Suppression of self-organized surface nanopatterning on GaSb/InAs multilayers induced by low energy oxygen ion bombardment by using simultaneously sample rotation and oxygen flooding

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Abstract

Time of flight secondary ion mass spectrometry (ToF-SIMS) is a well-adapted analytical method for the chemical characterization of concentration profiles in layered or multilayered materials. However, under ion beam bombardment, initially smooth material surface becomes morphologically unstable. This leads to abnormal secondary ion yields and depth profile distortions. In this contribution, we explore the surface topography and roughening evolution induced by O_2^+ ion bombardment on GaSb/InAs multilayers. We demonstrate the formation of nanodots and ripples patterning according to the ion beam energy. Since the latter are undesirable for ToF-SIMS analysis, we managed to totally stop their growth by using simultaneously sample rotation and oxygen flooding. This unprecedented coupling between these two latter mechanisms leads to a significant enhancement in depth profiles resolution.

Introduction

Recent progress in materials science necessitates knowledge and control of matter at the nanometer scale. Nowadays, given the complexity of the devices in terms of chemical composition as well as in dimension, their accurate characterization has become difficult and sometimes challenging. For this purpose, Time of Flight Secondary Ion Mass Spectrometry (ToF-SIMS) has become a crucial tool that is well adapted analytical method for the chemical characterization of concentration profiles in layered or multilayered materials. However, the reliability and accuracy of the interface characterization may be affected by methodological factors, which alter the depth resolution such as: macroscopical or microscopical initial roughness of the material and ion bombardment induced surface topography and roughening. In fact, material surface of initially smooth monocrystals becomes morphologically unstable under ion bombardment. This instability leads to the formation of self-organized nanostructures on the surface of the material such as nanoscale ripples [1], [2], [3], [4] and dots [5], [6], [7], [8]. These nanostructures on semiconductors surface have attracted much interest due to their potential applications in low-dimensional devices, particularly as ordered quantum nanodots for optoelectronics and quantum devices [9], [2], in magnetic storage technology [10] and for selective attachment of specific molecules [11]. On the other hand, extensive efforts in the form of experimental as well as theoretical studies have been devoted to understand the fundamental processes of ripples and dots formation on surfaces subjected to energetic ion bombardment [12]. The pioneering works by Bradley and Harper [13] referred to as BH model was the first successful theoretical approach to explain the ripple formation mechanism on an amorphous material. Later, other models have been also proposed which account for different experimental behaviors such as the presence or absence of saturation for the ripple amplitude, ripple orientation, ripple dynamics as well as the existence or not of kinetic roughening [14], [15].

While the formation of morphological features can be used in various applications, they are extremely undesirable in many occasions especially for depth profiling in ToF-SIMS. This kind of roughening brings about degradation of depth resolution. Therefore, improvement of the interface depth resolution in sputter depth profiling is a very important topic. Accordingly, the aim of this work was to study surface topography and roughening induced by low energy range 0.25 to 2 keV O_2^+ ion bombardment, corresponding to the typical depth profiling regime of the new generation of SIMS instruments. The objects in our experiments consist of GaSb/InAs multilayers deposited on silicon substrate because of their interest for application in opto- and micro-electronics [16], [17], [18]. Our overall objective is to try to examine the conditions under which depth profiling of such material is optimal and reliable.

Experimental

The investigated sample in this work was grown by Metalorganic Chemical Vapor Deposition (MOCVD) which can provide heterostructures with very abrupt interfaces. It consists of a stack of GaSb/InAs layers deposited on 300 mm (001)-Si substrate as shown in Fig. 1. The growth of GaSb layer was performed on (001)-Si substrate with an offcut angle of 0.11° along [110] direction. The substrate was first deoxidized in a Siconi^{TM} pre-clean chamber under an NF_3/NH_3 remote plasma. Prior to the growth, the substrates were heated above 800°C under H_2 atmosphere in the growth chamber to promote double step silicon surface structuration [19]. Subsequently, we used a two-step process to grow GaSb on Si. A nucleation layer between 10 nm and 40 nm was grown at 1 nm/min in the 350-450°C temperature range. The growth was then interrupted and the temperature was increased above 600°C under H_2 atmosphere. A GaSb epilayer was then grown with a V/III ratio around 1 at 30 nm/min. The InAs layer was deposited following a process flow published in previous work [20]. The top GaSb layer was then grown directly on InAs at 1 nm/min in the 350-450°C temperature range.

