

Ge-Ag interface at room temperature: An energy-dependent photoemission study

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The electronic structure of the Ge(111)-Ag interface has been studied using synchrotron radiation photoemission of the valence band at $h\nu=40.8, 80,$ and 130 eV (Cooper minimum for Ag $4d$ electrons) and of the Ge $3d$ and Ag $3d$ core lines at $h\nu=120$ and 450 eV, respectively. By using extensively the Cooper-minimum techniques it was clearly demonstrated that in the first monolayers (2–3) a strong chemical interaction takes place between Ge and Ag at room temperature with incorporation of Ag into the Ge surface lattice. Further Ag growth on top of the intermixed region follows the Stransky-Krostanov mechanism with almost pure Ag island formation. The present data constitute the first evidence of intermixing at the Ge-Ag interface.

I. INTRODUCTION

The elemental semiconductor—noble-metal interface is a subject of great interest and a growing number of systematic studies have recently appeared in the literature.^{1–7} One of the reasons for this interest is that no stable bulk compounds (i.e., stable silicides or germanides) are known for these systems, and contrary to the case of the reactive “ d ” metal-semiconductor systems,⁸ the formation of an abrupt interface could be expected when the noble metal is deposited on top of a semiconductor surface, as the traditional model for the Schottky barrier assumes.¹⁹ In the cases of Si + Au and Si + Cu,^{1,2,7} however, it is clear that considerable intermixing takes place and the interface has a nonzero width that has been interpreted as an alloy phase whose extension is possibly connected with the heat of formation of the Si-metal bond.¹⁰ The Si + Ag interface has been described as atomically abrupt in a photoemission report by McKinley *et al.*,³ whereas no spectroscopic information is available in the literature for Ge + Ag. The aim of the present paper is to present an extensive set of photoemission results on Ge + Ag. We show that on the scale of 1–3 monolayers a strong chemical reaction takes place with a severe intermixing at the interface. This new result has its counterpart in recent experimental observations for Si-Ag which are being published elsewhere^{5,6} and implies a change in the generally accepted picture for elemental semiconductor-Ag interfaces.

The present results come from a photoemission investigation with synchrotron radiation (SR) in

which considerable experimental efforts have been devoted to the growth of submonolayer films in order to follow, step by step, the formation of the first Ag monolayer. The most relevant changes in the interface chemistry are expected in this coverage interval.

Taking advantage of the unique tunability of synchrotron radiation (SR), we have exploited energy dependent photoemission, obtaining valence-band energy distribution curves (EDC's) at the Cooper minimum for the Ag $4d$ orbitals ($h\nu=130$ eV), where the d cross section for photoionization is strongly reduced and the substrate valence density of states can be revealed, and at two photon energies ($h\nu=40.8$ and 80 eV) where the metal d contribution is the dominant emission. This technique has been described earlier by Rossi *et al.*¹¹ and successfully applied to the reactive semiconductor—transition-metal interfaces and is of particular value in this case where the presence of a rather small amount of intermixing requires a high sensitivity to the details of the different contributions to the valence-band emission at the interface. The Cooper-minimum technique is complemented by an analysis based on the electron escape depth which has already been used in the Si—noble-metal cases (Cu and Au).^{1,4}

The organization of the paper is as follows: Details on the experiment are given in Sec. II and a full review of the experimental results and their internal organization is given in Sec. III. The discussion of Sec. IV is divided into three coverage intervals showing different stages in the interface growth. The summary is given in the concluding paragraph.

II. EXPERIMENTAL

Medium doped, *n*-type, germanium single crystals were cleaved *in situ* along the (111) plane at an operative pressure of 5×10^{-11} Torr. The high reproducibility of the clean valence-band EDC provides us with a good assumption that no relevant differences are present among the various Ge(111) surfaces, although we had no independent technique to check the cleavage quality.

