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Structural and electronic characterization of antimonide films made by magnetron sputtering

R Giulian¹, D J Manzo¹, J B Salazar¹, W Just¹, A M H de Andrade², J R Schoffen³, L A B Niekraszewicz¹, J F Dias¹ and F Bernardi³

¹ Ion Implantation Laboratory, Institute of Physics, UFRGS, Av. Bento Gonçalves 9500, Porto Alegre—RS, cep 91501970, Brazil
² Laboratory of Magnetism, Institute of Physics, UFRGS, Av. Bento Gonçalves 9500, Porto Alegre—RS, cep 91501970, Brazil
³ Institute of Physics, UFRGS, Av. Bento Gonçalves 9500, Porto Alegre—RS, cep 91501970, Brazil

E-mail: raquel.giulian@ufrgs.br

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Abstract

AlSb, GaSb and InSb films were deposited by magnetron sputtering on Si and SiO₂/Si substrates and their electronic and structural properties were investigated as a function of film thickness and deposition temperature. Elemental composition and thickness were investigated by Rutherford backscattering spectrometry and particle induced x-ray emission analysis, while x-ray diffraction provided information about phase and structure. Surface chemical composition was investigated by x-ray photoelectron spectroscopy. Here we demonstrate that polycrystalline AlSb films can be produced by magnetron sputtering, where films deposited at 550 °C attain a zincblende phase and exhibit the smallest amount of oxygen (compared to other deposition temperatures). GaSb grown by this technique at room temperature holds an amorphous structure, with excess Sb, but for films deposited at 400 °C the structure is polycrystalline, stoichiometric with a zincblende phase. InSb films with a thickness of 75 nm and thinner, deposited at room temperature, are amorphous and for increasing thickness the films attain a zincblende phase with polycrystalline structure. Sputtering performed at elevated temperatures yields films with improved crystalline quality.

Keywords: antimonide films, magnetron sputtering, XRD, GIXRD, XPS, RBS, PIXE

(Some figures may appear in colour only in the online journal)

1. Introduction

Antimonide-based semiconductors are good candidates for high speed, low power consumption electronic devices. Such properties are greatly pursued for the development of digital and analogue systems for data processing, imaging, communication and sensing, especially in portable equipment and satellites [1]. Crystalline films can be grown by molecular beam epitaxy (MBE), for example, but requires crystalline substrates with similar lattice parameters. Remarkable economy can result from the simple technology of producing amorphous films when compared to the generation of single crystals [2].

An alternative, cost effective technique that has been successfully used for thin film deposition is magnetron sputtering. It is a versatile technique that allows for good control of film thickness, composition and structure on a variety of substrates [3]. The characteristics of sputtering-grown films strongly depend on the deposition temperature, pressure, substrate, growing rate and other parameters [4–7]. But only a few works can be found reporting on the fabrication of antimonide films by magnetron sputtering, in particular regarding AISb thin films (see for example [8–10]). The electrical properties of AISb films are very promising and could be beneficial if better exploited. AISb has the highest bandgap among the
antimonides (~1.63 eV), therefore is suitable for applications in solar cells.

A few works on GaSb films deposited by magnetron sputtering have been reported (see for example [11, 12]) and the influence of deposition parameters on the structure and properties of the films is visible. A comprehensive investigation about the influence of deposition conditions on film properties is necessary to better exploit its technological benefits. There are many devices whose functions rely on both amorphous and crystalline phases, for example, phase-change random access memory devices. Recently, PCRAM devices have been suggested as a promising successor to flash memory, potentially offering fast access times, low power consumption and higher storage density [13]. A large number of Sb-based binary systems have shown phase-change characteristics; however, further investigation is necessary to overcome disadvantages like phase separation, which leads to premature failure. The low melting point of Ga is also ideal for lowering the reset power requirements of real memory devices [14, 15].

