In, N, P, Ga and As in the SiO_{2-x}/InP top portion of the InGaAs/InGaAsP microstructure following irradiation with the KrF laser and the RTA processing step. It can be seen that the concentration of Si and O remains almost constant for the sputtering time $0 \leq t_{sp} < 2500$ sec. This indicates a relatively uniform SiO_{2-x} layer. However, the O/Si ratio inside the layer is about 1.43, which is less than this ratio measured at the surface of the SiO_{2-x} layer (see Table 1). The possible reason for this decrease could be related to the preferential sputtering of oxygen.[27] The results indicate no presence of In and/or P atoms in the SiO_{2-x} layer. This is in contrast to the impurity free vacancy induced disordering effect observed in InP-based MQW microstructures that were RTA at 800-850°C for 2-3 minutes.[10,9] The related experiments have reported the presence of In and Ga atoms outdiffused to the cap SiO_2 layer at ≥ 0.5 at.%. Thus, the argument was made that the left behind group III vacancies or group V interstitials were responsible for an enhanced intermixing process. Given that the sensitivity of our XPS measurements for detection of In and P is not worse than 0.1 at.% (we have easily detected the presence of N in the SiO_{2-x} layer at about 0.5 at.%, as shown by the insets in Fig. 5 (a) and (b)), our results suggest that the contribution of outdiffusing In and/or Ga atoms to the QWI process must be at the impurity concentration level, i.e., < 0.1 at.%. A drastically reduced concentration of In and Ga

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atoms in SiO_{2-x} layer is consistent with the relative low RTA temperature (700°C) applied in our experiments. It is also possible that the efficiency of the outdiffusion process could depend on the quality of deposited SiO_{2-x} material. The concentration of In and P atoms increases at the SiO_{2-x}/InP interface. However, our results show a significantly greater concentration of In than would be expected for a stoichiometric InP.[10] It has been reported that in XPS depth profiling experiments, the ion sputtering induces chemical modification and leads to surface segregation of atoms.[28] For InP-based materials, the In/P ratio has always been observed greater than 1 (~1.4) due to the preferential removal of P during sputtering.[29] Our XPS depth profile shows that, at the maximum, the In/P ratio in the laser irradiated sample is significantly greater (2.3) than that in the non-irradiated sample (1.8). Next, this ratio decreases due to the presence Ga and As atoms in the microstructure. The increase of Ga and As in the InP layer is related to the outdiffusion from InGaAs and InGaAsP during RTA. This process could be, partially at least, related to the temperature induced defect formation in this material system where InP ($4.6X10^{-6}$/°C) has a lower thermal expansion coefficient than InGaAs (5.66 $X10^{-6}$/°C) and InGaAsP (5.0 $X10^{-6}$/°C).[30] [31] The results in Figure 5 (a) and (b) show that Ga and As outdiffuse more easily to the InP layer, and the concentration of these atoms is greater in the laser irradiated material than in the non-irradiated material. This is possibly the reason for a higher PL shift on laser irradiated sites. A small amount of N_2 observed in the SiO_2 layer is likely due to the diffusion of this element during the RTA process carried out in the N_2:H_2 environment. Also, the amount of C contained absorbates decreased from 6.7% to 1.66%, and eventually disappeared, as the sample was investigated further away from the surface.

