

EFFECT OF THE ANTIMONY THIN-FILM DEPOSITION SEQUENCE ON COPPER-SILICON INTERDIFFUSION

VPLIV ZAPOREDJA NANOSA TANKIH PLASTI ANTIMONA NA INTERDIFUZIJO BAKER-SILICIJ

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In this work we present a study of the effect of an antimony layer on the interdiffusion and formation of copper silicides while inverting the sequence of Cu and Sb deposition on Si(111) substrates. Thermal evaporation was used to deposit Cu/Sb and Sb/Cu bilayers on a Si(111) substrate heated at 100 °C, without breaking the vacuum. XRD and RBS analysis showed, for samples heat treated at 200 °C and 400 °C, a segregation of the three elements (i.e., Cu, Sb and Si) to the surface and diffusion in bulk ending in the formation of a layer made of a mixture containing the three elements at the samples' surface. After 200 °C annealing, in the Cu/Sb/Si system, we observed the formation of only Cu₂Sb, and for the Sb/Cu/Si system, there is the formation of the Cu₃Si and Cu₂Sb phases; after 400 °C annealing, the Cu-Sb-Si mixture is formed by the cohabitation of the Cu₃Si silicide and the Cu₂Sb intermetallic compound in the Cu/Sb/Si sample and only Cu₂Sb in the Sb/Cu/Si sample. For the Cu/Sb/Si sample, annealed at 400 °C, the SEM micrographs exhibit compound formation with crystallites that have a trapezoidal shape.

Keywords: thin film, PVD, diffusion, copper-antimony compound, copper silicide, Rutherford backscattering, scanning electron microscopy, X-ray diffraction

V delu predstavljamo študijo vpliva plasti antimona na interdifuzijo in formiranje bakrovih silicidov pri inverziji zaporedja nanosa Cu in Sb na Si(111)-podlago. Nanos plasti Cu/Sb in Sb/Cu je bil izvršen s termičnim izhlapevanjem na Si(111)-substrat pri 100 °C brez prekinitve vakuuma. XRD- in RBS-analize vzorcev, žarjenih pri 200 °C in 400 °C, so pokazale segregacijo treh elementov (Cu, Sb in Si) na površini podlage in difuzijo masivnega materiala s tvorbo plasti zmesi treh elementov na površini podlage. Po žarjenju sistema Cu/Sb/Si pri 200 °C je bil opažen nastanek Cu₂Sb, v sistemu Sb/Cu/Si pa nastanek faz Cu₃Si in Cu₂Sb. Po žarjenju pri 400 °C je bilo ugotovljeno sobivanje silicida Cu₃Si in intermetalne spojine Cu₂Sb pri vzorcu Cu/Sb/Si in samo Cu₂Sb pri vzorcu Sb/Cu/Si. Pri vzorcu Cu/Sb/Si, žarjenem pri 400 °C, SEM-posnetki kažejo nastanek spojin s kristali trapezoidne oblike.

Ključne besede: tanke plasti, PVD, difuzija, spojina baker-antimon, bakrov silicid, Rutherfordovo povratno sipanje, vrstična elektronska mikroskopija, difrakcija rentgenskih žarkov

1 INTRODUCTION

Antimony and copper are important elements for the design of silicon-based electronics devices; indeed, copper is predominant in interconnect metallization material for deep sub-micrometer technology due to its relatively low resistivity and electromigration resistance¹⁻³, and antimony is most frequently used as an n-type doping element during silicon crystal growth^{3,4}. Unfortunately, the extensive diffusion of Cu in Si is detrimental to the electrical performance of devices, because Cu can form recombination-generation centers in the active regions of Si. Additionally, it reacts with silicon at very low temperatures, even below 150 °C, forming Cu-Si silicides⁵⁻¹⁰ hence, diffusion barriers (e.g., an Sb layer) are necessary between the metallization copper and Si substrate. To the best of our knowledge, no study on the Cu-Sb-Si thin film system has been published in the literature so far, though a study of the Cu-Sb thin-film system deposited on glass substrate, annealed up to 600 °C, has shown that during annealing at up to 300°C, only the Cu₂Sb compound is formed.