InSb films have been deposited by magnetron sputtering on a variety of substrates and the characteristics of the films can be significantly different according to the deposition parameters. InSb can grow epitaxially on sapphire [16], polycrystalline and amorphous on glass [17, 18] or SiO₂ substrates [5]. InSb is well suited for applications in photo-electric and long-wavelength detection, and has been thoroughly explored for those qualities (see for example [4, 19]).

Here we present a comprehensive structural and electronic characterization of non-epitaxial AlSb, GaSb and InSb films deposited on Si and SiO₂/Si substrates by magnetron sputtering. Rutherford backscattering spectrometry (RBS), x-ray diffraction (XRD) and grazing incidence x-ray diffraction (GIXRD) analysis were used to investigate the influence of film thickness and deposition temperature on the structural properties of the films. Surface and near surface chemical composition were probed by x-ray photoelectron spectroscopy (XPS), yielding information about oxidation states.

2. Experimental

AlSb, GaSb and InSb films were grown by magnetron sputtering onto Si (1 0 0) and SiO₂/Si (thermal oxide) substrates with thickness ranging from 20 to 300 nm using an AJA Orion-8 Magnetron Sputtering System in the Laboratório de Conformação Nanométrica at UFRGS. The pressure prior to deposition was below 4 × 10⁻⁶ Torr and during deposition it was kept at 2 mTorr using a 20 sccm Ar constant flow and an adaptive pressure controller. The cathode diameter was 2 in, the cathode-substrate distance was 5.8 in (confocal configuration) with the substrate rotating at 40 rpm. All film thicknesses were set according to calibrated sputtering rates and were confirmed afterwards by cross-sectional scanning electron microscopy (SEM) images (estimated and experimental thickness values agree within the uncertainty (10%)). AlSb films were deposited by the co-sputtering of Al and Sb targets, with target power individually calibrated to yield films with approximately 1:1 atomic concentration: Al target was set at 75 W (DC) (power density of 3.70 W cm⁻²); Sb target was set at 100 W (RF) (power density of 4.93 W cm⁻²). A ~3 nm SiO₂ film was deposited on top of the AlSb film to hamper oxidation. The SiO₂ target power was 90 W (RF) (power density of 4.44 W cm⁻²). Samples were deposited at various temperatures, from 20°C–650 °C (for a given sample, both AlSb and SiO₂ were deposited at the same temperature). GaSb and InSb films were deposited by the sputtering of GaSb and InSb compound, polycrystalline targets (1:1 nominal elemental concentration), respectively, with substrate temperature ranging from 20°C–400 °C. GaSb target power was 50 W (RF) (power density of 2.47 W cm⁻²) while InSb target power was 100 W (RF) (power density of 4.93 W cm⁻²). The nominal thickness of the films, estimated by the deposition rate, ranged from 20 to 300 nm. The purity of the targets was greater than 99.95%.

RBS measurements were performed in a Tandetron accelerator using 1–1.5 MeV He⁺ ions with typical currents ranging from 10 to 20 nA. Backscattered particles were detected by a Si surface barrier detector placed at 15° with respect to the beam direction. Experimental data were simulated using the SIMNR [20] code. The ratio of In/Sn was determined using the particle induced x-ray emission (PIXE) technique, with 2 MeV protons and average current of 0.5 nA. The x-rays induced by the proton beam were detected by a Si(Li) detector with an energy resolution of 155 eV at 5.9 keV, placed at 135° with respect to the beam direction. The PIXE was standardized according to the H method described elsewhere [21]. To that end, thin XRF calibration standard films were employed. The PIXE spectrum was analyzed with the GUPIXWIN software package [22].

XRD measurements were performed in a Diffractometer D500 Siemens using a Bragg–Brentano (θ–2θ) configuration. GIXRD analysis was performed in a Bruker D8 diffractometer using 2θ configuration. Both XRD and GIXRD used Cu Kα radiation (1.5418 Å).

