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# Interaction of cobalt with the Si(1 0 0)2 × 1 surface studied by photoelectron spectroscopy

M.V. Gomoyunova [a], I.I. Pronin [a,*], N.R. Gall [a],
D.V. Vyalikh [b], S.L. Molodtsov [c]

[a] *A.F.Ioffe Physico-Technical Institute, Russian Academy of Sciences, 194021 St. Petersburg, Russian Federation*
[b] *Institut für Festkörperphysik, Technische Universität Dresden, D-01062 Dresden, Germany*
[c] *Institut für Oberflächen- und Mikrostrukturphysik, Technische Universität Dresden, D-01062 Dresden, Germany*

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## Abstract

Cobalt adsorption and condensation on the Si(1 0 0)2 × 1 surface as well as solid-phase reaction of $CoSi_2$ formation have been studied by high-resolution photoelectron spectroscopy with synchrotron radiation. We have measured the Si 2p and valence-band spectra after the Co deposition from a submonolayer coverage to 6 ML thickness and a subsequent annealing to 600 °C. Room temperature Co adsorption results in the loss of the initial surface reconstruction, and the chemisorbed Co atoms appear to be embedded into the upper layer of Si(1 0 0); however, no stable $CoSi_2$ was observed. With further metal deposition, a discontinuous solid solution Co—Si film was formed and the dissolved Si concentration decreased with the distance from the crystal surface. The formation of cobalt disilicide was found to occur in the range of 250–400 °C.

*Keywords:* Silicon; Cobalt; Solid phase epitaxy; Synchrotron radiation photoelectron spectroscopy

## 1. Introduction

The interaction between atomic Co and a Si single crystal surface is of great fundamental and technological interest, so it has been studied extensively for over 20 years [1–19]. This is mostly due to the fact that $CoSi_2$ formed at the interface exhibits a metallic conductivity, a high thermal stability, and, what is of especial importance, it can produce an atomically sharp interface with a Si substrate. The latter allows one to treat this system as a model metal–semiconductor interface. These properties of the system have provided its

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[*] Corresponding author. Tel.: +7 81 2247 9948; fax: +7 81 2247 1013.
*E-mail address:* igor.pronin@mail.ioffe.ru (I.I. Pronin).

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wide use in modern semiconductor electronics. The formation of the $CoSi_2$/Si interface has been thoroughly studied for the Si(1 1 1)7 × 7 surface, while the mechanism of the Co—Si(1 0 0)2 × 1 interaction is less clear, although it is this surface that is most important for silicon technology. The initial interaction which determines the output characteristics of the interface also remain poorly understood. Indeed, the results obtained by different researchers are contradictory in many aspects.

It was shown in Ref. [13] by coaxial impact collision ion scattering spectroscopy that room temperature adsorption of Co does not lead to any atomic surface reconstruction. At least two Co monolayers (MLs) can be adsorbed by an initially reconstructed surface, occupying consecutively three types of adsorption sites without the formation of $CoSi_2$. Recently, the Co adsorption on a Si(1 0 0)2 × 1 surface without its additional reconstruction was also considered in a theoretical study [12], where Co atoms were found to be adsorbed preferably by high coordination sites. One should note, however, that there are some discrepancies between the binding energies of Co on Si(1 0 0) 2 × 1 calculated in [12] and the sequence of the surface site filling found experimentally in [13].

A number of workers claim that adsorbed Co reacts immediately with Si(1 0 0)2 × 1, giving rise to $CoSi_2$ nuclei. As reported in Ref. [14], the reaction involves only Co atoms from the first monolayer, and a metal Co film with diffused Si atoms grows at larger coverage. On the contrary, it was found in Ref. [15] that Co atoms from 2 to 3 MLs were involved in the reaction to produce $CoSi_2$. The authors of Ref. [16] used a surface-extended X-ray absorption fine-structure (SEXAFS) technique to show that the interaction and the silicide growth mechanism depend strongly on the initial state of the surface, or on its preparation and purification procedures. In particular, a loss of the initial reconstruction was observed after the adsorption of the submonolayer Co on a chemically treated and annealed surface.

