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# The Co/Si(111) interface formation: a temperature dependent reaction

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# Abstract

We have investigated the reaction of Co with the Si(111) surface both at room temperature (RT) and at high temperature (500–650 °C). The temperature evolution of the RT deposited 10 ML film has also been studied. The films, prepared by the different methods, have been structurally characterized by means of primary-beam diffraction modulated electron emission. Auger electron spectroscopy has been used to follow their stoichiometric evolution. For RT deposition the films have been found to have a B-type (180° rotated with respect to the underlying Si(111) surface) cubic structure with a Co content and an interlayer spacing increasing with thickness. After 650 °C annealing, the films are completely reacted and have an unstrained B-type  $CoSi_2$  structure. High temperature (500 °C) deposition of Co leads to the formation of stoichiometric  $CoSi_2$  films. Both annealed and high temperature grown films are found to be Si terminated. © 2002 Elsevier Science B.V. All rights reserved.

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### 1. Introduction

Metal-semiconductor interfaces are widely used in microelectronic devices. In order to control and to improve their technological performances a detailed understanding of their atomic scale properties is necessary. Within this field the importance of ultrathin 3d metal silicide films on silicon is outstanding, due to the very sharp interfaces that can be obtained. In particular Co and Ni silicides have been widely characterized in the last 20 years [1–18]. They both have a  $CaF_2$  structure, with a very small lattice mismatch with respect to silicon: 1.2% and 0.4% respectively.

Literature reports silicon—Co silicide interfaces obtained by several different methods, the main ones being room temperature (RT) deposition followed by annealing at 500–650 °C (the so-called solid-phase epitaxy) and co-evaporation of Si and Co. When the films are deposited at RT, the thickness of the silicide layer is limited by diffusion.

The large amount of basic studies concerning Co silicides on Si(111) focus on different aspects

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of this system: silicide stoichiometry [4,5], bulk structure [6,7], interface structure [6,8], surface termination [6,7,9,10], electronic [5,11] and morphological [12,13] properties. Some results are well established, while others are still under discussion. It is accepted that the stoichiometry of the films obtained by solid phase epitaxy is CoSi<sub>2</sub> and that their structure is the CaF<sub>2</sub> one 180° rotated with respect to the substrate, the so-called B-type structure [1,2]. A lot of work has been dedicated to the study of the termination of the CoSi<sub>2</sub> structure (see for example [3,6,7,9,10] and references therein). Depending on the growth conditions Co-rich or Si-rich bulk terminated surfaces have been found [10]. Silicide films in some cases have been found to be terminated by some segregated silicon after annealing at high temperature, however the orientation and thickness of the segregated phase are still controversial [6,7,9]. Another open question is the presence of a possible A-type silicide phase in the films, claimed in some works [16,19]. Still under discussion is also the question of the stoichiometry of RT deposited films: it has been suggested to be CoSi<sub>2</sub> up to 4 ML and pure Co over 4 ML [17] or to evolve from CoSi<sub>2</sub> at 2–4 ML to CoSi at 4 ML to Co2 Si at 6 ML [4,5]. From a morphological point of view the presence of silicide islands at RT at least at low thickness is suggested in several works [5,12,18].

The differences in some details of the results point out the strong dependence of silicide formation on substrate preparation, reaction temperatures, substrate and Co cleanliness, Co deposition rate and other experimental parameters. Furthermore, the host of techniques used to characterize the system may have different depth and/or chemical sensitivity.

By co-evaporation of Co and Si stoichiometric  $CoSi_2$  films have been prepared; it is noteworthy, however, that recently metastable Co silicide phases with a composition between CoSi and  $CoSi_2$  have also been stabilized [14,15].

We started the study of the Co/Si(111) system in order to understand the temperature dependence of the reaction. With this aim we investigated films prepared with different methods. First we compared the RT reaction with 500 °C (HT) grown films, in terms of structure, film composition and quality. Then we studied the effects of annealing on a 10 ML film grown at RT in order to understand the role of temperature in the reaction kinetics.

We have started the study of the Co–Si(111) system by means of inelastically backscattered electron imaging [19]. It seemed then useful to investigate the open questions using a combined chemical and structural approach. We used primary-beam diffraction modulated electron emission (PDMEE) [20] to obtain chemically resolved information on the structure of the films and Auger electron spectroscopy (AES) to follow their stoichiometric evolution.