During subsequent heat treatments from 350 °C to 600 °C, the Cu₉Sb₂ phase nucleates and grows at the expense of the Cu₂Sb phase¹¹. In a previous work^{12,13}, the effect of temperature and doping-element redistribution on atomic interdiffusion between a thin copper layer and monocrystalline silicon, implanted and Sb⁺ unimplanted with different doses, was investigated. The results show the growth and formation of Cu₃Si and Cu₄Si silicides under crystallites shape dispatched on the sample surface, independently of the implantation dose. On the other hand, it was established that the copper layer is less and less consumed as the antimony dose increases, resulting in an accumulation of Sb ions at the silicide/Si interface and in the silicide layer close to the surface. However, the low antimony quantity in the presence has not led to an understanding of the reactions which could take place between the three elements and the inter diffusion mechanism involving the Sb and Cu layers and the Si substrate.

In the present study, we focus on copper, antimony and silicon atoms' interdiffusion in the Cu-Sb-Si thin-layers system, while inverting the sequence of evapo-

ration of the Cu and Sb thin films on monocrystalline (111) Si. Indeed, from a metallurgical point of view, it is very interesting to study the Sb/Cu/Si and Cu/Sb/Si sequences in order to obtain some information about the influence of Sb on the diffusion process and on the formation of the different phases.

2 EXPERIMENTAL PROCEDURES

N-type monocrystalline(111)-oriented silicon wafers with a resistivity of 1–3 Ω cm were used as substrates. In order to eliminate as far as possible the residual layer of silicon oxide, the wafers were etched in 10 % hydrofluoric (HF) acid for 20 s and rinsed in de-ionized water prior to loading into the vacuum system. Copper and antimony thin films were thermally evaporated alternatively, Cu/Sb as well as Sb/Cu, onto the Si(111) substrate heated at 100 °C for a better adhesion of the antimony layer. These evaporations were performed without breaking the vacuum of $5.23 \cdot 10^{-7}$ mbar obtained with a turbomolecular pump. An in-situ quartz crystal oscillator allows measurement of the thickness of the copper and antimony layers, which were 70 nm and 30 nm, respectively, in the Cu/Sb systems, and 100 nm and 30 nm, respectively, in Sb/Cu layers. In order to promote diffusion, conventional annealing treatments were performed at temperatures of 200 °C and 400 °C, for 45 min, in a quartz tube, under a vacuum of about $2.66 \cdot 10^{-7}$ mbar. It is important to note that the first steps range of the reaction at the different interfaces happen at these temperatures of annealing.

The surface morphology characterization of the samples, before and after the heat treatments, was carried out using a JEOL JSM-6460 scanning electron microscope with an accelerating voltage of 20 kV and an EDX analyzer. The primary energy of the electron beam is chosen to be equal to 10 keV in order to limit the analyzed depth to about 300 nm into the surface. A

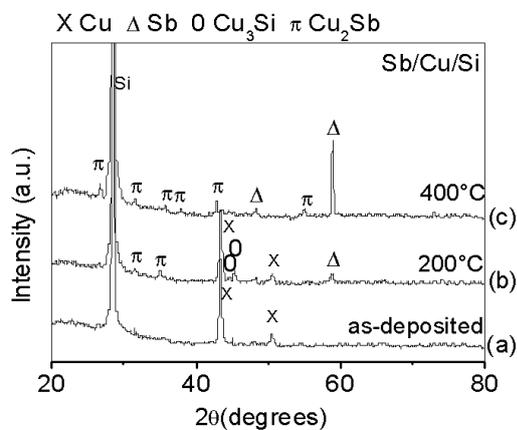


Figure 1: X-ray diffraction patterns of Sb/Cu bilayers on Si(111) substrate heated at 100 °C: a) as-deposited and annealed at b) 200 °C and c) 400 °C