Thus, the fundamental problems of Co—Si (1 0 0)2 × 1 interaction, such as the changes in the surface structure induced by adsorption, the type of Co adsorption sites, and the production of $CoSi_2$ nuclei at room temperature, are still under discussion requiring a further research effort. The aim of the present work was to investigate the earliest stages of the interaction between Co and Si(1 0 0)2 × 1, both at room and elevated temperatures. The main technique used was core-level photoelectron spectroscopy with synchrotron radiation.

## 2. Experimental

The experiments were carried out with the Russian–German beamline, which was recently created at the Berliner Elektronenspeicherring für Synchrotronstrahlung (BESSY), at a photon energy ($h\nu = 130$ eV) providing a maximum surface sensitivity of the Si 2p core levels. The photoelectron (PE) spectra were recorded in a wide solid angle oriented around the normal to the surface of the sample at room temperature. The overall energy resolution of the analyzer and the monochromator was set to 140 meV FWHM. The substrates used were Si(1 0 0) P-doped wafers with the P concentration of $10^{17}$ at./cm$^3$. The accuracy of the (1 0 0) facet was better than 0.1°. The samples were ex situ prepared by a wet chemical treatment using the method of Shiraki [20]. To clean the surfaces in vacuum the samples were flashed at 1200 °C and cooled slowly with the rate of less than 50 °C/min to provide a well-defined (2 × 1) LEED pattern and the surface uncontaminated with carbon and oxygen.

The chemical composition of the surfaces was monitored using the photoemission spectra. Cobalt was evaporated from a thoroughly degassed source, in which a wire of highly purified deposited material (99.99%) was heated by electron bombardment. The Co flux was $2 \times 10^{13}$ at./cm$^2$s in the standard operation mode. The Co source was calibrated in two ways: by using a standard quartz microbalance and by attenuating the W photoelectron signal of a special tungsten sample pre-exposed to a Co flux. We estimated the error of the Co coverage to be less than 20%. Cobalt was deposited at room temperature in amounts of 0.6–1 ML with Si 2p and the valence-band spectra were measured for each dose (1 ML is considered to be $6.8 \times 10^{14}$ at./cm$^2$, which is equal to the concentration of Si atoms on the (1 0 0) surface). After the total Co amount of 6 ML was deposited, the sample was gradually annealed in short-term cycles with increasing temperature. All the measurements were performed in vacuum higher than $2 \times 10^{-10}$ Torr.

## 3. Results and discussion

### 3.1. Si(1 0 0)2 × 1 photoelectron spectra

A representative Si 2p spectrum from a pure sample obtained by subtraction of the background fitted by the Shirley method is shown in Fig. 1. Before discussing its details, we would like to note that the surface atoms appear to be in a special state, as compared to bulk atoms, due to a break in the translational symmetry on the crystal surface. This produces the surface core-level shifts [21] and gives rise to surface electronic states in the valence band [22]. The core electron spectra of complex reconstructed surfaces normally show several surface modes of atoms with various local geometry. The Si 2p spectrum of the Si(1 0 0)2 × 1 surface, which is characterized by the presence of asymmetric dimers along the ⟨1 1 0⟩ substrate directions [23] (see Fig. 2a), was first found to exhibit four surface modes [24], but later their number was increased to five [25,26]. In these works the modes $S_u$ and $S_d$ were attributed to the upper and down dimer atoms, correspondingly. The energy shift of the $S_u$ component is about −500 meV. As for the component $S_d$ there are two points of view concerning the sign of its energy shift, and this is the major difference between the various studies. In Refs. [24,26], the $S_d$ shift was found to be positive and equal to 60 meV [24] or lying in the range of 30–130 meV [26]. In Ref. [25], the component $S_d$ had a negative shift, −24 meV. The third surface mode $S_1$ was attributed to the atoms of the second monolayer underlying the dimers. The interpretation of the other, less intensive modes ($S_2$ in [24], $S_2'$ and $S_2'$ in [25,26]) is still more ambiguous. For example, the authors of [25] attribute them to the third layer atoms located under and between the dimers. However, the authors of Ref. [26] believe that the component $S_2'$ is due to

