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Chemical depth profiling of III–V trenches containing InGaAs quantum wells with AlAs barriers selectively grown inside SiO_2 cavities was studied using magnetic secondary ion mass spectrometry (SIMS). The authors show that the depth resolution of SIMS profiles of III-As layers degrades under extremely low energy oxygen beam sputtering (<500 eV) due to ripple formation and an increase in surface roughness. Improved SIMS depth resolution was observed by increasing the incident angle (∼50°–65°). Finally, the authors report the effect of sample rotation and orientation of the ion beam with respect to the trenches on depth profiling of III-As layers using time-of-flight SIMS. © *2016 American Vacuum Society*. [http://dx.doi.org/10.1116/1.4944632]

I. INTRODUCTION

III–V semiconductors are promising materials for integration as channels in n-type metal-oxide-semiconductor field effect transistors. High-mobility properties of III–V channels, such as InGaAs or InAs, will allow the operation voltage to be improved.[1] Implementation of channels in 3D architectures, such as TriGate, FinFETs, requires the development of new characterization methods to provide accurate compositional analysis. Indeed, depth and lateral resolution become critical for accurate determination of heteroepitaxial interfaces for most characterization techniques, such as Rutherford backscattering spectrometry, secondary ion mass spectrometry (SIMS), X-ray photoelectron spectroscopy, etc.

SIMS is widely used in microelectronics for characterizing the interface abruptness and the chemical composition of layers due to its excellent depth resolution and sensitivity. In this work, we use magnetic SIMS and time-of-flight SIMS (TOF-SIMS) to study III–V trenches of 150–200 nm in width grown inside SiO_2 cavities on Si wafers. It was demonstrated that SIMS protocol using an averaged analysis for repetitive structures is promising and can be applied to study the physico-chemical properties of nonplanar structures.[2–4] An average method is based on the assumption that the structures are repetitive, and thus, the signal from many adjacent trenches approaches the signal that would be obtained from one single trench. Moreover, this protocol allows interface abruptness and chemical composition of layers in repetitive structures to be studied and to be correlated with photoluminescence measurements (PL). Furthermore, to obtain accurate chemical depth profiling the mechanism of interaction of the energetic ion beam with the sample should be understood and the sputtering conditions should be adjusted accordingly to maximize the signal of interest and minimize artifacts.

In this work, we discuss how to improve the SIMS depth profiling of III–V trenches containing InGaAs/AlAs quantum wells (QWs) under low energy oxygen bombardment. We show that low energy oxygen sputtering (below 500 eV) induces surface topography formation on InGaAs materials and thus degradation of depth resolution. The formation of ripples under oxygen sputtering for InGaAs and GaAs materials was demonstrated.[5–7]

The effect of the orientation of the oxygen ion beam with respect to the III–V structures on the SIMS depth resolution was also reported.[2] It was shown that parallel orientation of the oxygen ion beam sputtering with respect to the III–V trenches may improve the depth resolution, but sputter induced topography formation is still observed, which may cause the variations of ion yields and sputter rate.[8]

Also, we demonstrate the effect of the incident angle on the depth resolution using pretilted samples. Additionally, we show that sample rotation using TOF-SIMS can be used to reduce the formation of surface roughness; however, no improvement in depth resolution was observed because of shadowing effects in the trench structures. The reported results can be implemented for the characterization of III–V devices monolithically integrated on Si.

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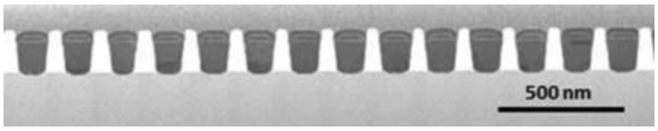

FIG. 1. Cross-sectional STEM image of III–V trenches selectively grown on patterned Si substrates by MOCVD.