All experiments were carried out on a ToF-SIMS V from IONTOF. The samples approximately 1 × 1 cm^2 in size were cut from standard 300 mm wafers. Controlled irradiation was carried out at room temperature by O_2^+ ions with ion energies of 0.25, 0.50, 1 and 2 keV. The ion bombardment was performed at a 45° incidence angle with a constant (for each ion energy) ion beam current density, beam raster size (300 × 300 μm^2) and sputtering depth. Sputtering was performed throughout the layers until reaching the bottom GaSb layer up to 105 nm from the surface of the sample (see figure 1.b). After ion irradiation the surface morphology was investigated by atomic force microscopy (AFM) in tapping mode. All measurements were conducted in air by using silicon tips.

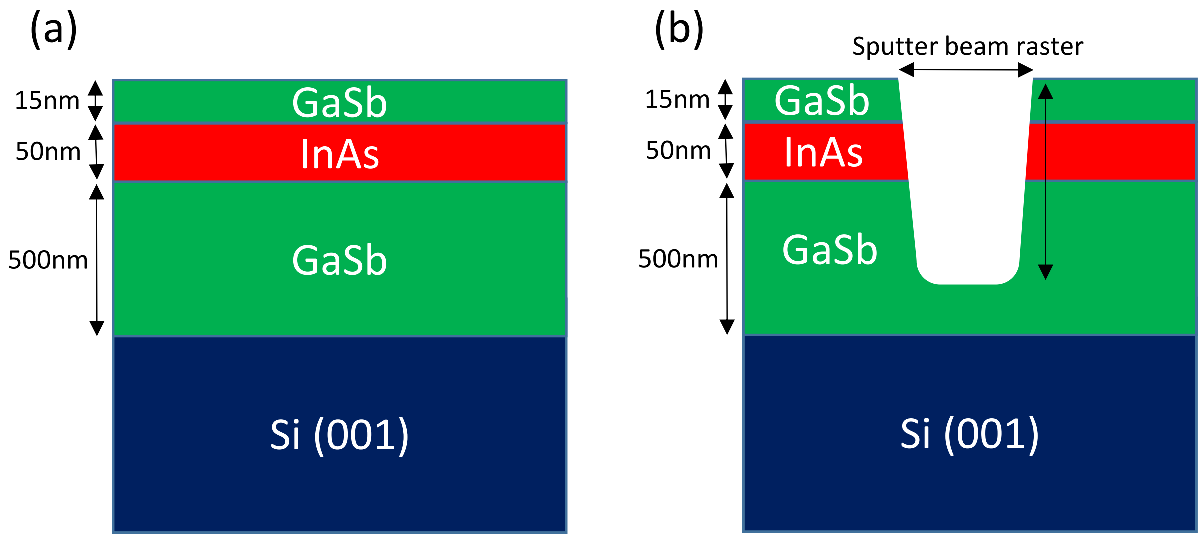

Figure 1: Structure of GaSb/InAs multilayers grown on Si substrate.

Results and discussion

The surface morphology of the sample before irradiation was studied using atomic force microscopy (AFM). The surface exhibits monoatomic steps as it can be seen from figure 2. The Root-Mean-Square (RMS) roughness extracted from a 1×1 μm^2 area of the sample is 0.68 nm.