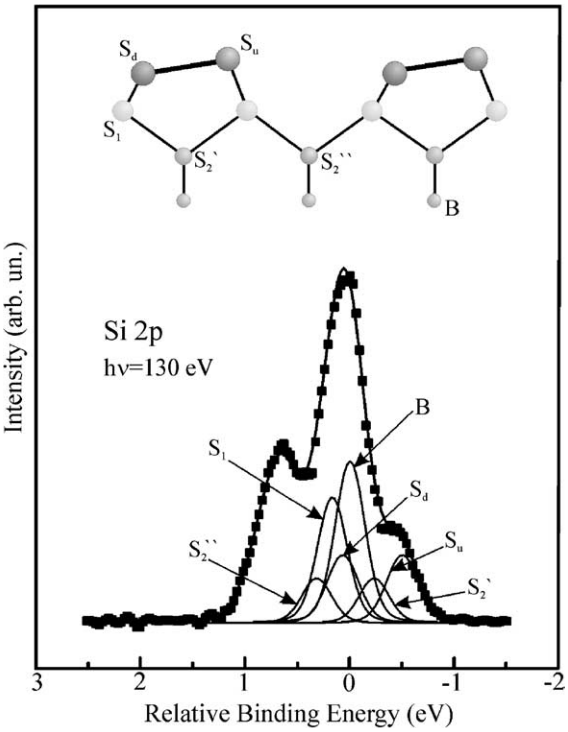

Fig. 1. Si 2p core-level spectrum with the subtracted background recorded from the Si(1 0 0)2 × 1 surface at room temperature with the photon energy of 130 eV. The solid curve through the data points shows the resulting fit. The decomposition of the spectrum into bulk subspectrum B and five surface subspectra is indicated only for the $2p_{3/2}$ component (thin lines). The designations of the subspectra (see text) are shown in the upper part of the figure.

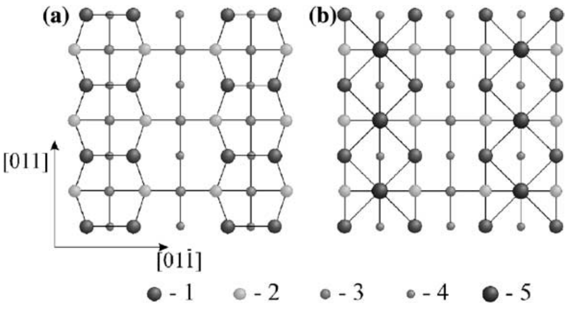

Fig. 2. Schematic atomic structure of the Si(1 0 0)2 × 1 surface (a) and the surface covered with 0.5 ML of Co (b). 1–4—Si atoms of the first through fourth monolayers, respectively; 5—chemisorbed Co atoms.

a half of the third layer and the component $S_2''$ is likely to be associated with vacancy-type defects.

Taking the above results into account, we used five surface components in addition to the bulk one when analyzing the Si 2p spectrum by means of the least square fit procedure. The spectral features were described by the Voigt functions [24] commonly used for this purpose. Such a function is a convolution of a Lorentzian function accounting for the lifetime of a core-level hole and a Gaussian function simulating the phonon broadening and the instrumental resolution. The FWHM of the Lorentzian (70 meV), the branching ratio of the $2p_{3/2}$ and $2p_{1/2}$ components (0.5) and their splitting (608 meV) were taken to be constant. The FWHM of the Gaussian, the relative binding energies of the individual components as well as their intensities were varied during the fitting. The best agreement with the experiment was achieved when the Gaussian width was 300 meV.

These findings are illustrated in Fig. 1. One can see that the spectral analysis yielding one bulk component and five surface ones provides a fairly good fit between the calculations and measurements. The energy shifts of the surface components relative to the bulk one are found to be $-510 \pm 20$, $-240 \pm 20$, $90 \pm 20$, $180 \pm 20$, and $330 \pm 20$ meV. These values are in good agreement with the available data cited above, so we also attribute the spectral features at $-510$ and 90 meV to the $S_u$ and $S_d$ modes, respectively. The large difference in the shifts is associated with the charge transfer from the down dimer atoms to the upper ones [27]. The component with the 180 meV shift is the $S_1$ mode. The last two modes are due to the atoms in the third layer (Fig. 1). According to Ref. [27], the difference in their shifts is due to the variation in the final state contribution for the atoms underlying the dimers and for those located between them.