The thin overlayers of Ag were prepared by evaporation from a bead on a tungsten wire, and the thickness was obtained using two quartz crystal monitors. The first crystal monitor was very close to the evaporation source, behind the evaporator shield; the second one was on the sample carousel and could be put in the sample position. The first crystal monitor was carefully calibrated against the second one in order to have the exact geometric correction factor. The desired coverages were typically obtained during a few seconds of evaporation by operating a shutter in front of the source and continuously monitoring the evaporation rate by means of the first thickness monitor. With this procedure we estimated a maximum error of 20% for the very low submonolayer coverages, and 5% or less for the higher coverages. The photoemission spectra obtained at submonolayer coverages on different cleaves had high reproducibility and consistent phenomenological trends for the estimated coverages. The evaporation source was positioned more than 10 cm away from the sample surface to avoid heating of the substrate; also the heating problem was reduced because of the low power required for Ag evaporations. The maximum pressure during the evaporation was 1×10^{-10} Torr, with all the low coverages done at 9×10^{-11} Torr. The photoelectron spectra were taken in the retarding mode with a double pass cylindrical mirror analyzer having the axis normal to the sample surface. The radiation source was the two 4° beam lines at the Stanford Synchrotron Radiation Laboratory¹² (SSRL) equipped with a "grasshopper" monochromator¹³ providing an intense *p* polarized monochromatic beam in the range 35–500 eV. The spectra at $h\nu=40.8$ eV were taken on the "new" 4° beam line at SSRL covering the photon energy range 35–500; all the other measurements were taken on the "old" 4° line covering the region 65–900 eV. The light was impinging on the sample surface at grazing incidence (15°). Very high counting rates, typically on the order of 10^4 count/s, were obtained at all the energies used, giv-

ing high statistical quality to the data. More details on the experimental apparatus were published by Pianetta *et al.*¹⁴

The kinetic energies of all the measurements were in the range 35–120 eV which gives the maximum surface sensitivity [escape depth 5–6 Å (Ref. 15)]. All the data are in this way directly comparable, referring to the same sampled region of the interface.

III. RESULTS

This section summarizes the experimental results discussed in Sec. IV. To make the discussion easier, we present relevant experimental trends in representative plots. Figures 1 and 2 give the coverage dependence of the valence band EDC's at $h\nu=80$ eV and at the Cooper minimum ($h\nu=130$ eV). Figure 3 gives the EDC's at $h\nu=40.8$ eV for selected low coverages and for the high coverages.

In the EDC's at $h\nu=40.8$ eV (Fig. 3) and $h\nu=80$ eV (Fig. 1), the Ag $4d$ contribution is dominant over the Ge *sp* contribution due to the relatively much higher cross section for the photoionization of the $4d$ orbitals. This makes it possible to easily detect the role of Ag in the determination of the valence density of states even when at very low [0.03 monolayers (ML)] Ag coverages. At $h\nu=130$ eV (Fig. 2), the $4d$ cross section¹⁶ has a deep minimum (Cooper minimum) so that the *d* contribution seen in the EDC is of the same order of magnitude as that due to the *sp* band of the Ge substrate. In this case the modification of the substrate electronic structure can be clearly followed. The physical information coming from Ge $3d$ and Ag $3d$ core lines are summarized as a function of the coverage in Figs. 4 and 5 (intensities versus coverage) and in Fig. 6 (binding energy versus coverage).

IV. DISCUSSION

In this section we discuss the results by making a division into three coverage regions which correspond to three different stages in the interface growth: the very low submonolayer coverages [Sec. IV A ($\theta \leq 0.3$ ML)], the completion of the first monolayer [Sec. IV B ($0.3 \text{ ML} < \theta < 1.08$ ML)], and the high coverages interval extending up to 10 monolayers (Sec. IV C). This division into three regions is merely to provide a phenomenological frame for the discussion, since different dom-

