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Electronic and atomic structures of a Sn induced $3\sqrt{3}\times 3\sqrt{3}$ superstructure on the Ag/Ge(111) $\sqrt{3}\times\sqrt{3}$ surface

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Abstract

We have investigated sub-monolayer coverages of Sn on the Ag/Ge(111) $\sqrt{3}\times\sqrt{3}$ surface. It was found that ≈ 0.45 monolayer (ML) resulted in a new, well-defined, reconstruction with a $3\sqrt{3}\times 3\sqrt{3}$ periodicity. The periodic structure of the surface atoms was verified by low energy electron diffraction and scanning tunneling microscopy. The electronic structure was studied in detail using angle resolved photoelectron spectroscopy and core level spectroscopy at a temperature of 100 K. Several surface bands were identified and their dispersions are presented along the $\bar{\Gamma}-\bar{M}-\bar{\Gamma}$ and $\bar{\Gamma}-\bar{K}-\bar{M}$ high symmetry lines of the $3\sqrt{3}\times 3\sqrt{3}$ surface Brillouin zone (SBZ). The $3\sqrt{3}\times 3\sqrt{3}$ surface has a metallic character since there is a strong surface band crossing the Fermi level near $\bar{\Gamma}$ -points coinciding with $\bar{K}$ -points of the 1×1 SBZ. The Fermi contour of the metallic band showed a hexagonal shape in contrast to the circular shaped Fermi contour of the initial $\sqrt{3}\times\sqrt{3}$ surface. Both empty and filled state STM images showed a hexagonal arrangement of protrusions with a local $\sqrt{3}\times\sqrt{3}$ periodicity and a superimposed modulation of the apparent heights resulting in a $3\sqrt{3}\times 3\sqrt{3}$ periodicity.

Keywords: Ge(111), Ag, Sn, monolayer, atomic structure, electronic band structure, STM, LEED, and ARPES.

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1. Introduction

It is well known in the field of surface science that various metals form one and two dimensional structures on semiconductor surfaces. Most of such structures are formed by adding a single metallic species on a semiconductor surface, such as Si(111) or Ge(111), resulting in a variety of reconstructions [1-7]. In other studies, tiny amounts (<0.2 monolayer (ML)) of a second metal have been evaporated onto the reconstructed surface induced by the first metal. Such tiny amounts are in several cases sufficient to form new reconstructions leading to modified surface electronic structures [8-12]. It is also interesting to form 2D structures consisting of two metals, where the second metal has a coverage of >0.2 ML ranging up to a 1 ML. After annealing, a binary surface alloy may form, i.e., a 2D surface layer with a new, well-ordered, atomic structure. These atomic structures are in most cases rather complex and they are difficult to model. However, these binary systems provide a new platform for the study of various physical phenomena based on interactions between the two metal species themselves and with the substrate. Ag and Sn are examples of two metals that form well-ordered surface alloys on Si(111) and Ge(111) [13,14]. In the case of Ge(111), a 3×3 periodicity is formed by 0.75 ML of Sn deposited on a Ag/Ge(111) $\sqrt{3}\times\sqrt{3}$ surface followed by annealing up to 300 °C [13]. Various amounts of Sn were investigated in order to check for other phases of the Sn/Ag/Ge(111) system. It was found that a Sn coverage in the range 0.25 – 0.45 ML gave rise to a $3\sqrt{3}\times 3\sqrt{3}$ periodicity after annealing.

In the present study, the electronic structure of the Sn/Ag/Ge(111) $3\sqrt{3}\times 3\sqrt{3}$ surface was investigated using angle resolved photoelectron spectroscopy (ARPES) and core level spectroscopy at 100 K (LT). Band dispersions were measured along the $\bar{\Gamma}-\bar{M}-\bar{\Gamma}$ and $\bar{\Gamma}-\bar{K}-\bar{M}$ high symmetry directions of the $3\sqrt{3}\times 3\sqrt{3}$ surface Brillouin zone (SBZ). The Fermi contour, due to a band crossing the Fermi level, was measured. In contrast to a circular contour of a similar band on the Ag/Ge(111) $\sqrt{3}\times\sqrt{3}$ surface, the one on the $3\sqrt{3}\times 3\sqrt{3}$ surface has a hexagonal shape. The atomic structure, studied by scanning tunneling microscopy (STM) at RT, shows a local hexagonal arrangement of small protrusions with a $\sqrt{3}\times\sqrt{3}$ periodicity. The apparent heights of the protrusions show a variation which results in an overall $3\sqrt{3}\times 3\sqrt{3}$ periodicity.

