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Origin of a surface state above the Fermi level on Ge(001) and Si(001) studied by temperature-dependent ARPES and LEED

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Variable temperature photoemission studies in the literature have revealed the presence of a surface state above the Fermi level on clean Ge(001). We present photoemission and low energy electron diffraction results from Ge(001) obtained between 185 and 760 K. Our measurements show a peak above the Fermi level with a maximum intensity at a sample temperature of around 625 K. At higher temperatures, we observe a gradual decrease in the intensity. Angle resolved spectra show that the surface state has a $\bar{k}_{\parallel}$ dependence and is therefore not attributed to defects. Very similar results were obtained on both an intrinsic (30 Ω cm) and a 10 m Ω cm *n*-type sample. The overall appearance of the spectral feature is found to be quite insensitive to sample preparation. Low energy electron diffraction investigations show how the sharp $c(4 \times 2)$ pattern becomes streaky and finally turns into a 2×1 pattern. The onset of the structure above the Fermi level takes place just before all $c(4 \times 2)$ streaks have disappeared which corresponds to a temperature of around 470 K. On Si(001), we also observe photoemission intensity above the Fermi level. It is weaker than on Ge(001) and appears at higher temperature. We find that the emission above the Fermi level can be explained by thermal occupation of the π^* band derived from a 2×1 ordering of asymmetric dimers on the surface.

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I. INTRODUCTION

Electronic properties of semiconductors have attracted a lot of interest based on technological importance as well as fundamental scientific issues. The discovery of electronic states localized at the surface resulted in a large experimental and theoretical effort to understand the observations. Among the most intensely studied surfaces are Si(001) and Ge(001). By combining theoretical calculations and experiments, they have been found to form various reconstructions composed of tilted dimers. The surface electronic structure is semiconducting with separated filled and empty surface state bands. The filled bands have been mapped out in a large number of photoemission reports, see, e.g., Refs. 1 and 2, while the empty bands are more elusive and are mainly known from calculations. From inverse photoemission³ and scanning tunneling spectroscopy,⁴ their existence have been demonstrated.

Using angle resolved photoelectron spectroscopy (ARPES), Kevan⁵ reported a state near the Fermi level (E_F) on a heated Ge(001) surface that seemed to appear simultaneously with the $c(4 \times 2)$ to 2×1 surface phase transition. It was observed near the $\bar{\Gamma}$ point and assigned to a dimer flip induced defect state in the band gap. Later, ARPES studies on heated Ge(001) reported this state to be actually located above E_F .^{6,7} Furthermore, the state showed a $\bar{k}_{\parallel}$ dependent intensity variation consistent with that of the surface band structure and it was suggested to be due to a partial occupation of the empty π^* band originating from the dimer down atoms. Several explanations to the occupation of the state above E_F were presented. One category of suggestions was based on a metallicity created by the flipping of the dimers. References 4 and 8 identified the symmetric state of the flipping dimers as a possible explanation based on scanning tun-

neling spectroscopy and Monte Carlo calculations, respectively. Reference 4 also suggested that the flipping motion of the dimers could give rise to a free-electron-like state near E_F . Self-doping by adatoms at elevated temperature^{6,7} belongs to another category. In this case, atoms that are released from step edges are believed to migrate on the surface and act as donors. A third suggestion is thermal occupation.¹ The temperature induced smearing of the Fermi-Dirac distribution will lead to a redistribution of the electrons into states that are empty at lower temperature.

Our results indicate that thermal occupation at elevated temperatures is responsible for the appearance of the π^* state in photoemission. We have also investigated the correlation of the state to the surface phase transition. By comparing the emission intensity of the state, as a function of temperature, with low energy electron diffraction (LEED) spot intensities we find that the appearance of the state is not coupled to the $c(4 \times 2)$ to 2×1 surface phase transition. ARPES at symmetry points of the surface Brillouin zone (SBZ) combined with calculated surface band structures^{6,9} from the literature allowed us to associate the intensity from the π^* state above E_F with the high temperature 2×1 phase. By dividing with the Fermi-Dirac distribution function, we estimate that the π^* band is 0.13 eV above E_F at $\bar{\Gamma}$. A structure 0.17 eV below E_F is assigned to the occupied π band based on a comparison with the valence band study by Kipp *et al.*¹⁰ Measurements on Si(001) show similar results, but here the π^* state is 0.24 eV above E_F and the π state is 0.45 eV below E_F .

II. EXPERIMENTAL DETAILS

The experimental work was conducted at beamline 33 situated at the MAX-I storage ring at the MAX-lab synchro-

tron radiation facility in Lund, Sweden. In the photoemission measurements, the energy resolution was about 80 meV and the angular resolution was normally $\pm 2^\circ$.