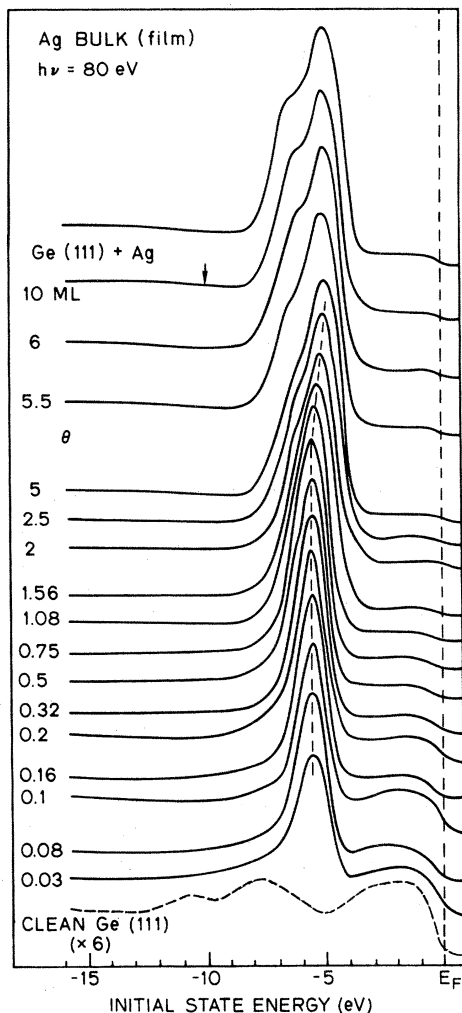


FIG. 1. Angle-integrated photoelectron energy distribution curves (EDC's) at $h\nu=80$ eV for Ge(111) + Ag at increasing coverages θ at room temperature, and for bulk Ag (film). The Ag coverages θ quoted in the figure are in monolayer units.

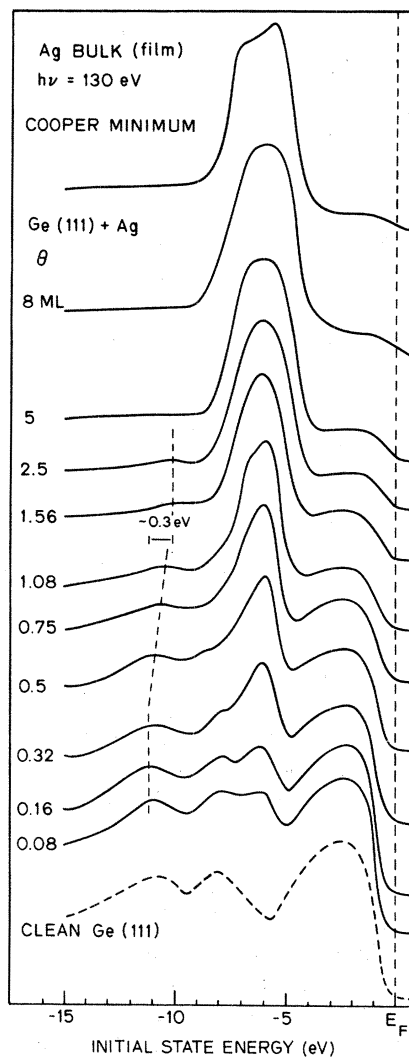


FIG. 2. Angle-integrated EDC's for Ge(111) + Ag and for bulk Ag (film) at the Cooper minimum for the Ag 4d photoionization cross section ($h\nu=130$ eV). The coverages θ are in monolayer units.

inant features are seen in these regions. However, there is no fundamental reason to separate these intervals since the properties of the interface are continuously changing throughout the whole coverage interval investigated.