2. Experimental details

The experimental setup to measure the electronic structure consisted of two UHV chambers, one for surface preparation, equipped with Ag and Sn evaporators, a sputter gun, a quartz crystal thickness monitor, and a residual gas analyzer. The other chamber had a SPECS Phoibos 100 electron analyzer and a low energy electron diffraction (LEED) optics which was used to align the sample azimuthally. Both chambers had base pressures of less than 1×10^{-10} Torr, while during surface preparation and evaporation the pressure in the preparation chamber was $< 4 \times 10^{-10}$ Torr. The experimental setup for the ARPES measurements is located at the I4 beamline at the MAX-III storage ring of the MAX-lab synchrotron radiation facility in Lund, Sweden. Beamline I4 provides linearly polarized synchrotron light in the energy range 13–160 eV. The ARPES data were obtained at energy and angular resolutions of ≈ 50 meV and 0.3° , respectively. A Ge(111) substrate was cut from an n-type (Sb) Ge(111) wafer, with a resistivity of 7–10 Ωcm at room temperature. It was degreased *ex-situ* by acetone and isopropanol and cleaned *in-situ* by repeated Ar^+ ion sputtering (1 keV) and annealing cycles. This procedure resulted in an atomically clean Ge(111) surface with a well-ordered $c(2 \times 8)$ structure as verified by LEED. The rates of the Ag and Sn evaporators were established, using a quartz crystal thickness monitor, to be 0.6 ML/min, where one ML is defined as the number of atoms per cm^2 on the unreconstructed Ge(111) surface (i.e. 7.2×10^{14} atoms/ cm^2). The preparation of the $\text{Sn/Ag/Ge/(111)}\sqrt{3} \times \sqrt{3}$ surface was done in two steps. Initially, 1.1 ML of Ag was deposited on the clean $\text{Ge(111)}c(2 \times 8)$ surface which resulted in a well-ordered $\text{Ag/Ge(111)}\sqrt{3} \times \sqrt{3}$ surface after annealing at 330°C for a few minutes. In the second step, 0.45 ML of Sn was evaporated on the $\text{Ag/Ge(111)}\sqrt{3} \times \sqrt{3}$ surface followed by annealing at 300°C for a few minutes. The formation of a $\text{Sn/Ag/Ge(111)}\sqrt{3} \times \sqrt{3}$ periodicity was verified by LEED.

STM studies of the $\text{Sn/Ag/Ge(111)}\sqrt{3} \times \sqrt{3}$ surface, prepared by the procedure described above, were performed in a separate UHV chamber at Linköping university. The Omicron VT-STM used in these studies has a W-tip that was prepared by electrochemical etching and further cleaned *in-situ* by electron beam heating. All STM measurements were performed in constant current mode (200 pA) and at room temperature (RT).

3. Results and discussion

3.1 Atomic structure

Figure 1 shows LEED results obtained after each surface preparation step. Figs. 1(a)–1(c), show well-defined diffraction spots of the clean Ge(111)c(2×8), Ag/Ge(111) $\sqrt{3}\times\sqrt{3}$, and Sn/Ag/Ge(111) $3\sqrt{3}\times3\sqrt{3}$ surfaces, respectively, at RT. The LEED patterns of the $3\sqrt{3}\times3\sqrt{3}$ surface in Figs. 1(d) – 1(f) were obtained at LT (100 K) at different electron energies. Rather weak, but well-defined $3\sqrt{3}\times3\sqrt{3}$ diffraction spots are present in the LEED patterns.