The structure of this paper is the following: in Section 2 we present the experimental setup used; in Section 3 we describe PDMEE and AES data, discussing RT growth, the evolution of the films as a function of temperature and 500 °C (HT) growth; in Section 4 we summarize the results and draw the conclusions.

# 2. Experimental setup

The experimental apparatus used is described in detail in Ref. [21]. PDMEE technique is based on the scattering-interference processes undergone by primary electrons in ordered solids, which result in a spatial modulation of the beam intensity in the surface and near surface region. As a consequence, the intensity of secondary electron emission, Auger emission in the present case, shows a strong dependence on the primary-beam incidence angle. The outgoing electron diffraction effects have been shown to be averaged out by the use of an angle integrated detector, such as the CMA used in this work [21]. In the present study a primary-beam energy of 3 keV has been chosen, to be in the forward focussing regime with rather narrow peaks. The surface sensitivity of the technique is determined by the escape depth d of the detected Auger electrons. Co MVV (54 eV, d = 4 Å) and Si LVV (92 eV, d = 4 Å) Auger intensities have been detected to follow the stoichiometric evolution of the films as a function of thickness and of temperature; the choice of low energy, surface sensitive Auger lines allowed to minimize the substrate

contribution. Co LMM (779 eV, d = 10 Å) and Si KLL (1623 eV, d = 20 Å) have been detected for PDMEE characterization. PDMEE intensity angular distributions (IADs) were obtained by changing the polar incidence angle with a step of 1°. The anisotropy of an IAD structure is defined as  $2(I_{\text{max}} - I_{\text{min}})/(I_{\text{max}} + I_{\text{min}})$  where  $I_{\text{max}}(I_{\text{min}})$  is the peak intensity maximum (minimum). To obtain complete PDMEE patterns the signal intensities were detected in an array of polar and azimuthal angles with a uniform density in its projection on the (111) plane; the polar angle range chosen was 0-82° and the azimuthal angle range 0-60°, starting from one of the substrate  $\langle 121 \rangle$  directions. The pattern was then symmetrized to obtain the complete 360° pattern, after having verified its symmetry on a few azimuths. The Co MVV and Si LVV Auger intensities as a function of thickness and of temperature were measured 15° out of sample normal, i.e. out of a forward focussing peak, in order to minimize possible structural dependent effects. The base pressure of the equipment was  $5 \times 10^{-11}$  Torr before deposition and raised as high as  $1 \times 10^{-10}$  Torr during Co evaporation. The substrates used were in situ cleaved Si(111) bars, giving a very sharp  $(2 \times 1)$  LEED pattern. Co was deposited by means of an electron

bombardment evaporator at a rate of approximately 1 Å/min, calibrated by means of a quartz microbalance. In the following we shall refer to Co nominal thickness since the actual film thickness depends on the kind of reaction taking place between the metal and silicon. One nominal Co monolayer (ML) corresponds to 3 ML of silicide if a stoichiometric CoSi<sub>2</sub> film is formed. The samples have been annealed by electron bombardment; 15 min annealing times have shown to be long enough to obtain equilibrium in the considered thermal reactions.

### 3. PDMEE and AES data

#### 3.1. Films grown at room temperature

Fig. 1 shows the intensity ratio of surface sensitive Co MVV to Si LVV Auger lines as a function of Co nominal thickness for RT deposition. To reproduce the Auger intensity ratios, simple models of intensity attenuation have been used [22]. The bulk Auger intensities included have been measured in the same experimental conditions on bulk Co and Si samples. The Co–Si intensity ratio calculated for the growth of an unreacted Co film



Fig. 1. Left panel: Co MVV to Si LVV Auger intensity ratio as a function of Co nominal thickness for RT ( $\circ$ ) growth and HT growth ( $\bullet$ ). The data are compared with attenuation models in the case of non-reacted Co growth (----), CoSi<sub>2</sub> growth (---), CoSi<sub>2</sub> with one ML of Si segregated on top of the growing film ( $\cdots$ ) and CoSi<sub>2-x</sub> growth (—) with x increasing linearly with Co coverage from 0 for clean Si to 1.5 at 10 ML. Right panel: Co MVV to Si LVV Auger intensity ratio as a function of annealing temperature for the RT deposited 10 ML Co film.

increases much more rapidly than the data, as shown in the figure. In the case of the formation of a stoichiometric  $CoSi_2$  layer the considered ratio increases much less than the data and stabilizes at the value expected for bulk  $CoSi_2$ . The data are well reproduced by assuming the formation of a homogeneous reacted layer, with a stoichiometry varying linearly from  $CoSi_2$  to  $CoSi_{0.5}$  as the amount of deposited Co increases. However, this rather crude model only gives an indication of the average Co concentration within the film. We have some evidence, discussed in the following, that RT growing films present a concentration gradient, being Co rich in the topmost layers.