For the XPS measurements, the as-synthesized GaSb and InSb samples (without any pre-treatment) were introduced into the analysis chamber at the D04A-SXS beamline end-station [23] at the Brazilian Synchrotron Light Laboratory (LNLS). The samples were investigated using the long scan, Ga 2p or In 3d, Sb 3d, O 1s and C 1s scan regions. The spectra were collected using an InSb (1 1 1) double-crystal monochromator at fixed photon energies (hv) of 1840 and 3000 eV. The hemispherical electron analyzer (PHOIBOS HAS500 150 R6) was set at a pass energy of 30 eV or 20 eV (for the specific case of O 1s and Sb 3d regions), and the energy step was 0.1 eV with an acquisition time of 100 ms/point. The base pressure used inside the chamber was around 2.0 × 10⁻⁸ mbar. The monochromator photon energy calibration was done at the Si K edge (1839 eV). An additional calibration of the analyzer’s energy was performed using a standard Au foil (Au 4f⁷/₂ peak at 84.0 eV). We also considered the C 1s peak value of 285.2 eV as reference to verify possible charging effects. The XPS measurements were obtained at a 45° takeoff angle at room temperature. XPSpeak version 4.1 was used to fit the XPS spectra. All peaks were adjusted using a Shirley-type background and an asymmetric Gaussian–Lorentzian sum.
function with 25% (1840 eV) or 10% (3000 eV) Lorentzian contribution.

3. Results and discussion

3.1. AlSb films

Figure 1 shows the diffraction patterns of AlSb films deposited on Si by magnetron sputtering at different temperatures. The main peaks corresponding to the zincblende structure of AlSb are clearly visible for samples deposited at 450 °C and 550 °C, and the respective crystallographic orientations are indicated on the graph. The intensity ratio of the peaks for those samples is similar to that of powder standards, suggesting the films are composed of randomly oriented crystallites. There is no evidence of crystalline AlSb formation for samples deposited at room temperature (not shown). For samples deposited at 650 °C the intensity ratio of the peaks dramatically changes and only two peaks remain. Although the position of such peaks coincide with two existing ones from the zincblende fase of AlSb ((200) and (400)), these two peaks may also be attributed to the formation of Sb2O5 monoclinic phase, the one at 2θ equals 29.0° corresponding to the (4 0 0) plane and the other at 60.5° relative to the (1 3 1) plane [24].

Figure 1 also shows the RBS results for the AlSb samples deposited at 550 °C and 650 °C. The relative concentration of each element was estimated using the SIMNRA [20] code and the results are shown in table 1. The thickness of the AlSb film was measured directly from cross-sectional SEM images (not shown). The relative concentration of Al and Sb in the AlSb film is approximately the same, estimated as 39 at.% each, with 22% of O atoms in the film (not considering the oxygen from the SiO2 layer). For samples deposited at 650 °C, a greater fraction of O atoms is incorporated in the film, approximately 66 at.%. Therefore, Al (18%) and Sb (16%) concentrations are proportionally reduced; the Al:Sb proportion, however, remains the same (within the uncertainty).

Das et al [25] suggest the difficulties in producing AlSb films by co-sputtering are mainly because of the difference in sputtering rate of individual elements. In their work, Chen

Table 1. Relative elemental concentration of AlSb, GaSb and InSb films deposited on Si and SiO2/Si by magnetron sputtering at different temperatures and with different thicknesses. The symbol X stands for Al, Ga or In, respectively. All values have an uncertainty of approximately 10%.

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Thickness (nm)</th>
<th>X (at.%)</th>
<th>Sb (at.%)</th>
<th>O (at.%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>AlSb 550</td>
<td>500</td>
<td>39</td>
<td>39</td>
<td>22</td>
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<tr>
<td>650</td>
<td>500</td>
<td>18</td>
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<td>GaSb 20</td>
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<td>InSb 20</td>
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</table>
found the deposition rate of Sb is about four times that of Al (both operating in DC mode). The results shown here indicate that AlSb films formed by co-sputtering exhibit comparable amounts of Al and Sb elements, according to the RBS results. The sputtering yield of Al and Sb targets are indeed very different, which was compensated by applying RF power in the Sb source, and DC power in the Al source. This way, the sputtering rate of Sb became comparable to that of Al, allowing a better control of film thickness and stoichiometry.