Fig. 1 (a)–(c) LEED patterns of clean Ge(111)c(2×8), Ag/Ge(111) $\sqrt{3}\times\sqrt{3}$ and Sn/Ag/Ge(111) $3\sqrt{3}\times3\sqrt{3}$ surfaces, respectively, obtained at RT. (d)–(f) LEED patterns of the Sn/Ag/Ge(111) $3\sqrt{3}\times3\sqrt{3}$ surface at different electron energies measured at LT (100 K). The amount of Sn was 0.45 ML.

Figure 2 shows empty and filled state STM images of the Sn/Ag/Ge(111) $3\sqrt{3} \times 3\sqrt{3}$ surface measured at RT for various sample bias voltages. In Fig. 2(a) a large scale ($156 \times 156 \text{ nm}^2$) empty state image is shown that gives an overview of the surface, showing islands on wide and flat terraces. There are two levels of terraces with a height difference of $\approx 1.6 \text{ \AA}$. The zoomed-in image ($51 \times 51 \text{ nm}^2$) in Fig. 2(b) gives a clearer view of the islands and the flat terrace. The islands have a 3×3 periodicity and are $\approx 3.0 \text{ \AA}$ higher than the flat terrace. It is clear that the islands have a hexagonal arrangement of protrusions and that the orientation of the unit cell is that of a 1×1 cell. In contrast, the flat terrace has a structure which has a different periodicity with a rather large unit cell that is rotated by 30° with respect to the 1×1 cell. This structure is described by a $3\sqrt{3} \times 3\sqrt{3}$ unit cell. However, some 3×3 regions are also present at the same level as can be seen in Fig. 2(b). Figures 2(c)–2(h) are small scale ($10 \times 10 \text{ nm}^2$) empty and filled state images of the same region measured consecutively by changing the tunneling bias. Figures 2(c) and 2(d) show empty and filled state images at a sample bias of $\pm 0.6 \text{ V}$, respectively. At positive bias, the image shows a clear arrangement of protrusions forming a hexagonal mesh. There are two kinds of periodicities that can be observed, one which is the initial $\sqrt{3} \times \sqrt{3}$, which corresponds to the distance between two protrusions, but there is also another periodicity that is three times larger than the initial one, as indicated by the $3\sqrt{3} \times 3\sqrt{3}$ unit cell. This cell contains nine protrusions which are arranged locally into the hexagonal pattern. The $3\sqrt{3} \times 3\sqrt{3}$ structure is more prominent in images recorded at lower absolute values of the sample bias, see Figs. 2(e) –2(h). The $3\sqrt{3} \times 3\sqrt{3}$ unit cell is 27 times larger than the 1×1 cell. The nine protrusions observed by STM only correspond to $1/3 \text{ ML}$ while the total amount of added atoms (Ag+Sn) is 1.5 ML . This suggests that the protrusions correspond to structural features consisting of several atoms, with an unknown combination of Ag and Sn atoms.

Fig. 2 (a) and (b) Large scale ($156 \times 156 \text{ nm}^2$) and zoomed-in ($51 \times 51 \text{ nm}^2$), empty state STM images of the $3\sqrt{3} \times 3\sqrt{3}$ surface obtained at RT, respectively. (c)-(h) Small scale ($10 \times 10 \text{ nm}^2$), empty and filled state STM images of the $3\sqrt{3} \times 3\sqrt{3}$ surface measured at various sample biases. All images were obtained in constant current mode (200 pA) at RT.