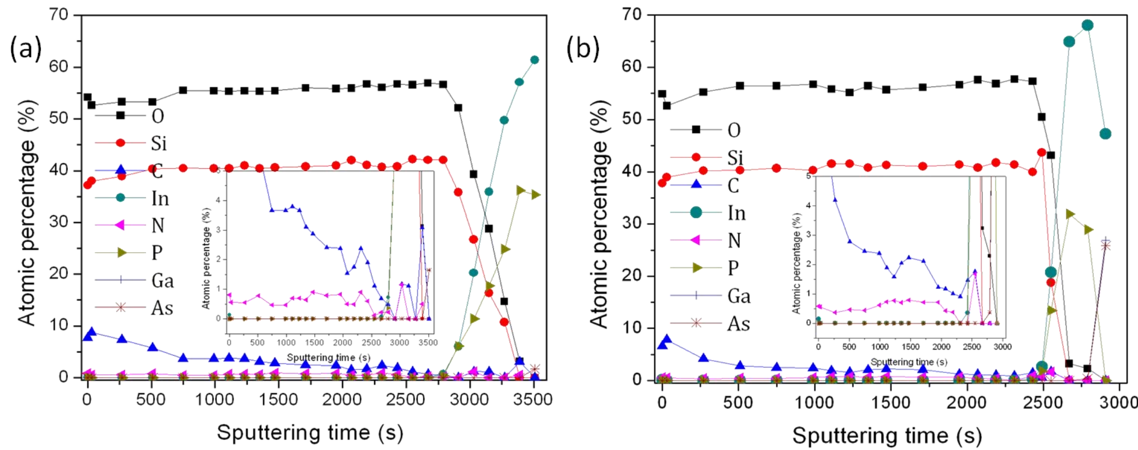

Figure 5. XPS depth profiles of O, Si, C, In, N, P, Ga and As in non-irradiated (a) and laser irradiated (b) SiO_2/InP/InGaAs/InGaAsP microstructures following their RTA.

SEM cross-section images of InP/InGaAs/InGaAsP microstructures in the SiO_{2-x}/InP region are shown in Figure 6 for the as-grown (a) and KrF laser irradiated (b) samples. A thin interfacial layer, approximately 8.65 nm thick, can be seen at the SiO_{2-x}/InP interface of the as-grown material. As reported in literture,[24] the likely origin of that layer is In_2O_{3-x} that is formed during the PECVD deposition of SiO_{2-x}. The SEM observation has been corroborated by the ellisopmetry measurements indicating that the total thickness of the SiO_{2-x} layer is ~ 7.7 nm. A thicker interface layer of ~53.05 nm has been observed in the sample irradiated with the KrF laser, as shown in Fig. 6 (b). This compares to the ellipsometry determined thickness of 45.3 nm. The ellipsometry measurements have also indicated that the laser irradiation leads to a 6-fold growth of the InP-oxide layer, while as large as a 14-fold growth of the In_2O_{3-x} layer has been suggested. The presence of In-oxides and InP-oxides is consistent with our XPS scans reported in Figure 3 and 4. We associate the QWI effect observed in these samples with the presence of the In_2O_{3-x} layer that has a greater thermal expansion coefficient ($6.15X10^{-6}$/°C)[32] than InP. A stress between In_2O_{3-x} and InP that is induced during the RTA contributes to an enahnced group III atom outdiffusion[31] and, consequently, to a relatively strong QWI process.

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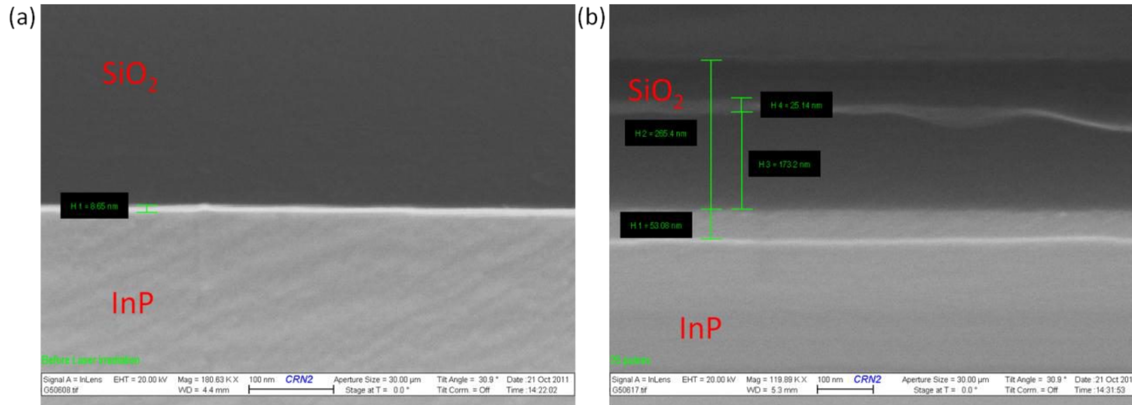

Figure 6. SEM image of InP quantum well sample coated with 243 nm SiO_2 layer before KrF laser irradiation and after irradiated with 25 pulses at $124 mJ/cm^2$