The presence of a reacted Si phase is confirmed by the Si LVV Auger line shape in the spectrum for 10 ML Co deposited at RT, shown in Fig. 2. A comparison with the clean Si case shows a change of line shape in the 75–85 eV energy region, indicating the formation of a chemical environment for Si, after RT deposition of Co, different from the one of clean Si.

Fig. 3 shows the IAD of Co LMM for increasing Co thickness along substrate  $[1\bar{2}1]$  azimuth. The IAD of Si KLL from the clean substrate is also shown along both  $[1\bar{2}1]$  and  $[\bar{1} 2 \bar{1}]$  substrate azimuth. The  $[1 \bar{2} 1]$  substrate Si IAD shows three main forward focussing peaks at 0°, 35° and 70° corresponding to [1 1 1], [1 0 1] and [11] atomic rows, respectively. The Co IADs significantly deviate from the substrate [121] IAD, mainly for the occurrence of a forward focussing peak in the  $50-60^{\circ}$  region. It has to be noted that a forward focussing peak at  $\sim 55^{\circ}$  can be detected in the  $[\bar{1} 2 \bar{1}]$  substrate IAD and originates from the beam alignment along the [010] atomic row. This suggests the formation of a cubic silicide phase, 180° rotated with respect to the silicon substrate (B-type). The position of the [010] peak in the films shows a gradual shift towards lower polar angles with increasing Co nominal thickness. The origin of this shift has been carefully investigated and has been attributed to a superposition of different effects. The first idea which has been taken into consideration is a progressive expansion of the vertical lattice spacing between the scatterers, hence between the crystal layers, with increasing Co nominal thickness. This is understood on the



Fig. 2. Si LVV Auger line shape for (a) clean Si, (b) 10 ML RT deposited Co film, (c) 10 ML HT deposited Co film, (d) 10 ML RT deposited and annealed film. The significant change in line shape in the 75–85 eV kinetic energy region indicates that Co reacts with Si.

basis of a releasing in-plane expansive epitaxial strain of the silicide. Furthermore, an increase in Co content in the silicide can also be responsible for an increase in the film lattice constant, as already observed in the case of Fe [23] and Co [15] metastable CsCl-like silicides. However, the 2 ML IAD shows a shift of the [010] peak of approximately 3° with respect to the relaxed bulk phase. This value is too high to be due to silicon-silicide lattice mismatch only, and can be explained by taking into account also a contribution from electrons backscattered from Si, that often induce an overlayer IAD mimic of the substrate [24,25]. The Si IAD in fact shows a pronounced higher order structure at approximately 62°, which can give a further contribution to the shift of this peak



Fig. 3. Top panel: Co LMM IADs as a function of thickness for RT growth (—) and for 10 ML RT deposited and annealed film (---) along substrate  $[1\bar{2}1]$  azimuth. Si KLL IADs (···) from the clean substrate along  $[1\bar{2}1]$  azimuth and along  $[\bar{1}2\bar{1}]$  azimuth are also shown. The shift of [010] peak to lower polar angles with increasing coverage indicates an increase in the lattice spacing with increasing Co concentration in the reacted phase. Bottom panel: Co LMM [111] peak anisotropy as a function of Co coverage for RT growth (•) and for the 10 ML Co RT deposited and annealed film ( $\blacktriangle$ ). The solid line is a guide for the eye.

towards larger angles. We observed also that a possible A-type silicide phase would not give any



Fig. 4. Co LMM and Si KLL IADs for the 10 ML HT deposited film in comparison with Si KLL IADs from the clean substrate, measured along  $[1\bar{2}1]$  and  $[\bar{1}2\bar{1}]$  substrate azimuths.

contribution to the initial shift of the peak, having a much less pronounced  $62^{\circ}$  structure (see Fig. 4).

The occurrence of a definite structure at about 35° in the 2 and 4 ML Co IADs also deserves some comments. In this angular region an A-type cubic structure presents a forward focussing peak due to the [101] atomic row. We have considered the possibility for this structure to be only due to backscattered electrons. The measured anisotropy of the peaks, however, is too large to be induced by backscattering only; therefore we attribute this peak to the presence of a fraction of silicide with an A-type structure, as already observed by means of backscattered electron imaging [19].