II. EXPERIMENT

A. Sample description

The III-As based heterostructures were grown selectively inside SiO_2 cavities of different widths on (100) oriented Si substrates, as shown in Fig. 1. The growth was performed using metalorganic chemical vapor deposition (MOCVD) in an Applied Materials tool. The trimethylgallium, trimethylaluminum, and trimethylindium as group-III precursors and tertiarybutylarsine as the group-V precursor were used. SiO_2 trenches of 180 nm deep aligned along the [110] direction with widths ranging from 100 to 300 nm were patterned on 300 mm Si (100) substrates using standard e-beam lithography and plasma etching. After removing the native oxide by dry NF_3/NH_3 based chemical etching in a Siconi^{TM} preclean chamber, a thin GaAs nucleation layer was grown at low temperature. Then, the temperature was increased to grow a stack of GaAs/AlAs/$In_xGa_{1-x}As$/AlAs/GaAs. The thickness of III–V layers was estimated from cross-section STEM measurements. The In-fraction $x = 0.33$ (nominal $x = 0.3$) in $In_xGa_{1-x}As$ was estimated from μPL measurements performed at room temperature.

B. SIMS and ToF-SIMS analysis conditions

The samples were analyzed with a Cameca SC-Ultra SIMS. The analysis was performed with an O_2^+ ion beam with an impact energy of 500 eV ($I_p = 19.3$ nA), incident at 42° to the sample normal. The sputtered area was limited to a raster of 200 μm × 200 μm with a small field aperture of 400 μm in diameter and analyzed area of 33 μm in diameter. The TOF-SIMS analysis was performed using a TOF-SIMS V ION-TOF instrument using a primary Bi_5^{++} source with 60 keV impact energy (1.05 pA) and an O_2^+ sputter ion beam at an energy of 500 eV (127 nA) and raster size of 200 μm × 200 μm. The scanned area of analysis was defined as 50 μm × 50 μm with 256 × 256 data points.

III. RESULTS AND DISCUSSION

A. Effect of impact energy

Chemical depth profiling of repetitive III–V trenches containing InGaAs QWs arrays and AlAs barrier layers of 8–9 nm and 10 nm in thickness, respectively, grown on nonplanar Si substrates were studied using SIMS. The average depth profiling that includes contributions from an array of around one hundred identical III–V trenches under different oxygen primary ion beam impact energies was studied and the indium depth profiles are shown in Fig. 2. The lateral width of the III–V trenches is around 150 nm. To superpose the different profiles and reduce the artifacts from depth calibration, the monocalibration was used (single average sputter rate).

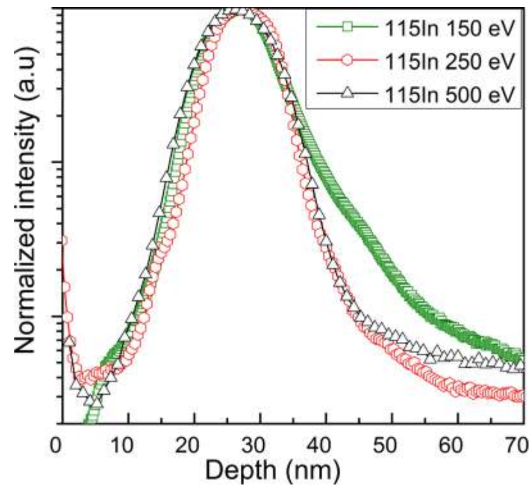

FIG. 2. (Color online) SIMS In depth profiles of InGaAs QWs of 8–9 nm in thickness under low energy oxygen sputtering: (a) 500 eV (triangles), (b) 250 eV (hexagons), and (c) 150 eV (squares).

The depth resolution under low oxygen ion beam bombardment (<500 eV) patterned InGaAs QWs is given in Table I. The effects of primary energy on depth resolution of III-As trenches were studied also on III-As sample grown on planar Si substrate. The planar sample containing the stack of GaAs/AlAs/InGaAs/AlAs/GaAs layers was investigated with similar analysis conditions as those used for patterned InGaAs QWs. The decay length upslope of indium profile (i.e., MOCVD growth direction) in nonplanar InGaAs is slightly higher than for planar InGaAs QW while the downslope decay is similar for both materials. The significant better depth resolution for planar III-As layers was not observed under low energy (<500 eV) oxygen ion beam sputtering.