The structure of AlSb films deposited by magnetron sputtering is significantly influenced by deposition temperature. Samples deposited at room temperature are amorphous and the absence of a crystalline structure makes it difficult to identify the formation of AlSb compound. The zincblende phase of AlSb becomes significant at 450 °C and above, as shown in figure 1. The structure of AlSb films deposited by pulsed laser ablation [25] or thermal evaporation [8] also exhibits a strong dependence with deposition or annealing temperature. According to Lal et al for AlSb films grown by thermal evaporation above 300 °C, degradation is observed and the film quality is reduced, with regions of excess Sb becoming apparent in selected area diffraction (SAD) analysis. We also observed a strong dependence of film quality with deposition temperature, measured by the relative fraction of O atoms in the film and the quality of the diffraction data. The XRD of samples deposited at 650 °C exhibit only two peaks, located at 2θ equals 29.0° and 60.5°. These could be due to the growth of crystallites in a preferred orientation, but also to the formation of Sb2O5. The RBS results undoubtedly show the presence of O for samples annealed at the highest temperature. There is no indication of an amorphous phase in the XRD results, which leads to the assumption that a crystalline oxide phase may be formed in films deposited at high temperatures. Sb2O5 is stable in its cubic or orthorhombic phase at temperatures up to 650 °C [26]. Sb2O5 nanocomposites can also be formed at elevated temperatures [27].

The present results suggest the ideal conditions to grow polycrystalline AlSb films on Si substrate by magnetron sputtering require deposition temperatures around 550 °C. Polycrystalline AlSb films were also obtained by Chen et al [10] for samples annealed in Ar after sputtering deposition, at 530–560 °C. The post annealing treatment performed by them, however, did not inhibit the formation of an amorphous phase, which is not present in our case. Therefore, it seems advantageous to perform the sputtering deposition at elevated temperatures, rather than annealing the samples afterwards. Nonetheless, we could still observe film degradation with time. For samples stored in atmospheric pressure at room temperature, the lifetime of AlSb films is a few months. After that the films oxidize and degrade.

3.2. GaSb films

Figure 2 shows RBS analysis of GaSb films deposited on SiO2/Si by magnetron sputtering, with the relative concentration of each element presented in table 1. Room temperature depositions result in films with excess Sb, despite the sputtering target being 1:1 polycrystalline GaSb. RBS results simulated with the SIMNRA code [20] suggest the presence of O, whose relative concentration is greater the thinner the film (not considering the oxygen from the SiO2 film). The O concentration is thickness-dependent, in spite of the Ga/Sb ratio remaining constant. According to these results, there are about two Sb atoms for every Ga one in the GaSb samples deposited at room temperature.

GaSb films 300 nm thick were also deposited on SiO2/Si with various temperatures, and the RBS for selected samples are shown on the right panel of figure 2. Clearly, film thickness and Sb/Ga relative concentration change dramatically for samples deposited at 400 °C, approaching 1:1 atomic concentration. In addition, the GaSb film appears 30% thinner in
samples deposited at the highest temperature, in comparison to GaSb films deposited at lower temperatures (despite the same deposition time and sputtering target power). Magnetron sputtering of the GaSb compound target results in non-stoichiometric GaSb films for deposition temperatures up to 300 °C, but at 400 °C the film not only becomes stoichiometric, but also the structure of the film becomes polycrystalline. GIXRD analysis for GaSb samples grown at different temperatures are shown in figure 3. Films deposited at temperatures up to 200 °C are clearly amorphous. At 300 °C, various peaks appear in the diffraction pattern, attributed to the formation of a mixed GaSb zincblende phase, metallic Sb and Sb2O3 (sernamontite). Samples deposited at 400 °C show a diffraction pattern with well-defined peaks belonging to the zincblende phase of GaSb, whose amplitudes resemble that of powder standards, suggesting the film is composed of randomly oriented crystallites. The shoulder on the left side of the peak corresponding to the (1 1 1) plane is indicative of a remaining Sb phase.