Slika 1: Rentgenski uklonski spektri Sb/Cu-plasti na Si(111)-podlago pri 100 °C: a) nanoseni in žarjeni pri b) 200 °C in c) 400 °C

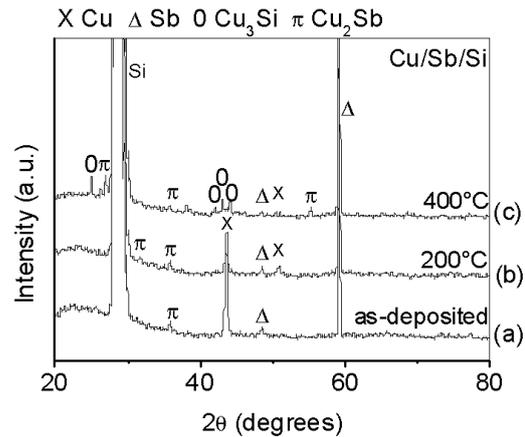


Figure 2: X-ray diffraction patterns of Cu/Sb bilayers on Si(111) substrate heated at 100 °C: a) as-deposited and annealed at b) 200 °C and c) 400 °C

Slika 2: Rentgenski uklonski spektri Cu/Sb-plasti na Si(111)-podlago pri 100 °C: a) nanoseni in žarjeni pri b) 200 °C in c) 400 °C

Siemens D5000 diffractometer, in θ – 2θ mode, was used to identify the formed compounds. The concentration profiles of the Cu, Sb and Si were determined with the help of spectra simulated with the RUMP program by fitting the experimental RBS spectra recorded at (2 MeV, $^4\text{He}^+$) with the detector positioned at 160° relative to the beam.

3 RESULTS AND DISCUSSION

Typical X-ray diffraction patterns corresponding to the Sb/Cu/Si(111) and Cu/Sb/Si(111) systems, as deposited, are shown in **Figures 1a** and **2a**, respectively. The nominal preheating of the substrate at 100 °C was used during evaporation in order to enhance the in-surface adhesion. On the XRD diagram corresponding to the Sb/Cu bilayers deposited on the Si(111), there is

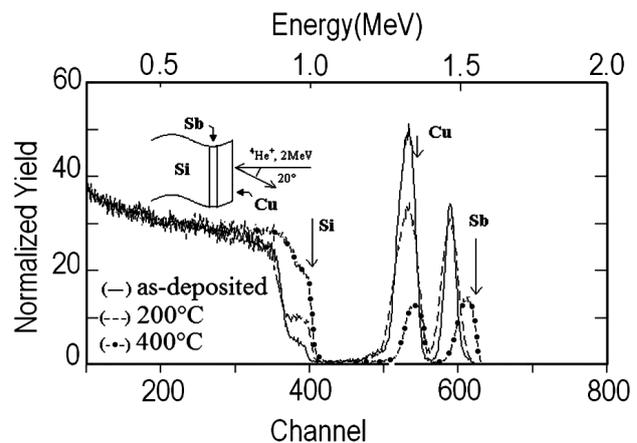


Figure 3: Rutherford backscattering spectra of Cu/Sb bilayers on silicon substrate heated at 100 °C: as-deposited and annealed at 200 °C and 400 °C

Slika 3: Spektri Ruthefordovega povratnega sipanja za Cu/Sb-plasti na Si(111)-podlagi pri 100 °C: nanoseni in žarjeni pri 200 °C in 400 °C