The bottom panel of Fig. 3 shows the [111] peak anisotropy as a function of Co nominal thickness. The maximum in the intensity of a

forward focussing feature is expected to occur when 5 or 6 scatterers are present along the considered atomic chain because of the interplay between focussing and defocussing processes [26]. In the case of RT Co deposition the anisotropy initially increases, then it becomes approximately stable around a value of 20% after 2 ML thickness, corresponding to only two scatterers along the [11] chain. This result is consistent with the initial formation of a crystalline reacted phase and the subsequent formation of a disordered Co layer on top of it, as already found by Pirri et al. [17]. The growth of the disordered Co phase compensates the increase of anisotropy expected as a result of the increase of thickness of the reacted ordered phase.

# 3.2. Films grown at HT

The increase of Co LVV to Si MVV Auger ratio for 500 °C (HT) deposited films as a function of Co thickness, shown in Fig. 1, is considerably slower than in the case of RT grown films. This indicates the formation of a reacted silicide phase. To investigate the evolution of its stoichiometry we have again compared the increase in the considered ratio with the one expected in the case of CoSi<sub>2</sub> formation, finding it slightly slower. The experimental data trend is better reproduced by allowing 1 ML of Si to float on the CoSi<sub>2</sub> growing film. Si-rich terminated silicides have already been observed after annealing at high temperatures [6,7,9,10]. It has to be noticed here that the same trend reported by the data would also be obtained by assuming the formation of islands of CoSi<sub>2</sub> covering a fraction of  $\sim 60\%$  of the substrate surface. However we can exclude this latter hypothesis by the inspection of the Si KLL IADs from the 10 ML film grown at HT shown in Fig. 4. If CoSi<sub>2</sub> grew by island formation, the Si KLL IADs would have a non-negligible contribution from substrate silicon. On the  $[1\bar{2}1]$  substrate azimuth one would then expect also the presence of a [010] peak at 55°, while on the  $[\bar{1} 2 \bar{1}]$  azimuth also the contributions at 35° and 70° due to forward focussing along substrate [1 0 1] and  $[1 \overline{1} 1]$  chains would be present. The Si IADs instead show the same structures as the Co signal, characterizing a B-type

silicide structure, without significant traces of a non-rotated bulk Si phase.

The Si LVV peak of the 10 ML Co HT deposited film is shown in Fig. 2. Its shape is rather different from the one of clean Si in the 75–85 eV energy region, but similar to RT deposited 10 ML thick films.

Fig. 5 shows the evolution of the Co IADs of the films grown at HT, as a function of nominal thickness along the substrate  $[1\bar{2}1]$  azimuth. Already at 2 ML the films show pronounced B-type silicide forward focussing structures evolving with thickness only in anisotropy and not in position. This indicates that the volume strain found in the case of RT growth is released when silicides are formed at HT. The contribution from electrons backscattered from the substrate, often observed at very low thicknesses, is not pronounced in this case because the complete reaction between the metal and silicon produces a film of much higher overall thickness. No traces of A-type phase is observed for HT deposition. The anisotropy of the [111] structure is shown in the bottom panel. In this case it presents a definite increase from 23% at 1 ML Co, to 45% at 6 ML and a slight decrease to 40% at 10 ML, in agreement with the formation of a CoSi<sub>2</sub> film growing in thickness.

The anisotropy of the [111] peak in the 1 ML film is rather high to be only due to backscattering from the substrate [24,25], hence it is an indication not only of the reaction of the film, but also of the presence of at least one Si atom on top of the reacted phase, in agreement with the Si-terminated phase formation proposed on the base of AES data.

To better characterize the symmetry and crystal local order of the 10 ML HT deposited film, we detected a complete PDMEE pattern. The stereographic projection of the Co LMM intensities is shown in Fig. 6, in comparison with the pattern of the Si KLL line of the clean substrate. In the Si(111) pattern [111], [101], [111] and [010] peaks are clearly evident, together with very well defined Kikuchi bands. The film pattern clearly shows the same symmetry rotated by 180° with respect to the clean Si pattern, confirming the formation of a B-type CoSi<sub>2</sub> phase. All of the structures of Fig. 6b, both forward focussing peaks



Fig. 5. Top panel: Co LMM IADs as a function of coverage for HT growth along substrate  $[1\bar{2}1]$  azimuth. Si KLL IADs  $(\cdots)$  from the clean substrate along  $[1\bar{2}1]$  azimuth and along  $[\bar{1}2\bar{1}]$  azimuth are also shown. Bottom panel: Co LMM [111] peak anisotropy as a function of Co thickness for HT growth. The solid line is a guide for the eye.