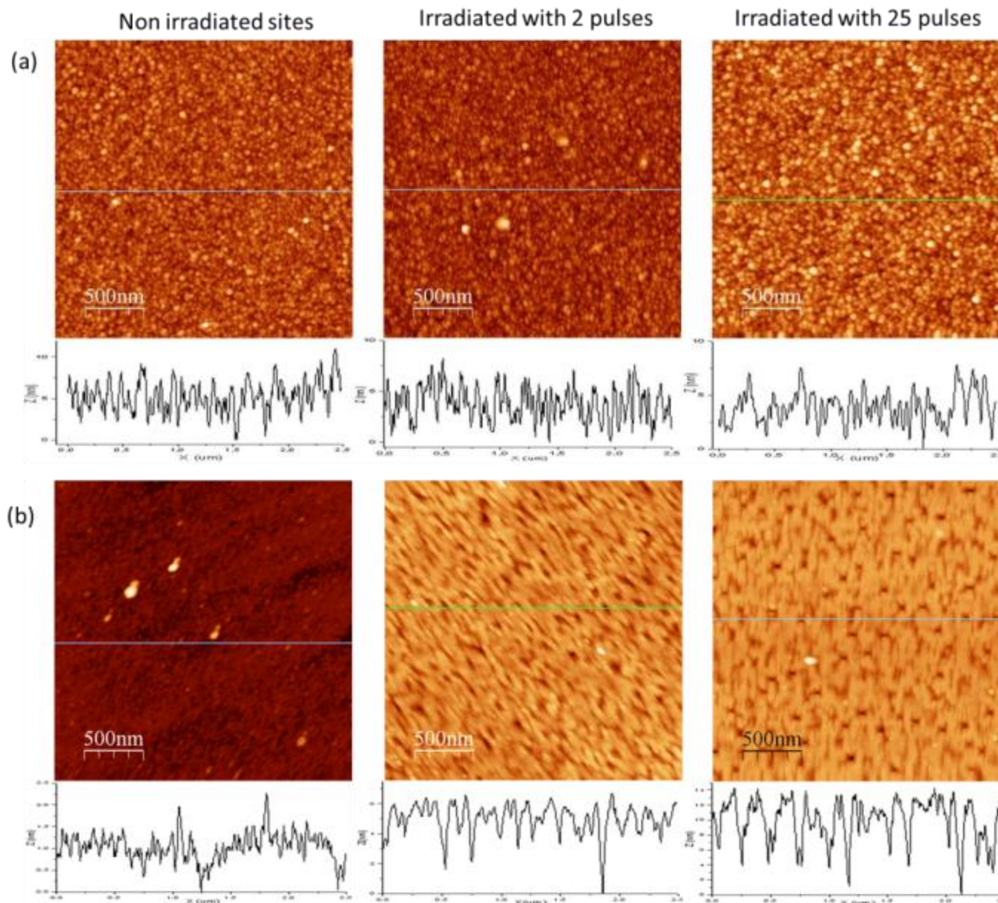

Figure 7. AFM images of SiO_2 (a) and InP (b) before and after KrF laser irradiation.

Figure 7 shows AFM images of SiO_2 (a) and InP (b) before and after KrF irradiation with 2 and 25 pulses. A similar grain structure is observed for both non-irradiated and irradiated SiO_{2-x} material. The average grain height was found decreasing only moderately from 6.42 nm to 5.22 nm after KrF laser irradiation.[4] In contrast, a significant change of the InP surface morphology has been observed following the irradiation with the KrF laser. Sub-wavelength periodic

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structures can be seen on the InP surface irradiated with 2 pulses (Fig. 7 (b). Formation of a similar InP surface morphology has been reported in literature.[33] Oxides have been known to influence significantly the surface morphology of high-temperature treated InP,[34] thus it is reasonable to associate these morphological changes with the laser-induced formation of oxides.

4. CONCLUSION

We have investigated the surface and interface properties of SiO_{2-x} coated InP/InGaAs/InGaAsP QW microstructures irradiated with a KrF laser and RTA at 700°C for 2 minutes. The XPS measurements show that the KrF laser does not modify significantly the surface chemical composition of the SiO_{2-x} layer. The laser-induced removal of carbon adsorbates has been observed along with the formation of SiC. Both in-depth XPS measurements and high resolution XPS suggest that no outdiffusion of In, Ga or P takes place in the investigated samples, although both SEM and AFM results show that the thickness of the interfacial SiO_2/InP layer increases following the KrF laser irradiation and RTA. We associate the presence of In-oxides and InP-oxides with the QWI effect observed in this experiment. These findings provide an important insight in the problem of the KrF laser induced defect formation and, in particular, formation of a layer of modified material that enhances significantly the QWI effect.

5. ACKNOWLEDGEMENT

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