Sputtering deposition at 400 °C, as shown here, results in GaSb films with superior crystalline quality, when compared to lower temperature depositions. The fraction of Ga and Sb atoms is approximately the same, according to RBS analysis, and the films attain a zincblende structure. The excess Sb observed in samples annealed at lower temperatures may desorb when the substrate temperature is held to 400 °C, as also shown by Nguyen et al [28]. Other reports also show the formation of GaSb phase by magnetron sputtering requires high temperature annealing [14, 15]. The referred works, however, show that samples deposited at room temperature and annealed afterwards at 500 °C still retain significant contribution of Sb phase.

Kalkan et al [12] show GaSb films, 300 nm thick, deposited by magnetron sputtering at room temperature, are amorphous and the deposited films are almost stoichiometric. They performed sputtering deposition from a stoichiometric GaSb source in DC mode with a sputtering power of 25 W. In our case, the sputtering power was 50 W in RF mode which resulted in amorphous, Sb-rich films (for room temperature depositions). With increasing deposition temperature (in our case), the excess Sb most likely evaporated and, at 400 °C polycrystalline, GaSb films were formed. Thermal annealing has been known for a long time to be an efficient way of improving crystalline quality, promoting the annihilation and coalescence of defects at high temperatures. In addition, the structure of GaSb films is strongly influenced by film thickness, not only for sputtering deposited films, as shown here, but also for films grown by the MBE technique, as shown by Rodriguez et al [29].

Figure 4 shows the XPS spectra at the Ga 2p3/2 and Sb 3d3/2 electronic levels of GaSb films 75 nm thick, deposited at room temperature. Since the Sb 3d3/2 peak overlaps with the O 1s one, the Sb 3d3/2 peak was analysed in order to obtain the chemical components associated. The inelastic mean free path of the photoelectrons coming from the Ga 2p3/2 and Sb 3d3/2 electronic levels are, respectively, 18 and 27 Å (\(h\nu = 1840\) eV) and 37 and 45 Å (\(h\nu = 3000\) eV) [30]. The Ga 2p3/2 analysis shows the presence of a component at 1118.0 eV that can be associated to the Ga–O bond [31]. The Sb 3d3/2 XPS spectra shows the presence of two different components at 537.3 eV and 539.8 eV. The former can be associated to Sb–Sb bonds while the last one is characteristic of antimony oxide (Sb–O) [32–34]. The ratio between the intensities associated to the Sb–Sb (I_{Sb–Sb}) and Sb–O (I_{Sb–O}) components, I_{Sb–Sb}/I_{Sb–O}, increases from 0.35 to 0.87 when increasing the depth probed in the sample (from \(h\nu = 1840\) eV to 3000 eV). It shows that the oxidation started at the surface region and the bulk region should be less oxidised.
InSb films were also studied as a function of thickness and deposition temperature. Figure 5 shows XRD analysis of InSb films deposited at room temperature with various thicknesses. No particular structure can be seen in films up to 75 nm thick. At 100 nm, InSb films show a polycrystalline structure with preferential orientation along the 220 direction. The diffraction peak located at 22.1° is indicative of cubic seramontite \(\text{Sb}_2\text{O}_3\) phase (COD 1011201). For 100 nm thick films and beyond, the area of the diffraction peaks scales linearly with film thickness.

As pointed by Scholte [17], crystallization kinetics proceeds by nucleation and subsequent diffusion-limited growth. Sputtering deposited films exhibit high activation energy, especially for thicknesses below 100 nm, mainly due to the high barrier against nucleation. The growth of InSb films by magnetron sputtering was also reported by Zens et al [5], their results, however, show the formation of polycrystalline films only after annealing at temperatures beyond 200 °C. Here, on the contrary, we show that films deposited at room temperature are also polycrystalline, if sufficiently thick (beyond 75 nm).