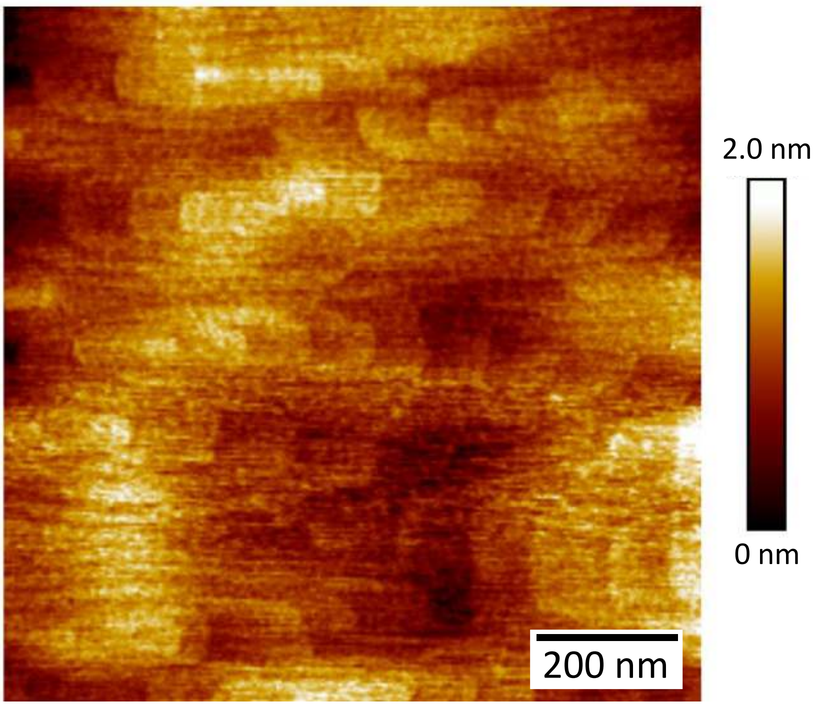

Figure 2: 1×1 μm^2 AFM image of surface topography before ion irradiation. RMS = 0.68 nm.

The optimization of ToF-SIMS conditions is crucial if accurate studies of the quality of interfaces, segregation and quantification in thin layers/structures are to be carried out.

Basically, the depth resolution depends on the primary ion beam (type, energy, incident angle) and the substrate. Therefore, the sample was analyzed by ToF-SIMS using oxygen ion beam with different energy in order to understand its effect. Hereinafter, we will first discuss the formation of the nanopatterns under ion beam bombardment to move on later to their suppression as well as the improvement of depth profile resolution.

A. Patterns formation under ion beam bombardment: transition from nanodots to ripples

Figure 3 shows the GaSb bombarded surface with oxygen ions at 45° incidence angle and different ion beam energies. One can observe the evolution of the surface topography until the formation of well oriented and long range ordered ripples pattern as the energy of the ion beam increases. For low incident beam energies 250 eV and 500 eV, nanodots are already being formed on the sputtered surface as shown in Fig.3.(a) and 3.(b). Increasing ion beam incident energy up to 1 keV leads to a more advanced state of corrugation with larger amplitude and more enhanced ordering. Finally, clearly regular and large rippled structure is formed at 2 keV incident beam energy. The observed pattern at 2 keV presents larger lateral sizes and heights compared to the other previous incident energies. These results demonstrate a transition from an isotropic (e.g. phase separation) to an anisotropic mechanism (e.g. ballistic or stress effects) as the energy is increased. Thus, while the formation of nanodots or ripples was highlighted as incidence angle dependence in the literature, here we prove that it is also reliant on ion beam energy. Moreover, the wave vector of the formed ripples is perpendicular to the incident beam direction. The impact of primary ion beam energy on the surface topography formation can be seen in table 1. Higher surface topography is formed when increasing the impact energy up to 2 keV (RMS=5.35 nm). The smoothest surface was obtained for 250 eV impact energy (RMS=1.45 nm). On the other hand, the wavelength and the amplitude of the nanopatterns become greater with the increase of the incident beam energy as it can be deduced from table 1. All of these observations imply that the topography of the GaSb/InAs surface changes from a less positionally ordered nanodots to a more ordered ripples chain structure aligned perpendicular to the ion beam direction. According to these results, the most convenient sputter regime for the GaSb depth profiling is the 250 eV oxygen ion beam energy. Highly the impact energy highly the surface topography and deterioration of depth resolution occurs. The effect of the primary energy on the depth resolution of GaSb/InAs material is similar to that observed on boron doped layers in Si where the best depth resolution in Si material is obtained at the lowest primary energy [21] and SiGe [22].