3.2 Electronic structure

The detailed electronic structure of the Sn/Ag/Ge(111) $3\sqrt{3}\times 3\sqrt{3}$ surface and underlying Ag/Ge(111) $\sqrt{3}\times\sqrt{3}$ surface is shown in Fig. 3. Figure 3(a) shows $\sqrt{3}\times\sqrt{3}$ and $3\sqrt{3}\times 3\sqrt{3}$ SBZs and highlights the high symmetry points and directions. In Figs. 3(b) and 3(c) the electronic structure of the Ag/Ge(111) $\sqrt{3}\times\sqrt{3}$ surface is shown measured by ARPES using a photon energy of 30 eV at RT. The band structure is presented along the $\bar{\Gamma}-\bar{M}-\bar{\Gamma}$ and $\bar{\Gamma}-\bar{K}-\bar{M}$ high symmetry directions of the $\sqrt{3}\times\sqrt{3}$ SBZ. The ARPES data are presented as second derivatives, along the energy axis, of the original photoemission intensity in order to also visualize weak but well-defined features. There are several bands labeled S_1-S_6 . Detailed discussions about the origins and dispersions of these bands can be found in Ref. 15. Figures 3(b) and 3(c) facilitate a comparison with the electronic structure of the Sn/Ag/Ge(111) $3\sqrt{3}\times 3\sqrt{3}$ surface. Figures 3(d) – 3(g) present the electronic structure of the Sn/Ag/Ge(111) $3\sqrt{3}\times 3\sqrt{3}$ surface. ARPES data were obtained using various photon energies, i.e., 17, 21.2, 30, 40, and 52 eV. In this paper, only 30 eV data is presented. All experiments were performed at low temperature (≈ 100 K). Figures 3(d) and 3(e) show the second derivative versions of the electronic structure in Figs. 3(f) and 3(g), along the high symmetry lines $\bar{\Gamma}-\bar{M}-\bar{\Gamma}$ and $\bar{\Gamma}-\bar{K}-\bar{M}$ of the $3\sqrt{3}\times 3\sqrt{3}$ SBZ. The derivation was executed along the energy axis, where dark features represent the bright and/or well-defined features in the original photoemission data shown in Figs. 3(f) and 3(g). In Figs. 3(d) and 3(e), a black curve is included which represents the edge of the projected bulk bands, whereas vertical lines mark the high symmetry points. The bands in the gap region are true surface bands and these are the only ones that are included in the discussion. There are seven bands labeled S_1-S_7 in Figs. 3(d) and 3(e). Four of them follow the initial $\sqrt{3}\times\sqrt{3}$ periodicity, while the other three bands belong to the $3\sqrt{3}\times 3\sqrt{3}$ periodicity. In Figs. 3(d) and 3(f), there are four bands along the $\bar{\Gamma}-\bar{M}-\bar{\Gamma}$ direction labeled S_1-S_4 within 1.4 eV below E_F . The S_1 band shows a very steep dispersion down to -1.0 eV at the outer $\bar{\Gamma}_1$ - points (or $\bar{K}_{1\times 1}$ -points). S_1 seems to be similar to the free electron like band of the initial $\sqrt{3}\times\sqrt{3}$ surface, see Fig. 3(b), except for a large downward shift of about 0.7 eV. The S_1 band of the Ag/Ge(111) $\sqrt{3}\times\sqrt{3}$ is discussed in detail in Ref. 15.

Fig. 3 (a) $\sqrt{3} \times \sqrt{3}$ and $3\sqrt{3} \times 3\sqrt{3}$ SBZs with high symmetry points and directions. (b) and (c) ARPES results from the initial Ag/Ge(111) $\sqrt{3} \times \sqrt{3}$ surface along the high symmetry lines $\bar{\Gamma}-\bar{M}-\bar{\Gamma}$ and $\bar{\Gamma}-\bar{K}-\bar{M}$ of the $\sqrt{3} \times \sqrt{3}$ SBZ, measured with a photon energy of 30 eV at RT. (d) and (e) ARPES results from the Sn/Ag/Ge(111) $3\sqrt{3} \times 3\sqrt{3}$ surface along the high symmetry lines $\bar{\Gamma}-\bar{M}-\bar{\Gamma}$ and $\bar{\Gamma}-\bar{K}-\bar{M}$ of the $3\sqrt{3} \times 3\sqrt{3}$ SBZ, measured with a photon energy of 30 eV at 100 K. (b) – (e) are plots of the second derivative along the energy axis of the original photoemission intensity data. (f) and (g) show the original photoemission data as gray scale images from which the second derivative plots in (d) and (e) were derived. Note that the symmetry point labels used in (b) – (g) correspond to the $\sqrt{3} \times \sqrt{3}$ SBZ.