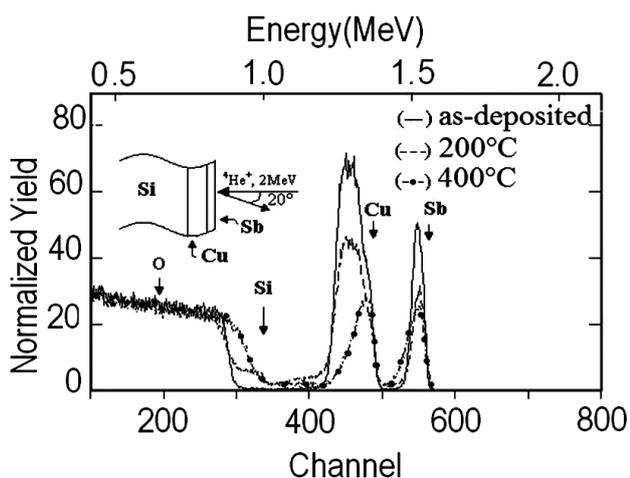
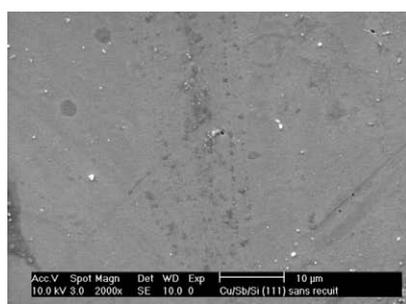


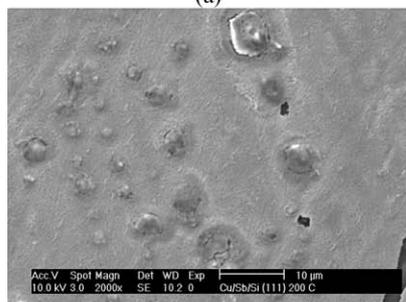
Figure 4: Rutherford backscattering spectra of Sb/Cu bilayers on silicon substrate heated at 100 °C: as-deposited and annealed at 200 °C and 400 °C

Slika 4: Spektri Ruthefordovega povratnega sipanja za Sb/Cu-plasti na Si(111)-podlagi pri 100 °C: naneseni in žarjeni pri 200 °C in 400 °C

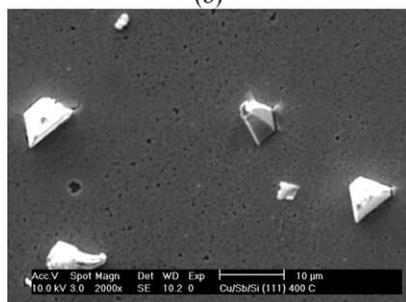
clear evidence of the main (111)Cu and (200)Cu reflection lines, and the absence of that of the evaporated antimony layer, which could be amorphous or formed from nanometric grains. In the Cu/Sb/Si sample, in addition to the high texturization of the antimony layer, we can see, before any annealing, the apparition of the Cu₂Sb intermetallic compound. RBS spectra, for both as-deposited samples, shown in **Figure 3** and **Figure 4**, reveal that the different interfaces are not abrupt. This means that the preheating of the substrate during evaporation has already led to atomic interdiffusion at the different interfaces. Particularly in the Cu/Sb/Si sequence case, where the RBS spectrum shows a slight shift of the silicon signal towards the high-energies side, with a 30 % Si concentration in the outer layer. This is synonymous with the silicon atoms segregation to the sample surface, which has as a consequence the beginning of the asperities formation shown on the corresponding micrograph, (**Figure 5a**). On the other hand, in the Sb/Cu/Si system, we can see the displace-



(a)



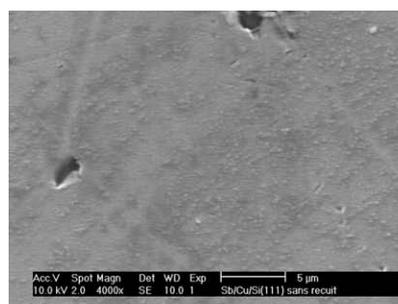
(b)



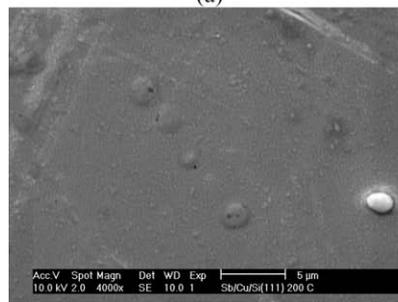
(c)