B. Patterns suppression and enhancement of depth profile resolution

Many different macro topography formations on materials surface induced by ion bombardment were already observed, such as ripples, cones, pyramids, and terraces… The formation of ripples and changes of ion yield under oxygen sputtering has been reported for many III/V materials such as GaAs, GaSb and InAs [23]. Nevertheless, it was not the case for GaP material under oxygen ion bombardment with the energy of 10.5 keV and the incidence angle of 37°. While the formation of morphological features can be used in various applications, they are undesirable for depth profiling in ToF-SIMS. In fact, this kind of roughening brings about degradation of depth resolution. One method to optimize depth resolution is oxygen flooding inside the chamber where gaseous oxygen is injected close to the sample's surface through a capillary. The oxygen flooding is used to saturate the analysis chamber with oxygen and more precisely at the vicinity of sample surface in order to create a completely oxidized layer at its surface prior to sputtering and to maintain it during the sputtering process. When this complete oxidation condition is achieved, maximum yield enhancement and optimized depth resolution and to suppress ripple formation. Thus, in the second part of our experiments, we performed ToF-SIMS analysis under oxygen flooding.

Figure 4.b shows the AFM image on the GaSb surface bombarded at 250 eV under oxygen leakage. As we notice, formation of small nanodots is observed similarly to the sample bombarded at 250 eV without oxygen flooding (figure 4.a). However, we can note a significant improvement in the surface roughness (RMS=0.57 nm) with respect to the sample analyzed without oxygen leakage (RMS=1.45 nm) as reported in table 2. Thus, we demonstrate that oxygen flooding is an effective method to enhance surface topography in GaSb/InAs multilayers. However, we still have formation of nanopatterns that can deteriorate depth resolution. Thus, in order to try to suppress totally the surface topography formed, we performed depth profiling under sample rotation which is an effective method to improve depth resolution profiling by smoothing the surface during ion beam sputtering [24]. Figure 4.c shows the obtained AFM image with sample rotation. The latter leads to a smooth and flat surface compared to without rotation. No formation of nanopatterns is evidenced. An improvement in the RMS is clearly observed as represented in table 2. Furthermore, in figure 4.d, we performed the experiment under simultaneously O_2 flooding and sample rotation. The latter procedure leads to more improvement in the RMS (see table 2). Figure 5 shows the superposition of the In and Ga depth profiles obtained at 250 eV under O_2^+ sputtering for three cases: a) with sample rotation and without O_2 flooding, b) with O_2 flooding and without sample rotation, c) with sample rotation and O_2 flooding. The depth resolution of upslope of In and Ga profiles is similar. However, higher depth resolution of downslope (3 nm/decade for In and 4 nm/decade for Ga) using sample rotation and O_2 flooding compared to other cases is observed (8 nm/decade for In and 6 nm/decade for Ga for case b) and 6 nm/decade for In and 5 nm/decade for case a)) for Ga. We demonstrate that under these conditions the preservation of the initial surface topography is achieved.