A second band, S_2 , is present at $\overline{M}_1$ -points at an energy of -0.4 eV and has a downward dispersion toward $\overline{\Gamma}$ -points. It is, however, difficult to follow the complete dispersion, see Fig. 3(d). Another band, labeled S_3 , is located within the energy range 0.8 – 1.4 eV below E_F . The band has a strong appearance along the $\overline{\Gamma}$ – $\overline{M}$ line with energy maxima at the $\overline{M}_1$ -point with a binding energy of -1.0 eV. Furthermore, it disperses downward toward $\overline{\Gamma}_1$ -points. S_3 is degenerate with another band S_4 at $\overline{\Gamma}_1$ -points. The S_4 band has a downward dispersion to -1.6 eV along the $\overline{\Gamma}$ – $\overline{M}$ line. Both S_3 and S_4 belong to the initial $\sqrt{3} \times \sqrt{3}$ surface since both have dispersions similar to bands described in Ref. 15 for Ag/Ge(111) $\sqrt{3} \times \sqrt{3}$, see Fig. 3(b). In Figs. 3(e) and 3(g), the surface band structure along $\overline{\Gamma}$ – $\overline{K}$ – $\overline{M}$ of the $3\sqrt{3} \times 3\sqrt{3}$ SBZ is shown. Band S_5 has maxima at $\overline{M}_1$ -points and exists within the energy range of 0.6 – 1.0 eV below E_F . This band was also observed on the initial Ag/Ge(111) $\sqrt{3} \times \sqrt{3}$ surface as shown in Fig. 3(c). One more band, S_6 , which also belongs to the Ag/Ge(111) $\sqrt{3} \times \sqrt{3}$ surface, appears within a similar energy range, i.e., 0.8 – 1.2 eV below E_F , see Fig. 3(c). There is, however, an obvious difference in the dispersion of the band. The maxima observed at $\overline{M}_1$ -points in case of the Ag/Ge(111) $\sqrt{3} \times \sqrt{3}$ surface, has changed to minima, cf. Figs. 3(c) and 3(e). This qualitative change of the dispersion of this band was also observed when the Ag/Ge(111) $\sqrt{3} \times \sqrt{3}$ surface was converted to Ag/Ge(111) 6×6 by adding 0.2 ML of Ag [16]. Finally the S_7 band has an energy minimum of -0.9 eV at the $\overline{K}_1$ -point and disperses upward along $\overline{\Gamma}$ – $\overline{K}_1$ line to its maximum energy of -0.4 eV at $k_{\parallel}=0.4 \text{ \AA}^{-1}$ and then it disperses downward toward $\overline{\Gamma}$. The band exhibits a similar dispersion between $\overline{K}_1$ and $\overline{M}_1$ with a maximum energy of -0.4 eV at $k_{\parallel}=0.8 \text{ \AA}^{-1}$, see Fig. 3(e). Figures 3(f) and 3(g) show the electronic structure data from the $3\sqrt{3} \times 3\sqrt{3}$ surface as intensity maps in the energy versus $k_{\parallel}$ plane along the high symmetry lines $\overline{\Gamma}$ – $\overline{M}$ – $\overline{\Gamma}$ and $\overline{\Gamma}$ – $\overline{K}$ – $\overline{M}$ of the $3\sqrt{3} \times 3\sqrt{3}$ SBZ. Some of the bright dispersing features correspond to surface bands, while others originate from transitions in the bulk band structure.