A valence-band spectrum of the silicon surface is shown in Fig. 3. It is similar to that reported in Ref. [17], being typical for the photon energy used.

### 3.2. Cobalt adsorption on Si(100)2 × 1

The Co adsorption on Si(100)2 × 1 at room temperature changes appreciably the spectra of both valence and Si 2p electrons (Figs. 3 and 4). It is clear from Fig. 4 that the 0.6 ML Co deposition leads to the disappearance of the step in the low energy $2p_{3/2}$ sublevel slope of the Si doublet, which is due to the $S_u$ mode of the upper atoms in asymmetric dimers. This mode is quite isolated from the other spectrum, allowing one to follow its behavior during the adsorption. It is important to note that the upper dimer atoms are localized in the uppermost substrate layer and that these atoms are most sensitive to adsorption. For this reason, our analysis of the spectrum clearly revealed only the $S_u$ mode and the conjugate $S_d$ mode, the others being united into the $B_1$ component. While this approach is somewhat simplified, it is often used to study adsorption processes. For example, it was successfully employed in [17] to study the system concerned.

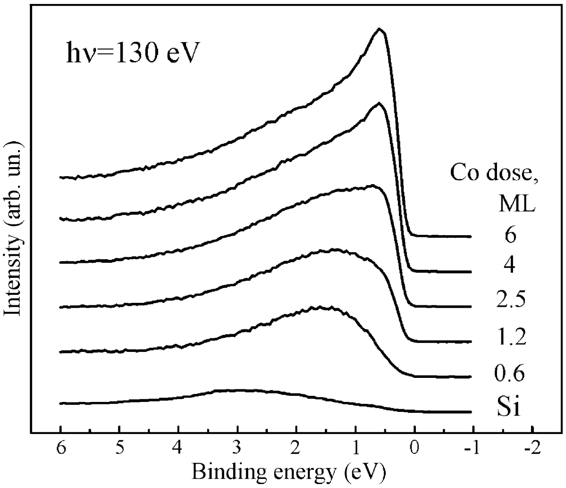

Fig. 3. Valence-band spectra from samples with different amounts of Co deposited at room temperature, $h\nu = 130$ eV. Spectra are normalized to the initial photon flux.

The spectral analysis illustrated in Fig. 4 shows that the 0.6 ML cobalt adsorption does lead to a complete disappearance of the $S_u$ mode. But simultaneously, a new S component arises with the energy shift of $-290$ meV, whose intensity is lower than the initial $S_u$ intensity. The $S_u$ disappearance may result from either the disappearance of dimers or a strong chemical shift arising from the interaction between Si atoms and the adsorbate. It seems unlikely that asymmetric dimers persist on the

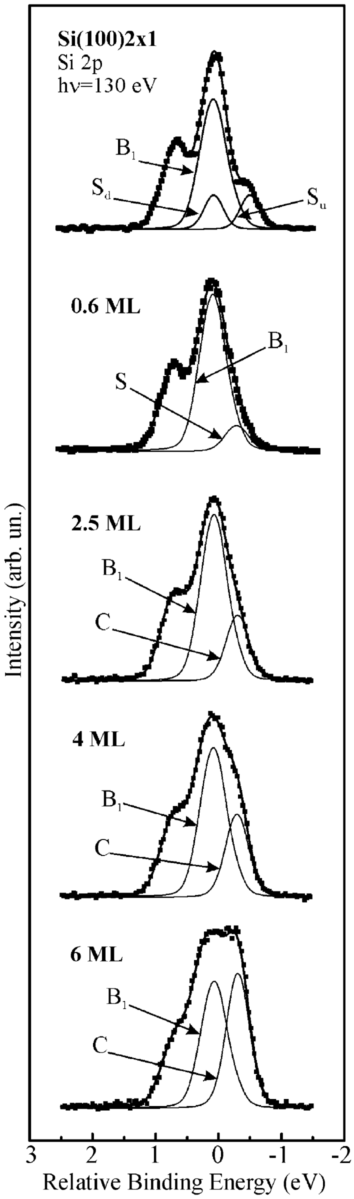

Fig. 4. Si 2p core-level spectra taken at $h\nu = 130$ eV from samples with different amounts of Co deposited at room temperature. Spectra are normalized to the maximum intensity.