GIXRD analyses were performed on samples with 300 nm thick InSb films deposited at various temperatures, and the results are shown in figure 6. Films deposited at 400 °C show the most prominent peaks, with an amplitude ratio that resembles that of the powder diffraction data; hence, we infer the films may be composed of randomly oriented crystallites. Samples deposited at room temperature and 200 °C exhibit the \(\text{Sb}_2\text{O}_3\) contribution (shoulder to the left of the (1 1 1) peak).

The elemental composition of the films was probed by RBS and PIXE analyses. The RBS technique has no resolution to distinguish between the In and Sb backscattered ions, given these two elements have close atomic mass. PIXE analysis of InSb samples (not shown) reveals that the relative concentration of these elements is the same within the uncertainty (85 µg cm\(^{-2}\) and 83 µg cm\(^{-2}\), respectively, with an uncertainty of 5%). The RBS spectra were simulated with the SIMNRA code [20] to estimate the relative concentration of InSb and oxygen in the films and the results are listed in table 1. The spectra for selected samples, 300 nm thick, deposited at room temperature and 400 °C, are shown in the main panel of figure 7. The inset shows a magnified view of the peak corresponding to the InSb for samples with different thicknesses.

Combining the RBS and GIXRD results we can see the InSb films on SiO\(_2\)/Si substrate has a well-defined interface when deposited at low temperatures (up to 200 °C). At 400 °C the straggling becomes non-negligible, and at the same time the InSb film becomes more crystalline. Diffraction from oxide phases is not apparent at the highest deposition temperature.

Figure 8 shows XPS results for InSb samples 75 nm thick, deposited at room temperature, measured with 1840 eV (bottom) and 3000 eV (top) photon energies. In this case, the inelastic mean free path of the photoelectrons coming from the In 3d\(_{3/2}\) and Sb 3d\(_{3/2}\) electronic levels are, respectively, 33 and 27 Å \((h\nu = 1840\,\text{eV})\) and 52 and 45 Å \((h\nu = 3000\,\text{eV})\) [30]. The Sb 3d\(_{3/2}\) XPS spectra are shown on the left, with two main components related to the In–Sb (537.0 eV) and Sb–O (539.7 eV) bonds [34]. The ratio between the components, \(I_{\text{Sb,3d}}/I_{\text{Sb,5s}}\) increases from 0.75 to 1.15 when increasing the depth probed in the sample. Again, the contribution from the Sb–O component is greater for lower photon energies, meaning...
there is a relatively more appreciable amount of Sb–O bonds closer to the surface. The In 3d5/2 spectra is also shown on the right panel of figure 8. The contribution from In–O appears separated by 0.9 eV from the In–Sb one [34]. The ratio between components, \( I_{\text{In-Sb}}/I_{\text{In-O}} \), is approximately constant and equal to 1.11 (1.27) for \( h\nu = 1840 \text{ eV} \) (\( h\nu = 3000 \text{ eV} \)), showing no significant enrichment of any In component at the surface. Comparing with the XPS results obtained from the GaSb films, it is possible to observe an enrichment of the Sb–O component at the surface for both films.

4. Conclusions

In summary, we studied the formation of AlSb, GaSb and InSb films produced by magnetron sputtering on Si and SiO\(_2\)/Si as a function of film thickness and deposition temperature. Polycrystalline, zincblende phase AlSb films were obtained with deposition temperatures between 450 °C and 550 °C. Higher or lower temperatures result in amorphous films, dominated by oxygen contamination. GaSb films deposited at room temperature are amorphous with excess Sb. Sputtering of 300 nm thick films, at 400 °C, yields polycrystalline, stoichiometric GaSb films with zincblende phase. XPS analysis reveals the formation of Sb–O at the surface of GaSb films (deposited at room temperature). InSb films formed by room temperature sputtering can be amorphous, if films are thinner than 75 nm, or polycrystalline, for 100 nm thick films and beyond. Increasing deposition temperature promotes the growth of InSb films with better crystalline quality, still polycrystalline in nature. The surface of InSb films is a mixture In–Sb, as well as indium and antimony oxide compounds.

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