and higher order structures, are much broader than the ones of the clean substrate. This is due to



Fig. 6. Stereographic projections of Si KLL intensities from clean substrate (a) and of Co LMM intensities from the 10 ML HT grown film (b). The maximum polar angle is  $82^{\circ}$ . On both patterns [1  $\overline{1}$  1], [1 0 1], [1 1 1] and [0 1 0] peaks are marked. The film pattern shows a  $180^{\circ}$  rotated symmetry with respect to the clean Si pattern (B-type CoSi<sub>2</sub> phase).

the smaller probing depth of Co Auger electrons with respect to Si ones and to differences in the structure of the atomic chains in the two materials. Furthermore, the films have certainly a worse long range order than the Si substrate.

# 3.3. Annealing of room temperature deposited films

Fig. 1 also reports the evolution of Co MVV to Si LVV ratio for the 10 ML Co film as a function of temperature from RT to 650 °C. In the RT—300 °C range the ratio shows an abrupt decrease, indicating that such an increase in temperature is sufficient to promote the reaction of Co with Si substrate atoms. For higher temperatures the Co–Si ratio has a much slower decrease and finally reaches a value of ~0.23, close to 0.3 expected for a bulk  $CoSi_2$  phase. We attributed the lower experimental value measured on the film to an already observed [7,10] Si surface enrichment, consequence of sample annealing.

On the basis of the same arguments used in the preceding paragraph concerning HT grown films, we can exclude the presence of silicide islands, and discuss the Si line shape excluding contributions from the substrate. The Si LMM line shape of the 650 °C annealed film, shown in Fig. 2 (curve d), is different from the 20 ML HT Co film one, indicating that the two preparation procedures lead to different surface configurations. The 10 ML HT line shape is more similar to the bulk Si line and in fact can be reproduced by a superposition of curve d with a few Å of Si on top. The analysis of the Auger line shapes indicates that the amount of Si on the topmost layers is higher in the case of HT deposited films. This is possibly due to the fact that for films deposited at HT the reaction starts already at the first stages of growth and reaches its stable configuration, whereas when a 10 ML film is deposited at RT and then annealed the Si diffusion is limited by the presence of the unreacted Co laver.

The Co LMM IAD of the 10 ML Co film annealed at 650 °C along the substrate  $[1\bar{2}1]$  azimuth, shown in Fig. 3, is very similar to the 10 ML HT deposited film of Fig. 5, confirming that the crystal phase of the silicide resulting from the two preparation methods is the same.

Also the anisotropy of the [1 1 1] peak of the RT deposited and annealed 10 ML film, shown in the bottom panel of Fig. 3, shows that the crystal quality of the films increases significantly after annealing, the measured value (40%) being comparable with the one of the 10 ML HT deposited film.

# 4. Conclusions

We have investigated the reaction of Co with  $Si(1 \ 1 \ 1)$  surface in different conditions in the 0–10 ML range by a multi-technique approach.

At RT progressive Co deposition leads to the formation of a cubic (111) phase with increasing Co concentration and increasing lattice parameter,

covered by a non-ordered Co layer growing in thickness. Our data are in agreement with previous findings by Derrien et al. concerning the evolving stoichiometry of the films [4] and with Pirri et al. concerning the presence of a growing layer of unreacted Co on top of the silicide [17]. The orientation of this phase is mainly B-type, but at low thicknesses a fraction of A-type islands are present, in agreement with our previous work [19].

An annealing up to 650 °C of the 10 ML Co film deposited at RT results in the complete reaction of Co with Si forming a bulk stable B-type CoSi<sub>2</sub> phase.

Film deposition at 500 °C leads to the formation of a film with a B-type  $CoSi_2$  structure. Any possible strain due to Si and  $CoSi_2$  lattice mismatch is shown to be completely released with this preparation method.

The effect of temperature has hence been shown to be the same, independent of the deposition mode, in promoting the reaction of Co with Si. A slightly higher temperature, however, is necessary to promote RT deposited films reaction. The stoichiometry and the crystal structure of the silicide phase obtained with the different preparation methods have been found to be the same.

The silicide films obtained with the different procedures have been shown to be Si-rich on their outermost layers, due to surface energy minimization processes. Some differences in the Auger line shape, however, indicate that the amount of surface Si is larger for HT deposited films than for films grown at RT and successively annealed.

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