The effect of the primary energy on the depth resolution profiling of III–V materials is different to that observed for boron doped layers in Si.[9] The best depth resolution in Si material of 0.8 nm/decade is obtained at the lowest primary energy ($E_p = 150$ eV) compared to InGaAs layers, where depth resolution degradation is observed. It can be related to higher surface roughness formation, which is four times higher under 150 eV (rms = 4.3 nm) compared to 500 eV (rms = 0.9 nm) energy under oxygen irradiation approximately 80–90 nm in depth observed using atomic force microscopy (AFM) measurements. Note that although the initial roughness of a single III–V trench is low (rms = 0.5 nm), the presence of the facets on the top of the III–V trenches will degrade the depth resolution on these samples.

TABLE I. Depth resolution of indium profile in InGaAs QWs on patterned Si substrates under different energy of oxygen sputtering.

Primary energy (eV)	500 eV (41°)	250 eV (41°)	150 eV (52°)
Decay length upslope (nm/decade)	5.0	5.1	5.8
Decay length downslope (nm/decade)	5.0	5.0	12.0

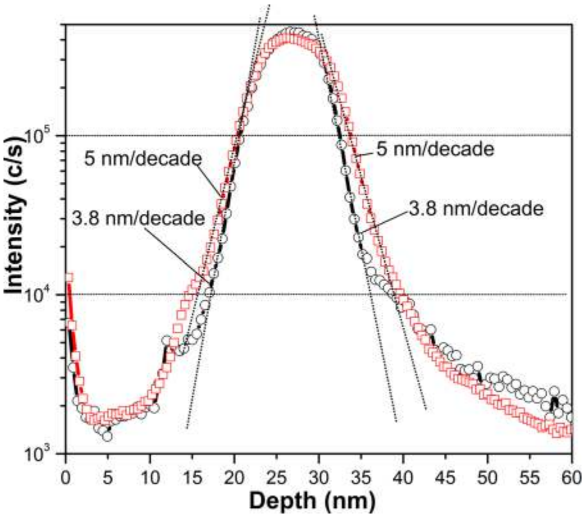

FIG. 3. (Color online) SIMS profile of InGaAs QWs on patterned Si substrates under oxygen sputtering and an incident angle of 50° (black circles), and of 62° (red squares).

B. Effect of incidence angle

To improve SIMS depth resolution, the effect of impact angle (45°–60°) on SIMS depth resolution was studied using a Cameca SC-Ultra SIMS. The impact angle is defined with respect to the sample, 0° being normal incidence. However, in this instrument, the variation of incident angle (∼2°–5°) is limited by the variation of sample voltage and, therefore, requires different beam alignments for each angle. In order to perform the experiments with a single beam alignment, a metallic stand was used to tilt the sample at 12° to increase the impact angle (the sample normal is tilted away from the ion source resulting in a more grazing incidence). Figure 3 shows the comparison of chemical profiles of nonplanar InGaAs/AlAs QWs with (62°) and without sample tilting (50°) at 250 eV oxygen sputtering. Note the monocalibration was applied before superposing the SIMS profiles. The improvement of depth resolution at the InGaAs/AlAs (downslope) interface using the sample tilting of more than 1 nm/decade is observed. A similar improvement was observed for reference planar InGaAs QW samples. However, the experiments performed on boron delta-doping layers in Si demonstrated a different effect on depth resolution using sample tilting. The significant degradation in depth resolution for Si material is observed contrary to InGaAs using sample tilting. It was previously shown that a primary O_2^+ beam at the incidence angle 45° gives the best depth resolution,[9] minimizing the ripple formation during sputtering of Si.