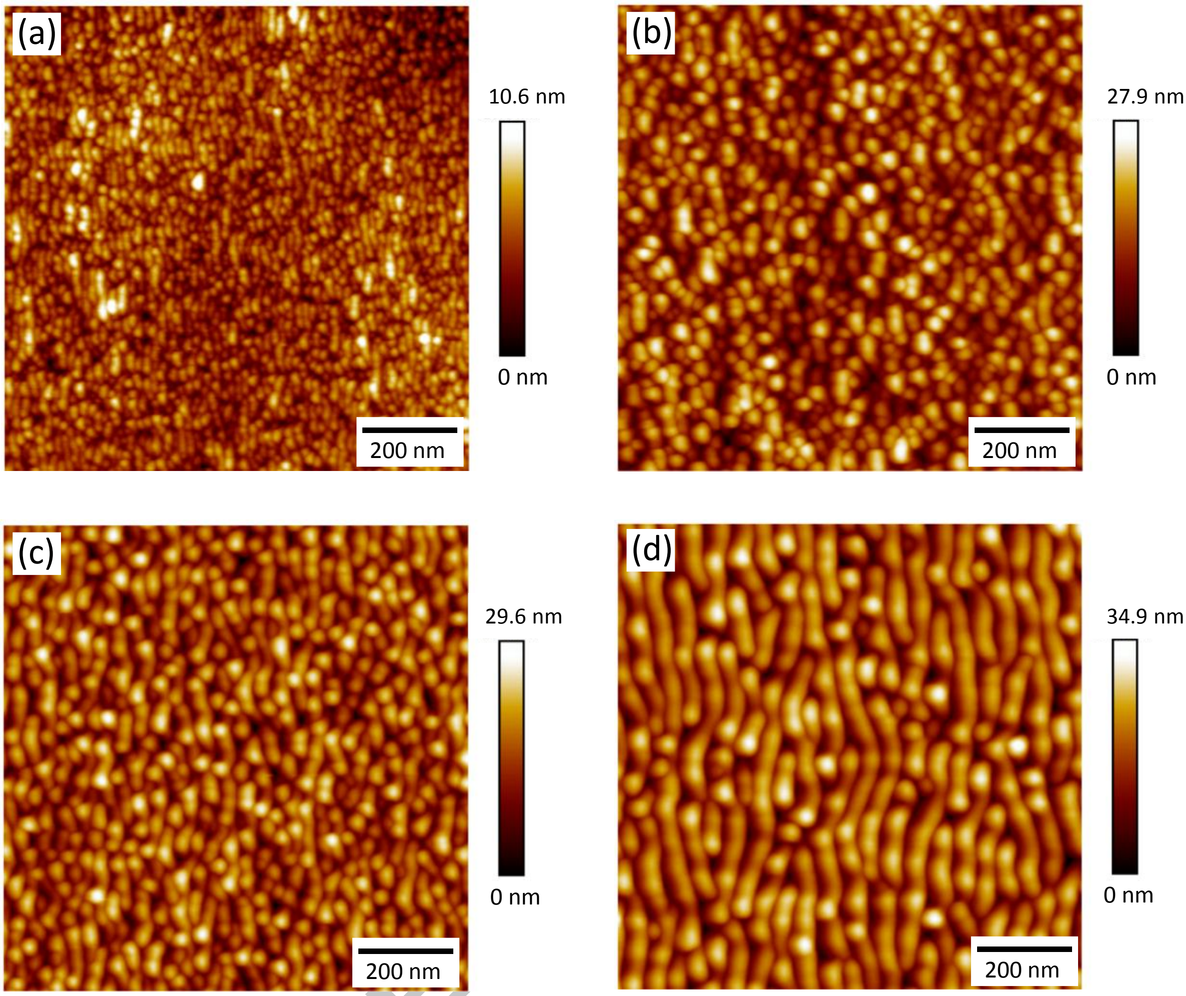

Figure 3: 1×1 µm^2 images of sample bombarded with oxygen ions at 45° incidence angle for different ion beam energies; a) 250 eV, b) 500 eV, c) 1 keV and d) 2 keV.