Figure 5: SEM micrographs from the surfaces of Cu/Sb bilayers on silicon substrate heated at 100 °C: a) as-deposited and heat treated at b) 200 °C and c) 400 °C

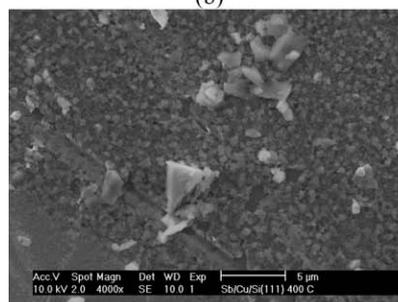
Slika 5: SEM-posnetki površine plasti Cu/Sb na Si(111)-podlagi pri 100 °C: a) naneseni in žarjeni pri b) 200 °C in c) 400 °C



(a)



(b)



(c)

Figure 6: SEM micrographs from the surfaces of Sb/Cu bilayers on silicon substrate heated at 100 °C: a) as-deposited and heat treated at b) 200 °C and c) 400 °C

Slika 6: SEM-posnetki površine plasti Sb/Cu na Si(111)-podlagi pri 100 °C: a) naneseni in žarjeni pri b) 200 °C in c) 400 °C

ment of the Cu RBS spectrum to lower energy, due to its diffusion into the silicon substrate. Again, a shoulder appears on the right-hand side of the Cu spectrum, which means that an appreciable amount of the mole fraction of Cu ($x = 68\%$) has already segregated to the surface through the antimony top layer. In this latter case, the diffusion of copper atoms is favored. In this pre-reactional diffusion, the displacement of Sb atoms appears timorous because of the low diffusion coefficient of Sb into Si. Indeed, according to Fahey et al.¹⁴, the Sb diffusion coefficient in Si, under equilibrium conditions, is $1.2 \cdot 10^{-19}$ cm²/s. However, in the solid state, antimony is partially soluble in copper, with the solubility decreasing with temperature, and, it is more plausible that Cu atoms dissolve in the antimony layer rather than the opposite, because the atomic radius of Cu (0.128 nm) is smaller than that of Sb (0.145 nm).

After 200 °C annealing (**Figure 1b**), X-ray diffraction patterns for the Sb/Cu/Si system show that in the inner copper layer the copper reacted with Si and Sb to give reflection lines corresponding to the Cu₃Si and Cu₂Sb phases. Whereas in the outer copper layer system (Cu/Sb/Si) we see the formation of only Cu₂Sb, which leads to a decrease of the (111) and (100)Cu reflection lines' intensities, as indicated in **Figure 2b**). For this latter case, the surface morphology shows that the asperities of the as-deposited samples have served as nucleation sites for the first diffusion steps on the sample surface (**Figure 5b**). According to the RBS signals of the silicon, for both samples at 200 °C annealing, the evolution of an enhanced silicon concentration in the surface is very easily seen. When copper is the first deposited layer onto the silicon, an important quantity of copper ($x(\text{Cu}) = 63\%$) has diffused in the bulk as a consequence of its exceptionally fast diffusivity in the Si substrate. Copper is known as the fastest diffusing element in silicon among all the transition metals, for depths of a few micrometers. For instance, the Cu diffusion coefficient in Si is $1.4 \cdot 10^{-6}$ cm²/s at 400 °C and copper atoms move interstitially in the lattice to form an interstitial solid solution^{15,16}. For the Cu/Sb/Si sample heat treated at 200 °C, (**Figure 3**), the RBS signal of antimony seems to decrease in terms of yield intensity versus the diffusion of silicon and the presence of copper atoms at the surface, to form the mole fractions 39 % Cu, 15 % Sb and 45 % Si mixture layer that is 140 nm thick. This phenomenon is more pronounced during 400 °C annealing, for both samples, where the heights of the copper and antimony signals have drastically decreased due to the transport of the three elements, (**Figures 3 and 4**). Besides, the rear edges for both metallic peaks have extended to lower channel numbers, while the front edge of the silicon signal has extended to the higher channel numbers. This clearly indicates that intermixing has occurred across the different interfaces and in the surface. In both cases, the formed alloy is evaluated from the slopes of the rear edge of the metal layers from the