surface whereas $S_u$ disappears because of the chemical interaction with the adsorbate, since this mode experiences only a slight energy shift for a chemically active metal like cesium, whose adsorption does not lead to the disappearance of asymmetric dimers [28]. So, the observed changes in the spectrum indicate an atomic rearrangement of the Si surface due to Co adsorption involving, at least, the upper dimer atoms.

Note that an atomic rearrangement has often been registrated in the adsorption of metals on this Si surface. Specifically, I-, II- and III-group metals typically transform asymmetric dimers to symmetric ones. For instance, authors [29] observed a gradual disappearance of the $S_u$ mode and the appearance of a new component between $S_u$ and the bulk mode due to the K adsorption. The intensity of the new feature for a monolayer coverage became twice as high as that of $S_u$, because the latter corresponds to a half of the Si monolayer, whereas the symmetric dimer mode is determined by the whole monolayer. In our case, the S intensity is too low to identify it with a symmetric dimer mode, although S is located between $S_u$ and $B_1$. Therefore, we believe that the Co adsorption on the Si(1 0 0)2 × 1 surface leads to the loss of its dimeric reconstruction. A similar idea was discussed in Ref. [16].

As for the adsorption sites of chemisorbed Co atoms, our findings fit well with the adsorption model suggested in the work [16] using the SEXAFS method to study this system. In agreement with this model, we think that Co atoms are incorporated into the upper monolayer of the unreconstructed crystal surface in between four Si atoms to produce seven interatomic bonds with the substrate. Two Si atoms from the first coordination sphere of the chemisorbed Co are located in the second layer and constitute bridge bonds. The other five Si atoms are located in the second coordination sphere; four atoms reside in the upper monolayer around it, interacting with the Co atom to saturate the surface dangling bonds. The last one is in the third subsurface layer directly under the adatom.

One can find in the literature some additional arguments in favor of this model. One is the conclusion from a theoretical study [12] that the adsorption of Co atoms on the Si(1 0 0)2 × 1 surface energetically favors the formation of a maximum number of interatomic bonds with Si (seven in our case). Then, the model discussed is consistent with the idea that all dangling bonds of surface atoms are preferably saturated by adsorbed particles. When the adatoms are localized at the sites, the saturation becomes possible if the adparticles are grouped into parallel atomic rows oriented in the same way as the dimer rows on a clean Si(1 0 0)2 × 1 surface (Fig. 2b). The (2 × 1) diffraction pattern of Co adsorption obtained in Refs. [16,17] is another evidence to support the above adsorption model. It is also easy to see that the filling of these sites with adatoms should occur if the coverage $\Theta$ is equal to 0.5. The fact that we did not observe the dimer $S_u$ mode at $\Theta = 0.6$ (Fig. 1) close to a saturation coverage also supports this model. Finally, we would like to comment on the negative shift of the new S mode. It was pointed out in Ref. [19] that a negative shift is characteristic of adamantine disilicide, which is produced when Co atoms find themselves in tetrahedral voids of the Si lattice [30]. The adsorption sites we are discussing correspond to the atomic Co positions in the upper layer of just this kind of silicide. One could keep in mind that the equilibrium phase of $CoSi_2$ with the $CaF_2$-type of structure exhibits a positive energy shift [17–19,31]. The fact that we did not observe such a spectral feature allows the conclusion to be made that no nucleation centers of a stable $CoSi_2$-like compound are formed on the Si(1 0 0)2 × 1 surface at room temperature. This point of view is supported by the authors of [17] but it disagrees with the conclusion in [14] that this kind of compound is indeed produced after the deposition of the first Co monolayer. A possible reason for this discrepancy, which can also be found in studies made by other techniques, is the sensitivity of the Co—Si (1 0 0)2 × 1 interaction to the pretreatment of the initial crystal surface [16]. Note also that the sample used in Ref. [14] was cut with the accuracy of 1° only.