A. Low-coverage region ($\theta \leq 0.3$ ML)

At the very lowest coverages the Ag 4d signal is composed of a sharp peak, almost symmetrical in shape, at 6 eV below the Fermi level, i.e., at higher binding energies than in the Ag bulk. This situa-

tion is common to all the semiconductors—noble-metal interfaces and is usually explained as a situation where the noble-metal atoms are almost isolated at the substrate-vacuum interface and the photoemission peak is more atomiclike than bandlike.^{1,2,5} This also has consequences on the core line binding energies as will be discussed later in Sec. IV D. The 4d peak grows in intensity without shifts in energy, within the accuracy of the experiment, up to 0.3 ML as seen in the $h\nu=80$ eV EDC's of Fig. 1. The Ag 3d core lines grow in intensity linearly with the increase in coverage; the

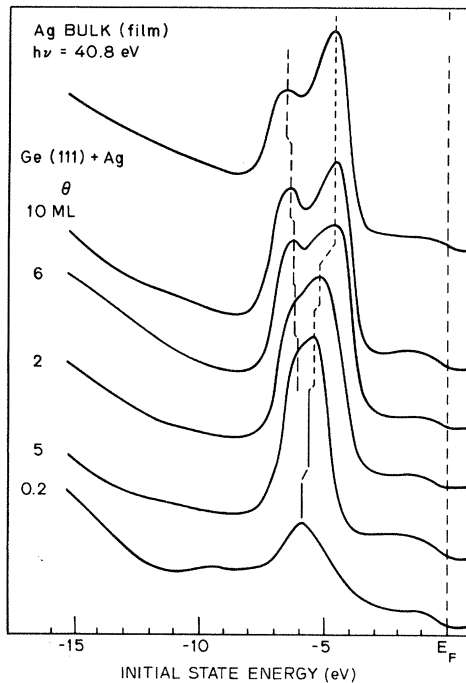


FIG. 3. Angle-integrated EDC's for Ge(111) + Ag and for pure Ag (film) at $h\nu = 40.8$ eV. Due to matrix elements effects the evolution of the Ag $4d$ peak shape is much more evident than at the other energies used.

decrease of the Ge $3d$ intensity is also linear.

At the Cooper minimum (Fig. 2) it is possible to see the beginning of the Ag signal already at $\theta = 0.08$ with no appreciable modifications of the Ge valence band at these coverages. Nothing can be said about the Ge surface states in this range since the photon energies used give very little sensitivity to those states.

The situation in this low-coverage region is quite

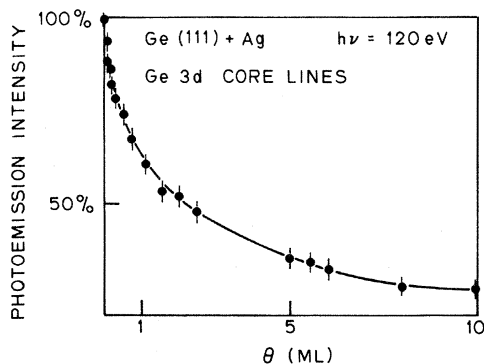


FIG. 4. Intensity profile of Ge $3d$ core line photoemission as a function of the coverage. The intensities are normalized to the clean Ge(111) signal.

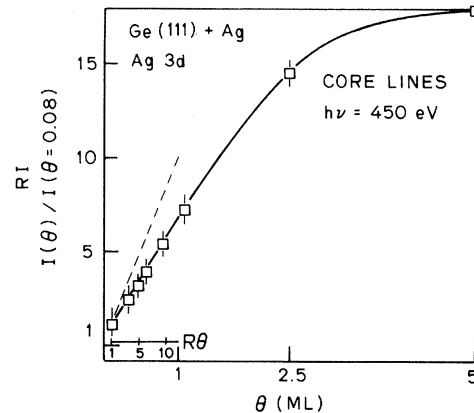


FIG. 5. Intensity profile of Ag $3d$ core line photoemission as a function of the coverage. The reduced intensity (RI) [$I(\theta)/I(\theta=0.08)$] is plotted versus the reduced coverage $R\theta$ ($\theta/0.08$ ML) for the submonolayer range. In the submonolayer range, the dashed line shows the expected trend for RI vs $R\theta$ in the case of the formation of a uniform monolayer.

different from that of the highly reactive interfaces like Si-Pt where dramatic changes in the substrate emission are already seen for 0.1 ML of metal, probably due to a surface disruption mechanism.¹⁷ This confirms the known fact that the interaction between Ag and Si is definitely weaker than in the higher reactive cases.