metallic signals, and it is evident from the RBS spectra of the Sb/Cu/Si sample, that the slopes of the rear edges of the Cu and Sb are more important than in the Cu/Sb/Si sample. These rear edges of the Cu and Sb RBS signals were simulated while taking the 12 % Cu, 5 % Sb, 83 % Si and 26 % Cu, 6 % Sb, 68 % Si for Cu/Sb and Sb/Cu bilayers, respectively. For both systems, the diffusion of all the elements is clearly visible and is also dramatic on the signals' shape with a perturbation of the silicon substrate over about 1300 nm in depth. This Cu-Sb-Si mixture is really formed by the cohabitation of the Cu₃Si silicide and the Cu₂Sb intermetallic compound in the Cu/Sb/Si and only the Cu₂Sb in the Sb/Cu/Si sample, after 400 °C annealing **Figures 1c and 2c**. In this latter case, the disappearance of Cu₃Si silicide to the benefit of the Cu₂Sb phase is foreseeable owing to the fact that the growth and formation of the second compound is energetically more favorable, as will be argued later in this paper. The persistence of the two compounds in the Cu/Sb/Si sample means that the reaction is less pronounced than in the Sb/Cu/Si sample.

Equilibrium phase diagrams of the Sb-Si², Cu-Sb⁴ and Cu-Si¹⁷ binary alloys, which bind the ternary Cu/Sb/Si system, have been well known for a long time. Among the binary systems with an important segregation, Cu-Sb and Cu-Si are particularly interesting for their strong tendency to form ordered compounds. And it is known that Sb/Si is a simple eutectic system, which induces a low limited mutual solubility of Sb and Si and, with no intermediate phase formation because Sb has a high mass and low diffusivity in Si. Indeed, the solid solubility of Sb in Si is very low: the maximum equilibrium solubility of Sb in Si is 0.1 % during an equilibrium processes, whereas that of silicon in Sb is negligible. In the solid state, antimony is partially soluble in copper, with the proportion decreasing with temperature. The solid solubilities of 1.5 % and 3 % Sb in Cu are reached at temperatures of 250 °C and 300 °C, respectively. A maximum solubility of 5.8 % antimony in a copper matrix is reached at a temperature of 645 °C⁸. On the other hand, the copper atoms' diffusion in Si is essentially interstitial with an activation energy of 0.43 eV and a limited solubility of about $1 \cdot 10^{15}$ cm⁻³ at 600 °C^{18,19}. In addition, Cu-Sb and Cu-Si couples are completely miscible with the formation of ordered compounds, such as Cu₂Sb, Cu₃Sb, $\eta\text{Cu}_{11}\text{Sb}_2$, Cu₉Sb₂ and Cu₃Si, Cu₄Si, Cu_{0.83}Si_{0.17}, Cu₅Si, respectively, depending on the composition in the solid solution. However, it is reported as the growth and formation, as the first phases of Cu₂Sb and Cu₃Si in their corresponding binary systems, respectively. This formation of Cu₃Si and Cu₂Sb phases in our case is in conformity with results reported in the literature^{11,20} and is in agreement with both the prediction²¹ and the thermodynamic minimization of the free enthalpy ΔH . From the Cu-Sb system, the reported free energies of formation of the Cu₂Sb, Cu₉Sb₂, $\eta\text{Cu}_{11}\text{Sb}_2$ intermetallic compounds are