One can conclude from the analysis of the valence-band spectra in Fig. 3 that the adsorption of 0.6 ML of Co results in essential spectral changes. There is a new broad peak located at 1.3 eV relative to the Fermi level. As the cross-section of photoexcitation of Co 3d valence electrons is much larger than those of Si 3s and 3p electrons at the photon energy used, the major contribution to the observed maximum is made by the Co 3d electrons. This means that the spectrum in Fig. 3 (0.6 ML) indicates the chemisorbed Co state. It differs strongly from the spectrum of $CoSi_2$ (see Section 3.4) showing once more that no clusters of this stable phase arise during the Co adsorption on Si(1 0 0)2 × 1 at room temperature.

### 3.3. Cobalt condensation on silicon at room temperature

One can see in Fig. 4 that the relative intensity of the $B_1$ component decreases, whereas the −300 meV feature monotonically rises in the whole range of thicknesses, at least, up to 6 ML. It is marked with the letter C in Fig. 4, since its origin seems to be different from that of the S mode for chemisorbed atoms, which we observed in the submonolayer coverage and which should become weaker in polylayer coverages. The energy position of C falls within the range of expected energy shifts for atomic Si in the solid solution Co—Si, which was found in [14,17,18,31] to be −(200–400) meV. The conclusion about the solid solution formation was also supported by Auger electron spectroscopy [14]. We attribute the C component to the atomic Si in the solid solution Co—Si as well. The reason for the coincidence in the energy positions of the C and S features requires a further study. The negative sign of the C mode may result from the enhancement of the interatomic relaxation due to the Co 3d valence electrons.

It seems surprising at first sight that the intensities of the Si bulk component ($B_1$) and the C component should be close to each other after the deposition of 6 ML of Co. Indeed, the bulk silicon is expected to be buried under the film of solid solution. We interpret this effect assuming that Co—Si solid solution films are not continuous, and some of the surface remains to be covered only with chemisorbed Co. However, this area cannot be large considering the relation between the intensities of the components and a low Si concentration in the solid solution. Note that the abnormally high intensity of the bulk Si component was also observed in [17] after the deposition of 5 ML of Co on Si (1 0 0)2 × 1 at room temperature. Moreover, the intensity of this component was even higher than that of the Si component in the Co—Si solid solution. The authors of Ref. [17] explained this effect as being due to pinhole formation. Contrary to the authors of [14], we believe that the Co—Si solid solution is not uniform in depth, the Si concentration being decreased with approaching the surface and with growth of the film thickness. This idea is supported by the absence of surface components of this phase in the spectra. Indeed, if the solid solution were uniform in depth, the spectrum for 6 ML Co (Fig. 4) would have a surface mode in addition to the intensive bulk C mode. Its absence indicates that there are no Si atoms at the surface and in the near-surface layer, or their concentration is very low. The spectra of valence electrons also supports the Co—Si solid solution nonuniformity (see below).

The valence-band spectra in Fig. 3 show that the deposition of 0.6–6 ML of Co results in a gradual growth of the photoelectron intensity over the whole energy range, while the energy distribution maximum shifts to lower BE's, i.e. to the Fermi level. When going from 0.6 to 1.2 ML of Co coverage, the main peak becomes broader and slopes more and steeper toward the Fermi energy. With further Co deposition, a new peak arises with the energy a few tenths of an electron-volt lower than the Fermi energy. Its position is very close to that of Co [31] and is an evidence for a gradual conversion of the solid–solution Co—Si film to a pristine cobalt film. This result shows that a film formed due to the Co deposition at room temperature is heterogeneous and the Si concentration decreases toward the surface.