B. The first monolayer formation ($0.3 < \theta < 1$ ML)

The second region is more interesting because it is possible to see the beginning of a gradual shift

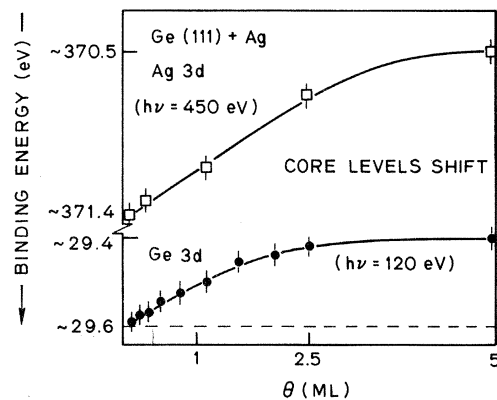


FIG. 6. Binding energy shifts for Ge $3d$ and Ag $3d$ core lines as a function of the coverage θ . The curves the best fits of the experimental data.

of the Ag $4d$ band towards lower binding energies, i.e., towards a more bandlike situation (see Fig. 1). But the key point in the understanding of the interface growth is seen in the spectra at $h\nu=130$ eV of Fig. 3, where the shift towards lower binding energies of the deepest structure (10.5 eV below E_F), mostly composed of Ge s states, takes place and is almost completed within this interval. In this respect it should be noted that the general trend of this structure is a shift towards lower binding energies whenever the sp^3 configuration, which is known to give the maximum bandwidth,¹⁸ is modified. Thus a shift of this structure towards lower binding energy has been observed in all the elemental semiconductor— d -metal reactive interfaces where compounds are formed.^{17,19,20} In the present case the shift is thus the first direct spectroscopic evidence of a chemical interaction taking place between Ag and Ge at the interface with the formation of new hybrid orbitals between the two atomic species so that the sp^3 configuration is modified.

The total shift in this case is ~ 0.3 eV, which is less than the analogous shift observed for the Si—transition-metal “reactive” interfaces, where shifts of 1 eV have been detected, e.g., for Si-Pd (Refs. 11 and 19) and Si-Pt.¹⁷ The explanation of this difference can probably be found in the fact that the Ge-Ge covalent bond is weaker than the Si-Si covalent bond and the Ge atomic arrangement is probably less distorted when interacting with Ag than it is in the very reactive cases.²⁰ We emphasize that the present paper gives the first information on the distortion of the sp^3 configuration in Ge due to the interaction with a metal, and the first set of Cooper-minimum data on a Ge— d -metal interface. Thus there are no other data available for comparison.

Photoemission data cannot give structural information, but nevertheless it can be very useful to address the question of the average depth distribution of the Ag atoms in the interface. This can be assessed on the basis of the core lines intensities versus coverage. Plots of the Ag $3d$ core line emission intensities after normalization to the 0.08 ML coverage, and of the Ge $3d$ core line intensities are given in Figs. 4 and 5, respectively. The values of the Ag $3d$ curve up to one monolayer are, in the manner the data are plotted, independent of the absolute calibration of the thickness monitor, and the information is very reliable. One can see that the increase of the Ag $3d$ signal intensity is smaller at the submonolayer coverages than should be expect-

ed if a uniform layer was formed. In other words, when the coverage is doubled within these submonolayer coverages, the photoemission intensity of Ag is not doubled; typically 30% of the signal is lost. This provides evidence that Ag must penetrate into the sample, and at least part of it is beneath the first layer.

Our results are qualitatively similar to those recently found by means of surface potential and ion scattering spectroscopy (ISS) (Ref. 21) measurements where the reconstructed ($\sqrt{3}\times\sqrt{3}$) Ag layer was concluded to lie beneath a pure Si surface layer after heating to 400 °C.

It is important to note that this argument is fairly unambiguous for small coverages. An alternative assumption of early Ag agglomeration at $\theta < 1$ could hardly explain the data. The formation of Ag islands of such large dimensions that a considerable fraction of the signal is lost due to escape depth effects²² cannot be favored.