(−4.23, −0.54 and −0.29) kJ/mol, respectively²², whereas from the Cu-Si system, those of the Cu₃Si, Cu₄Si, and Cu₅Si silicides are (−4.1, −3.4, and −2.9) kJ/mol, respectively²³. Thermodynamically, the formation of the first stable compound at the interface requires the lowest free enthalpy and is consequently favored. In other words, the Cu₂Sb and Cu₃Si compounds have the lowest heat of formation of their mother compounds and are energetically more favorable. While adopting the same argumentation, if these two compounds are in competition, it is clear that the formation of Cu₂Sb is more favorable than that of Cu₃Si. Indeed, these results confirm that copper atoms are the dominant diffusion species in both compounds, which is in agreement with the rule that stipulates that the majority of atoms in the formed phase should be more mobile than the minority atoms²⁴. The observed compounds' compositions (XRD spectra) in the *equilibrated* mixture (RBS spectra) show that an equilibrium exists between the Cu₃Si and Cu₂Sb phases, starting from 200 °C. This temperature of formation, surprisingly low compared to those leading to the formation of silicides and intermetallic compounds of refractory metals, can be attributed to the high diffusivity of the copper in both the antimony and silicon matrices²⁵.

Figure 5 illustrates the evolution of the surface morphology with temperature, examined by scanning electron microscope, for the Cu/Sb/Si(111) sample. The first micrograph (magnif. 2000-times) of the as-deposited sample shows some asperities, probably due to the preheating of the silicon substrate; this contrasts with the presupposed uniform aspect that the evaporated metallic layer on the cold substrate should have. Starting from 200 °C, as seen in **Figure 5b**, crystallites sprinkled on the samples' surface, begin to take shape according to the same oblique lines. After the 400 °C annealing (**Figure 5c**) we can see large white crystallites with a micrometric dimension of the trapezoidal shape, grown at surface as consequence of the great interdiffusion between the three elements. This is due to the segregation and coalescence of the elements above the sample surface, leading to the growth and formation of Cu₂Sb and Cu₃Si crystallites with heights of over one micrometer, which is equivalent to the simulated thickness of the coalesced metal layers. Particularly for the Cu₃Si silicide, a similar epitaxial growth has already been reported in an octahedral shape on Si(100)²⁶ and different shapes with and without an intermediate metallic layer⁹. In this study, such oriented crystallites' formation was not observed for the Sb/Cu/Si(111) sequence, in spite of enlargement of 4000-times, (**Figure 6**). Indeed, in this case, according to X-ray diffraction diagram, there is no formation of the Cu₃Si phase at 400 °C.

4 CONCLUSION

The XRD, RBS and SEM techniques have been used to investigate atomic interdiffusion in Cu/Sb and Sb/Cu bilayers deposited on monocrystalline silicon with a (111) orientation. In the Cu/Sb/Si sample, in addition to the high texturization of the antimony layer, we already see, before any annealing, the appearance of Cu₂Sb intermetallic compound due to substrate heating at 100 °C. Heat treatments of 200 °C and then 400 °C, reveal a strong Cu-Sb-Si intermixing between the three elements for both systems, resulting in compounds formation. After the 200 °C annealing, in the Cu/Sb/Si system, we see the formation of only Cu₂Sb, and for the Sb/Cu/Si system, there is the formation of the Cu₃Si and Cu₂Sb phases. After 400 °C annealing, the Cu-Sb-Si mixture is formed by the cohabitation of the Cu₃Si silicide and the Cu₂Sb intermetallic compound in Cu/Sb/Si and only Cu₂Sb in the Sb/Cu/Si sample. For both systems, the diffusion of all elements is clearly visible and is also dramatic on the RBS signals' shape with a perturbation of silicon substrate of about 1300 nm in depth. Indeed, at 400 °C, SEM micrographs for Cu/Sb/Si sample show the formation of Cu₂Sb and Cu₃Si crystallites with heights of over one micrometer, which is comparatively equivalent to the simulated thickness of the coalesced metal layers. In this study, such oriented crystallites formation were not observed for the Sb/Cu/Si(111) sequence in spite of the SEM micrograph enlargement of 4000-times.

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