### 3.4. Cobalt disilicide formation

At the second stage of the solid-phase epitaxy, the samples with 6 ML Co were annealed by a stepwise (each time for ~2 min) heating. After each anneal, the samples were cooled to room temperature and the photoelectron spectra were taken. The spectral features indicating a solid-phase reaction were first found at ~250 °C, becoming more pronounced with increasing temperature. The Si 2p spectra are presented in Fig. 5. Their changes are opposite to those previously obtained after the Co deposition. The broad flat feature becomes gradually double-humped, and a step arises in its right slope. Eventually it resembles the pure substrate spectrum.

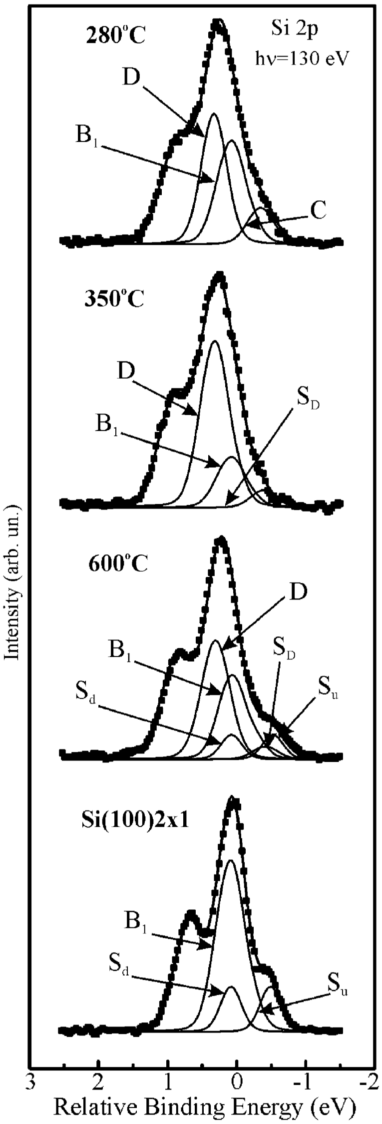

Fig. 5. Si 2p core-level spectra taken after the annealing of a sample covered with 6 ML of Co, $h\nu = 130$ eV.

The energy distribution curves were fitted with the subspectra presented in Fig. 5. One can notice two main trends in the behavior of the Si 2p spectra of the samples annealed at 280 °C, as compared with "as deposited" sample: (i) the intensities of the fitting components $B_1$ and C are reduced, while the signal from the Co—Si solid solution is decreased much greater, and (ii) a new component D appears with a positive energy shift of 300 meV.

Similar positive shifts of the Si 2p core-level features were observed for cobalt disilicide with a stable $CaF_2$-type structure [14,17,18,31]. A positive shift of 300–350 meV relative to the energy position in the stable phase was found for metastable $CoSi_2$ with a CsCl-like structure [32,33]. From these data, we attribute the component D to the Si bulk contribution of cobalt disilicide with the $CaF_2$ structure, which is usually grown in a solid-phase reaction. Note that its positive shift may be due to the charge transfer from the Si to Co sites, an effect known for $CoSi_2$ with the $CaF_2$ structure [30]. Relaxation phenomena which decrease the BE of electrons in photoemission spectra may result in a positive shift, if they become weaker in $CoSi_2$ than in Si. It is known, however, that the main contribution to the relaxation shift due to valence electrons is made by interatomic relaxation, which is largest for metals. Since $CoSi_2$ is a metal (silicon is a semiconductor), it is unlikely that the final state contribution is smaller for $CoSi_2$ than for silicon.

The temperature increase to 350 °C suppresses the pristine silicon component $B_1$ and leads to further growth of $CoSi_2$ component D which becomes dominant in the spectra. The component C, which shows a negative shift, has a lower intensity and varies its energy position to some extent that may be interpreted as being due to a different origin. Its energy shift relative to bulk $CoSi_2$ component D is nearly −600 meV, which agrees well with the value for the surface component $S_D$ in a $CoSi_2(100)$ single crystal [34]. The above arguments indicate the disappearance of the solid–solution phase and the termination of the solid-phase reaction to produce an epitaxial $CoSi_2(100)$ film. Annealing the sample to 600 °C modifies the spectrum again: the intensities of $CoSi_2$ components D and $S_D$ are reduced, while the signal related to the substrate component $B_1$ is increased. Simultaneously the surface component $S_u$ appears indicating the production of pinholes, areas of the exposed reconstructed $Si(100)2\times1$ surface. We suppose that pinholes result from $CoSi_2$ clustering.