Thus a clear result of the present research is that a strong chemical interaction takes place between Ag and Ge, and that the Ge surface is disrupted with the incorporation of Ag in the topmost layer of the substrate. How deep Ag goes into the surface is difficult to assess, but on the basis of escape depth considerations it is possible to conclude that almost all Ag is within the first 2–3 layers of material.

C. The high-coverage interval ($1 < \theta < 10$)

In the high-coverage interval the three most relevant observations are the following:

(i) The valence spectra evolve towards the EDC's typical of pure Ag which is basically reached between 6 and 10 ML (see Figs. 1–3).

(ii) The Ge $3d$ emission intensity, although decreasing, remains considerably high, i.e., about 30% of the signal typical for pure Ge is still present at the high coverages.

(iii) The binding energy shift of the Ge $3d$ core levels reaches a plateau above 2.5 ML.

These experimental results give indications about the growth mechanism of Ag on top of the first intermixed Ge-Ag overlayer. It is also apparent that structural investigation like low-energy electron diffraction (LEED) IV profiles, ISS, and surface extended x-ray absorption fine structure (EXAFS) would be important complements to the present results.

The fact that an almost pure Ag valence band

EDC is obtained for coverages larger than 6 ML is not compatible with the presence of a substantial quantity of Ge (which is obviously present as seen from the intense $3d$ core signal) in an intermixed phase. In fact the valence band for a thick intermixed layer should be dramatically different from that of the pure metal as has been shown, for example, in the case of Si-Au intermixed phases.^{1,7} Probably a minor fraction of Ge is still present in the overlayer, as will be discussed later, but the presence of the strong Ge core signal intensity must be thought of as originating from a spatially distinguished region of the surface with respect to that originating the Ag signal.

The presence of an intense Ge $3d$ signal is compatible with two models: the island growth or Stransky-Krostanov (SK) mechanism and the layer-by-layer (Van de Merwe) growth of Ag, with some Ge dissolved, and with Ge segregated to the topmost surface layer. Surface segregation from the substrate has been found in some related systems, Si-Au (Ref. 1) and Si-Cu (Ref. 2) at room temperature, and Si-Pd after annealing.²³

Our photoemission data cannot be conclusive on this point, but some clear indications are present in the valence-band and core level data. In the case of a Ge surface on top of a Ag-rich layer some spectroscopic evidence of Ge-Ag bonds should be expected. This situation in fact should be similar to that present on the bulk side of the interface when Ag atoms are in contact with the Ge substrate, which has been studied at the low Ag coverages. The presence of strong bonds between a metal-rich phase and a segregated top layer of semiconductor has clearly been found for the annealed Pd-Si interface.²² From the present data no indications of such a situation are evident so that the alternative model of Ag island formation appears likely. We can explain the photoemission results as a sum of the signal coming from Ag-rich islands and from the uncovered part of the Ge-Ag mixed phase, 2–3 layers thick.

In this case the decrease of peak C in the Cooper-minimum spectra of Fig. 2 at increasing coverages can be explained with the gradual decrease of the uncovered area, as more Ag is deposited, and with the smaller emission with respect to the strong Ag signal coming from the islands.

Our data suggest a model of a complex interface with a narrow mixed interface, two or three layers thick, and almost pure Ag islands grown on top of it. This model could also explain the constant value of the Ge binding energy at the high cover-

ages. The Ge atoms are either in the "stable" intermixed region or very diluted in the Ag islands and are not very sensitive to further Ag enrichment. The presence of two different species of Ge is given further support by a slight broadening (~ 0.1 eV) observed at large θ for the Ge $3d$ core lines.