For III–V materials, an increase in sputter rate by more than twice using sample tilting was also observed. We assume that the difference in sputter rate is related to oxygen

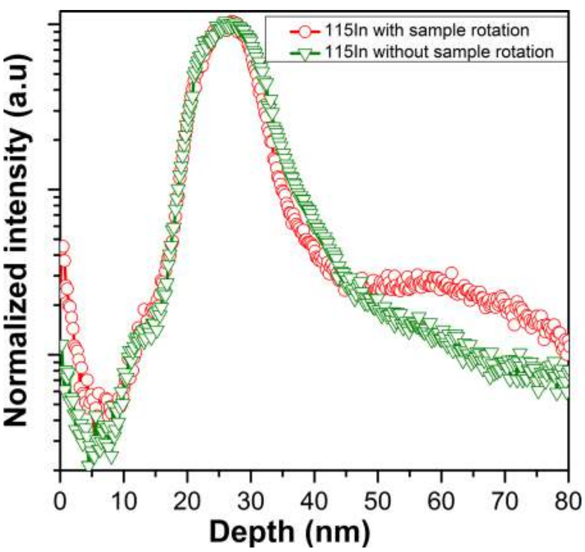

FIG. 4. (Color online) ToF-SIMS depth profiling of III–V trenches selectively grown on patterned Si substrates using sample rotation (red circles) and without (green triangles) under oxygen sputtering of 500 eV and Bi_5^{++} and energy of 60 keV.

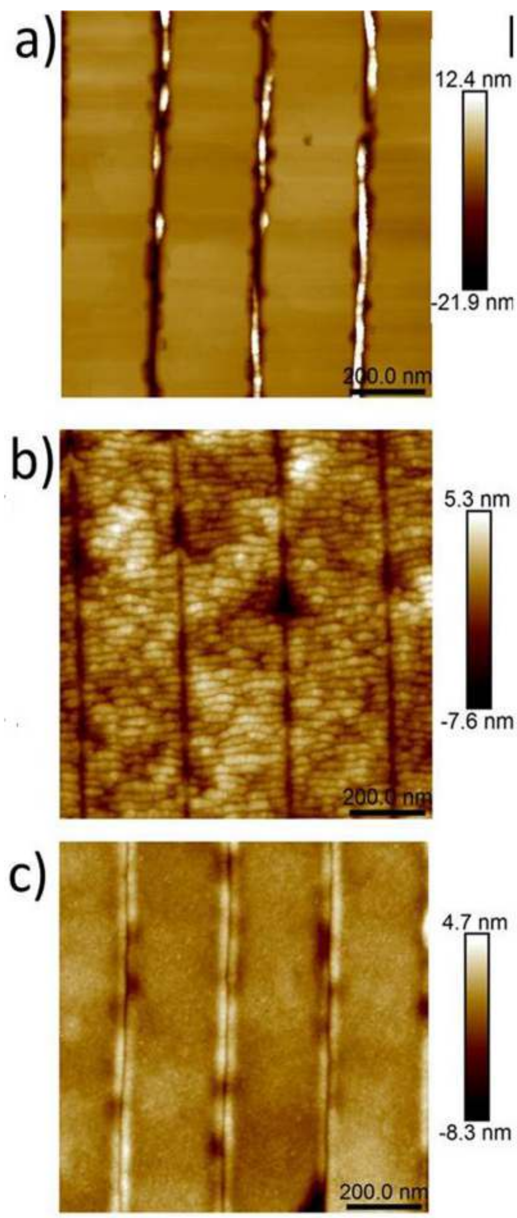

FIG. 5. (Color online) AFM images of III–V trenches (a) initial topography, (b) after sputtering in parallel orientation with respect to III–V trenches, and (c) under sample rotation and oxygen sputtering (500 eV) and Bi_5^{++} (60 keV).

incorporation and the increased ripple amplitude on the III–V surface as observed by AFM.