The above results are supported by the valence-band spectra measured in the same experimental conditions as the Si 2p spectra (Fig. 6). Here, the drastic spectral changes indicating a solid-phase reaction were also observed after annealing at temperatures higher than ~250 °C. Comparing the data in Fig. 6, one can see that the strong peak at 0.4 eV BE resulting from the metal Co phase becomes nearly extinct after annealing. A trace of this peak shows itself as a shoulder in the steep right slope.

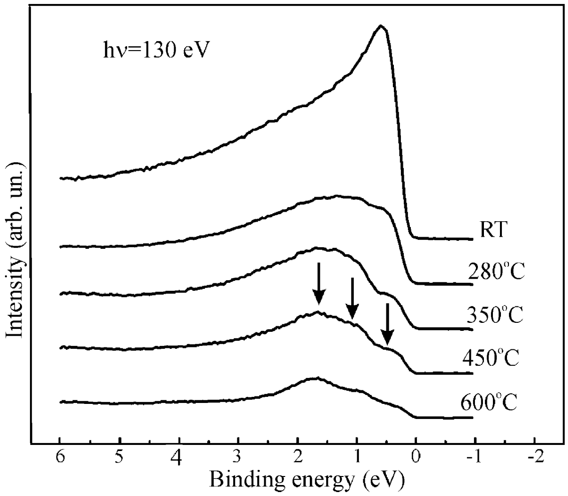

Fig. 6. Valence-band spectra recorded after the annealing of a sample covered with 6 ML of Co, $h\nu = 130$ eV.

There are two maxima of the electronic density of states of $CoSi_2$ with the $CaF_2$ structure [3,35]. The first one is due to Co 3d antibonding states and is located at ~1.7 eV below the Fermi level; it has a fine structure at a lower binding energy. The second maximum at −3.7 eV is attributed to the bonding states of mixed Si 3p and Co 3d character. The maximum with the fine structure was indeed observed in the spectra taken after annealing at $T \geqslant 350$ °C (Fig. 6), when the solid-phase reaction between Co and Si was found to be accomplished. The presence of these features at −1.7, −1.1 and −0.55 eV (indicated by the arrows in the figure) is an additional support for $CoSi_2$ formation. The reason for the absence of the second maximum in our spectra is the use of high-energy photons. This peak was observed only at rather low photon energies, between 20 and 40 eV [3,14,35], while it was not monitored at 115 eV [17], i.e. at a photon energy close to that used in the present study.

The crystal annealing to 600 °C reduces the valence band spectral intensity while its shape is practically preserved unchanged. This is also indicative of a partial exposure of the crystal surface due to the annealing. Indeed, the photoexcitation cross-sections for Si s and p electrons are much smaller than those for Co 3d electrons. Therefore, the contribution made by the exposed crystal surface appears to be small, and the valence band spectrum is determined by the $CoSi_2$ remaining on the surface, which accounts for the preserved spectral shape.

## 4. Summary

The use of core-level and valence-band photoelectron spectroscopy has enabled us to suggest a self-consistent physical mechanism of the interaction between Co and Si(1 0 0)2 × 1. The adsorption of Co submonolayers at room temperature leads to the disappearance of the initial surface reconstruction. We suppose that a similar effect may occur in other adsorption systems with a multivalent adsorbate capable to saturate the substrate dangling bonds. The deposition of larger Co coverages in the range of 1–6 MLs produces a discontinuous metallic film with dissolved Si atoms. No silicide phases have been observed at room temperature. Annealing of the films at temperatures between 250 and 350 °C gives rise to cobalt disilicide in agreement with [14,17]. At higher temperatures (~600 °C), the area covered with $CoSi_2$ becomes smaller, showing exposed surface regions.

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