Further information is obtained analyzing the spectra taken at $h\nu=40.8$ eV (Fig. 3) which are very useful in this high-coverage range. At this photon energy the $4d$ splitting into two peaks is much more evident than at $h\nu=80$ eV where only a shoulder on the higher binding energy side of the peak appears. The splitting has a crystal-field contribution that starts to build up only at relatively high coverages ($\theta=6$) and the bulklike situation is completely reached with 10 ML. This provides evidence that the dimensions of the Ag islands are large enough around 10 ML to produce Ag clusters with a typical bulklike photoemission signal, and indirectly confirms that some Ge is present in the early stage of the Ag island growth, becoming eventually a negligible impurity at the high coverages. It would be premature at the present time to push these arguments further and make a detailed model because of the arbitrariness of several parameters and the lack of more direct structural investigation on this interface.

Thus we will limit ourselves to state that the spectra suggest a qualitative picture for the Ag growth which is basically in agreement with the SK mechanism as proposed by Venables *et al.* for Ag-Si,²⁴ provided that the actual substrate on which the island growth takes place is not a single Ag chemisorbed layer but a mixed Ge-Ag phase two or three layers thick.

Our picture for the Ge-Ag interface contrasts the one proposed for Si-Ag by McKinley *et al.*³ In fact, these two silver-elemental semiconductor systems should be very similar, and the model of an atomically abrupt junction is oversimplified as the present results and recent reports on Si-Ag demonstrate.^{5,6,21} In this connection we also note that Venables *et al.*²⁴ have shown that at room temperature, the Stransky-Krostanov and Van der Merwe growths become very similar due to the presence of almost two-dimensional islands so that a clear distinction between the two processes becomes highly speculative, although SK growth was shown to be favored in the Si-Ag case at high coverages.²⁴

In summary, our results show that such phenomenology is also applied to Ge-Ag. We are also able to add new information. In particular we demonstrated that the bond between Ag and Ge

has characteristics similar to those of the bonds between Si and other *d* metals usually referred as more "reactive."⁵ We further showed that the intermediate layer in the SK is a strongly intermixed Ge-Ag phase; and that in the beginning of the nucleation of islands some Ge is still present dissolved with Ag. The presence of this alloyed Ge-Ag phase is probably responsible for the quick convergence towards almost pure Ag overgrowth in the sense that it acts as a membrane against the interdiffusion. The hindrance of an extended intermixing due to this membrane effect is consistent with recent results by Cros *et al.*²⁵ They prepared a Si-Au-Ag interface with a monolayer of Au deposited onto a Si(111) substrate before growing the Ag film, and observed a strong intermixing of Si and Ag. The interpretation is that the weakening of the Si-Si covalent bond caused by the Si-Au mixed phase makes the Ag-Si interdiffusion easier. Tentatively, transferring this argument to the Ge-Ag case we could say that in absence of *ad hoc* weakening processes the Ag-Ge reaction and intermixing is very quickly saturated. Further studies on the temperature dependence of the extension of this phase could give more insight on the kinetic of this interface reaction.

V. CONCLUSIONS

A systematic spectroscopic investigation of the Ge-Ag interface at room temperature, making use

of the Cooper minimum method and at high surface sensitivity, shows that intermixing takes place at the Ge-Ag interface with the formation of bonds between Ge and Ag atoms in a region extending for 2–3 layers of material. On top of this intermixed region the Ag metal grows in islands with a small amount of Ge dissolved in it. Thus the Ge-Ag interface cannot be considered as a sharp interface and the interpretation of its electronic properties [in particular the very low Schottky barrier height ~ 0.35 eV (Ref. 26)] cannot rely upon the traditional abrupt junction models⁹ but requires the consideration of the role played by the electronic states formed as a consequence of the intermixing processes.

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⁸The common definition "reactive *d*-metal–semiconductor interfaces" refers to those systems that give silicidlike or germanidlike compounds, e.g., Si-Pd, Si-Pt, Si-Ni (Refs. 9, 11, 12, and references quoted therein), Ge-Pd, Ge-Ni (Ref. 13). Usually the noble metal–elemental semiconductor interfaces are not termed "reactive," also if considerable intermixing takes place as for Si-Au and Si-Cu, because of the absence of stable stoichiometric bulk silicides.

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