C. Sample rotation

Figure 4 shows the comparison of depth profiling of III–V trenches grown on patterned Si wafers under oxygen irradiation with and without sample rotation using ToF-SIMS. The III–V trenches were aligned parallel to the oxygen beam during the experiments without sample rotation. Higher depth resolution (6 nm/decade) using sample rotation compared to results obtained without sample rotation (7.5 nm/decade) is observed. The artifact of sputtering and tailing effect on the indium profile using sample rotation can be seen in Fig. 4, as the appearance of a bump at 50 nm in depth. To better understand the improvement with and without sample rotation (in parallel orientation) and artifact of sputtering, AFM measurements were performed at approximately 40 nm in depth. Figure 5 shows that using sample rotation leads to a smooth and flat surface on III–V materials compared to profiles acquired without rotation (parallel orientation) where the surface of the III–V materials roughens. However, high topography variation observed at both sides of the III–V trench/SiO_2 interfaces are similar to those using the perpendicular orientation of the ion beam to the trenches (see Fig. 5). It seems that using sample rotation, although it reduces roughness on the III–V materials, brings the sample into the nonpreferable orientation of the ion beam for most of the depth profile, which leads to a shadowing effect during oxygen sputtering. Note that, in this case, the shadowing effect can be seen at both sides of the trench.

IV. SUMMARY AND CONCLUSIONS

The depth resolution has to be optimized in order to understand SIMS signals correctly and to avoid artifacts for accurate depth profiling of repetitive structures of low lateral dimension. We have studied SIMS depth profiling of InGaAs QWs in SiO_2 cavities on silicon substrates using an averaged analysis. We have demonstrated the effects of the primary energy, the ion beam incident angle on SIMS depth profiling. High depth resolution SIMS profiles (<4 nm/decade) can be obtained using extremely low primary ion energy (<500 eV). However, the results have shown that reducing the primary oxygen ion beam impact energy down to 150 eV leads to an increase in the surface roughness and thus depth resolution degradation. It is explained by the ripple formation observed by AFM measurements under oxygen irradiation. Furthermore, the effect of ion beam orientation with respect to the trenches was studied. Additionally, depth resolution might be affected by the incident angle of oxygen ion beam. It was demonstrated that depth resolution improves when increasing the oxygen ion beam incident angle. Less roughness on the III–V surface under oxygen irradiation using sample rotation was observed by AFM. However, no significant depth resolution improvement and the tailing effect were observed. This can be explained by a shadowing effect at III–V/SiO_2 interfaces due to the sample being perpendicular to the ion beam for part of the time during sample rotation. This was observed using AFM and is consistent with previously reported observations for perpendicular orientation of the ion beam with respect to the trenches, although in the case of sample rotation the shadowing effect is seen on both sides of the III–V trenches.

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[1]A. Del Alamo, Nature **479**, 317 (2011).
[2]V. Gorbenko *et al.*, Phys. Status Solidi RRL **9**, 202 (2015).
[3]V. Gorbenko *et al.*, Microelectron. J. **147**, 219 (2015).
[4]W. Vandervorst, A. Schulze, A. K. Kambham, J. Mody, M. Gilbert, and P. Eyben, Phys. Status Solidi C **11**, 121 (2014).
[5]F. A. Stevie, P. M. Kahora, D. S. Simons, and P. Chi, J. Vac. Sci. Technol., A **6**, 76 (1988).
[6]S. W. MacLaren, J. E. Baker, N. L. Finnegan, and C. M. Loxton, J. Vac. Sci. Technol., A. **10**, 468 (1992).
[7]A. Karen, Y. Nakagawa, M. Hatada, K. Okuno, F. Soeda, and A. Ishitani, Surf. Interface Anal. **23**, 506 (1995).
[8]P. F. A. Alkemade and Z. X. Jiang, J. Vac. Sci. Technol., B **19**, 1699 (2001).
[9]M. Juhel, F. Laugier, D. Delille, C. Wyon, L. F. T. Kwakman, and M. Hopstaken, Appl. Surf. Sci. **252**, 7211 (2006).