3.3 Constant energy contours

Figures 4(a)-4(d) present Fermi contours and constant energy contours at -0.1 eV for the Ag/Ge(111) $\sqrt{3} \times \sqrt{3}$ and Sn/Ag/Ge(111) $3\sqrt{3} \times 3\sqrt{3}$ surfaces. The contours at E_F , i.e., the Fermi “surface”, has a slightly weaker intensity due to the steep slope of the Fermi-Dirac function, while the contours at lower binding energy show the shape of the contours more clearly. In Figs. 4(a) and 4(b), the contours are circular around a $\bar{\Gamma}$ -point of the $\sqrt{3} \times \sqrt{3}$ SBZ, but there is an “opening” on the right side that is due to polarization effects which result in an asymmetric emission intensity [17]. It is interesting to note that the contours of the $3\sqrt{3} \times 3\sqrt{3}$ surface, in Figs. 4(c) and 4(d), show a clear hexagonal shape which is in contrast to the circular contours of the initial Ag/Ge(111) $\sqrt{3} \times \sqrt{3}$ surface. However, these contours exhibit similar asymmetry in the emission intensity as in the $\sqrt{3} \times \sqrt{3}$ case [17].

Fig. 4 (a)-(b) and (c)-(d) Constant energy contours of the Ag/Ge(111) $\sqrt{3} \times \sqrt{3}$ and Sn/Ag/Ge(111) $3\sqrt{3} \times 3\sqrt{3}$ surfaces at RT and LT respectively, measured at a photon energy of 30 eV. The contours in (c)-(d) reveal the existence of hexagonal electron pockets around $\bar{\Gamma}$ -points of the $3\sqrt{3} \times 3\sqrt{3}$ SBZs, coinciding with $\bar{K}_{1 \times 1}$ -points, instead of the circular contours observed for Ag/Ge(111) $\sqrt{3} \times \sqrt{3}$ in (a)-(b).

3.4 Core level spectra

In Fig. 5, we present Sn 4*d* and Ge 3*d* core level spectra from the Sn/Ag/Ge(111) $3\sqrt{3}\times 3\sqrt{3}$ surface measured at a photon energy of 80 eV at LT. The Sn 4*d* core level spectrum consists of a well-resolved spin-orbit split component without any clear shifts.

Fig. 5 (a) and (b) Sn 4*d* and Ge 3*d* core level spectra obtained at 100 K from the Sn/Ag/Ge(111) $3\sqrt{3}\times 3\sqrt{3}$ surface, using a photon energy of 80 eV. A Doniach-Šunjić line shape was used to fit the Sn 4*d* (Ge 3*d*) spectra with a Gaussian width of 263 (365) meV, a Lorentzian width of 199 (84) meV, and an asymmetry parameter of 0.03 (0.03).

The spectrum is satisfactorily fitted by just one component with a Gaussian width of 260 meV and Lorentzian width of 200 meV. The slight asymmetry on the low kinetic energy side of the spectrum could be accounted for by an asymmetry parameter $\alpha=0.03$ in the Doniach-Šunjić line shape [18]. The existence of just one sharp component indicates that the Sn atoms are located at similar, well-defined, positions on the $3\sqrt{3}\times 3\sqrt{3}$ surface. The Ge 3*d* spectrum in Fig. 5(b) is quite featureless and there are no obvious shifted components, although the spin-orbit split is

well-resolved. The spectrum is fitted by one component with a Doniach-Šunjić line shape, using a Gaussian width of 365 meV, a Lorentzian width of 84 meV, and an asymmetry parameter of $\alpha = 0.03$. The lack of obvious shifts indicates that also the Ge atoms are located in reasonably well-defined positions on the $3\sqrt{3} \times 3\sqrt{3}$ surface.

4. Conclusions

In this study, we have presented a detailed investigation of the electronic and atomic structures of Sn/Ag/Ge(111) $3\sqrt{3} \times 3\sqrt{3}$ at 100 K. Large scale overview images obtained by STM show islands on flat regions with 3×3 periodicity, while the flat regions themselves exhibit a structure with a $3\sqrt{3} \times 3\sqrt{3}$ periodicity. Small protrusions are arranged in a hexagonal pattern as shown in small scale STM images. ARPES measurements were employed to investigate the band dispersions along the high symmetry directions and constant energy contours were obtained for the metallic band. The Fermi contours showed a hexagonal shape in contrast to the initial $\sqrt{3} \times \sqrt{3}$ surface. There are seven surface bands labeled in the ARPES data, among these, four belong to the initial $\sqrt{3} \times \sqrt{3}$ surface.

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