	Initial topography	**Under 250 eV O_2^+ sputtering**	**Under 500 eV O_2^+ sputtering**	**Under 1 keV O_2^+ sputtering**	**Under 2 keV O_2^+ sputtering**
RMS (1×1 µm^2) in nm	0.68	1.45	3.37	4.03	5.35
Nanopatterns wavelength (λ) in nm	X	25	33	40	52
Nanopatterns amplitude (A) in nm	X	4	5	7	9

Table 1: Roughness measurements of GaSb/InAs multilayers under oxygen bombardment at different energies using TOF-SIMS.

Conclusion

In this study, we investigated the impact of O_2^+ sputtering of GaSb/InAs multilayers by a correlative study using ToF-SIMS and AFM. We highlighted the formation of nanopatterns which evolve with incident beam energy. A continuous improvement of depth resolution by lowering down the O_2^+ beam impact energy to 250 eV is observed. Moreover, we demonstrated that in order to obtain a smooth sputtering of GaSb/InAs, the optimal condition is the use of O_2 flooding and sample rotation which allows suppression of surface nanopatterning induced by the incident ion beam thus leading to higher depth profiles resolution.

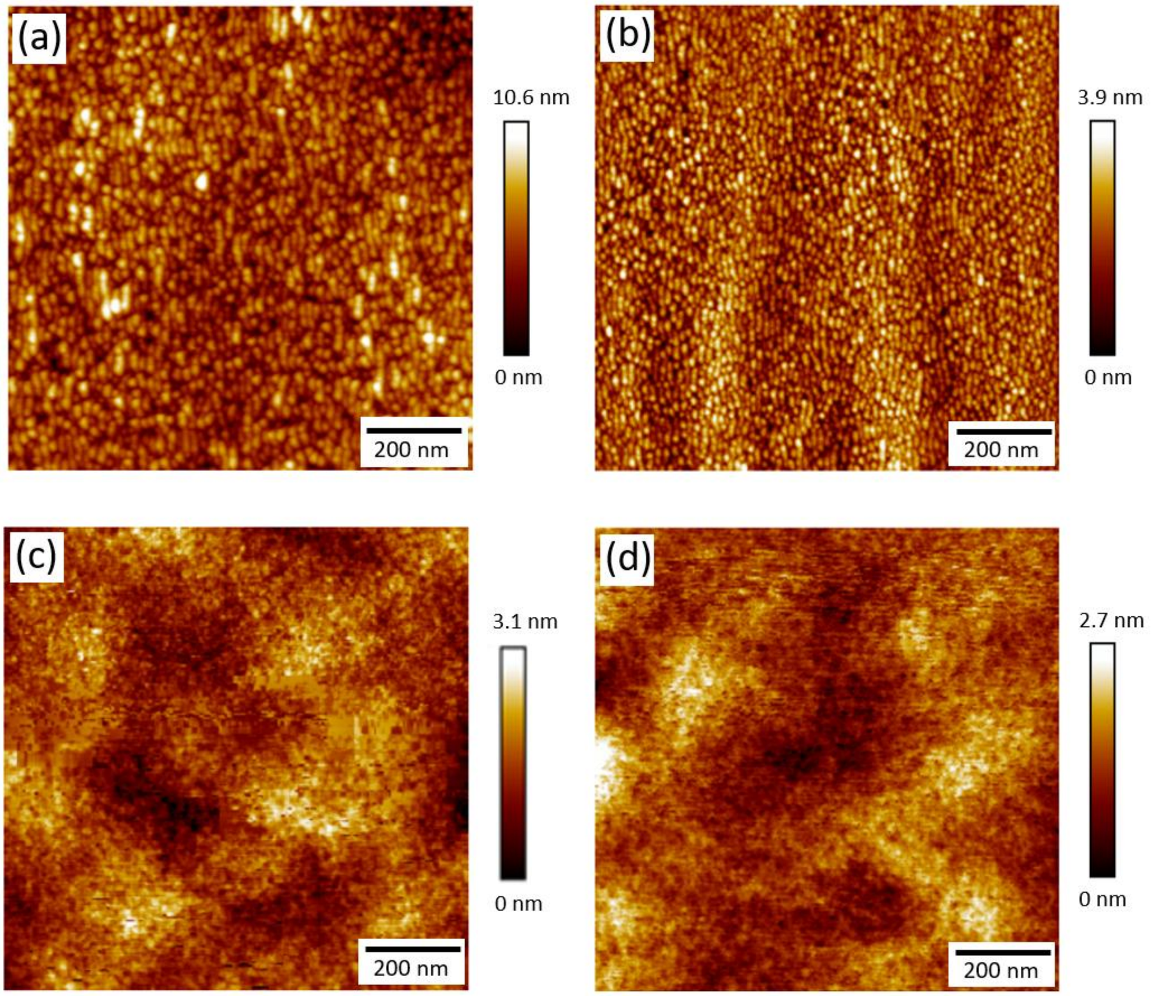

Figure 4: 1×1 μm^2 of sample bombarded under 250 eV oxygen ions at 45° incidence angle. (a) no O_2 flooding and no rotation, (b) O_2 flooding, (c) with rotation (d) with O_2 flooding and sample rotation.

	Under 250 eV O_2^+ sputtering	Under 250 eV O_2^+ sputtering with O_2 flooding	Under 250 eV O_2^+ sputtering with sample rotation	Under 250 eV O_2^+ sputtering with O_2 flooding and sample rotation
RMS (1×1 µm^2) in nm	1.45	0.57	0.46	0.39

Table 2: Roughness measurements of GaSb/InAs multilayers under 250 eV oxygen bombardment with O_2 flooding and with sample rotation using TOF-SIMS.

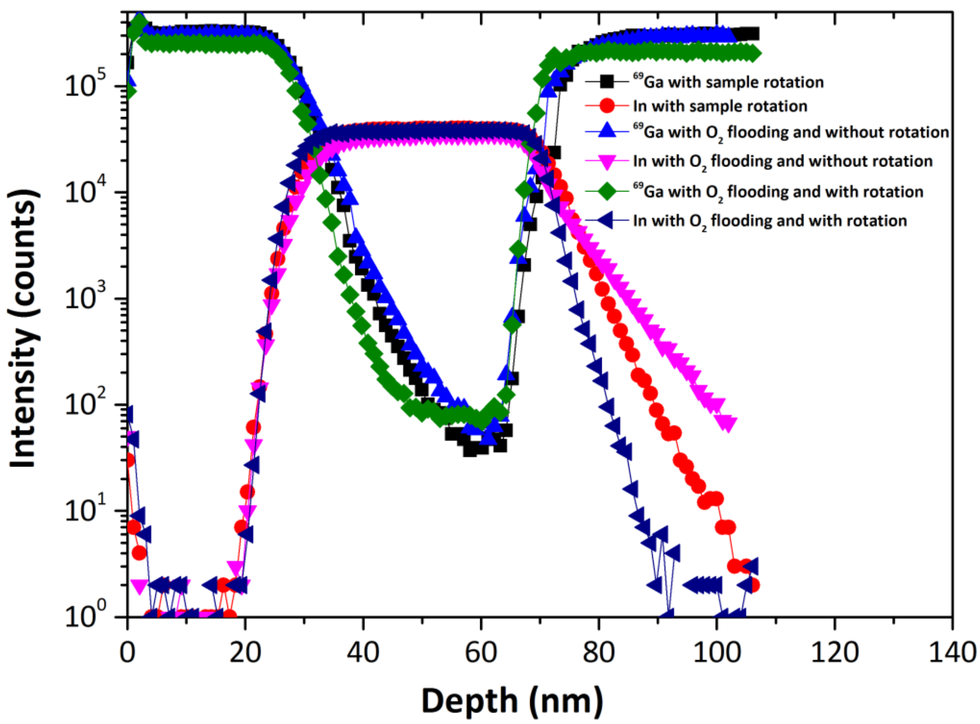

Figure 5: Ga and In depth profiles of the GaSb/InAs multilayers.

Acknowledgments

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