The data were obtained in a temperature range between about 185 and 760 K. This was made possible by the use of liquid nitrogen cooling in combination with direct resistive sample heating using a custom built heating device that alternates, in the kilohertz range, between passing the heating current through the sample and sending the signal from the electron analyzer to the data taking computer. Temperatures above approximately 550 K were monitored with an IR pyrometer. The lower temperatures were calibrated to the heating current at a separate occasion using a thermocouple clamped onto the sample. Sample cleaning was done by several cycles of Ar^+ sputtering (500 eV) and annealing to about 960 K. The measurements were repeated with two different Ge(001) samples, one n -doped 10 m Ω cm (antimony) and one intrinsic 30 Ω cm. Both samples produced similar intensities of the state above E_F , but the high voltages required for heating the intrinsic sample rendered the LEED images less clear. We therefore only present data from the n -doped sample. The Si sample, n -doped (phosphorous) 2 Ω cm, was thoroughly outgassed and, as a last step, annealed several times at 1520 K for 1–2 s. E_F of a Ta foil in electrical contact with the sample was used as reference in the photoemission data.

III. RESULTS AND DISCUSSION

Normal emission valence band spectra from a clean Ge(001) surface at various temperatures are shown in Fig. 1(a). The spectra have been normalized with respect to the photon flux. At an energy of 70 meV above E_F , there is a structure labeled S. A similar structure was reported by Kevan⁵ more than 20 years ago. It was placed below E_F but several later reports^{1,6,7} agree on a position above E_F . Even though there is a discrepancy between the energy positions in the study by Kevan and the other reports, it is believed to be the same structure. Both Kevan⁵ and Nakatsuji *et al.*⁶ reported a monotonically increasing intensity of S with higher temperatures up to around 500 and 680 K, respectively. Using the intensity in the valence band spectra 0.4–0.5 eV below the S structure as a reference, we find that S reaches a peak intensity that is about two times higher than in those previous reports. The present results also reveal that S decreases in intensity at temperatures higher than about 625 K. The highest valence state was identified by Kipp *et al.*¹⁰ to originate from the occupied dangling bond of the dimer up atoms. In Fig. 1(a), this state is labeled S_{up} , and the position is 0.17 eV below E_F . Since the energy position of the S_{up} state is 0.1 eV lower in our study compared to Ref. 10, it is reasonable to assume that the component 0.27 eV below E_F that they identified as the valence band maximum (VBM) is the component we find at 0.37 eV below E_F .

In addition, spectra covering a larger energy range were also recorded in order to monitor shifts of bulk and surface states. From those spectra, a shift toward higher binding energy by about 0.15 eV could be observed in the temperature range 185–875 K of the bulk state 3 eV below E_F at $\bar{\Gamma}$. The

FIG. 1. Photoemission spectra (raw data) and LEED images obtained at temperatures from 185 to 760 K. (a) State S is found above E_F , dashed line, at elevated temperatures. The occupied dangling bond state, S_{up} , originating from the dimer up atoms is marked in the lowest spectrum. The spectra have been normalized to the photon flux. (b) Crops of the first quadrant of LEED images taken with a beam energy of 119 eV. The $(\frac{3}{4}, \frac{1}{2})$ spot, vertical arrow, and the (1,1) spot, horizontal arrow, are marked in the 185 K panel.

broadening at higher temperatures does, however, make it difficult to determine the magnitude of this shift accurately. A similar shift of 0.1 eV was observed in Ref. 1 between spectra obtained at room temperature and directly after annealing. This was explained in terms of a change in E_F toward the conduction band minimum. Reference 6 explains a similar shift as being due to a change in the band bending as well. It should be noted that we did not observe such a shift on the Ge 3d core level nor on the bulk state 0.37 eV below E_F , so it is possible that the shift of the 3 eV bulk state has a different origin.

In the initial report,⁵ the S component was correlated to surface changes, as observed in LEED. The appearance of S and the $c(4 \times 2)$ to 2×1 transition was found to coincide at a temperature of around 220 K. We have performed a similar study and photoemission spectra together with first quadrant crops of LEED patterns, obtained using the same heating currents, are displayed in Figs. 1(a) and 1(b), respectively. At lower temperatures, below 550 K, we estimate the error in the absolute temperature to be on the order of a few 10 K while at higher temperatures, where an IR pyrometer was used, the error is expected to be below 10 K. Even though the absolute temperature could not be determined exactly, the photoemission spectra and their corresponding LEED patterns were obtained at the same temperature. This was ensured by precise control of the heating current and careful timing. The intensity of S in Fig. 1(a) starts to increase above

FIG. 2. Intensity of the $(\frac{3}{4}, \frac{1}{2})$ LEED spot (dotted curve). The intensity of state S (solid curve) is estimated by fitting a Gaussian, as shown in the inset. In the fitting procedure, another Gaussian is used to estimate the intensity from the occupied dangling bond state, S_{up} , originating from the dimer up atoms.

395 K, reaches a maximum around 625 K, and then decreases. To estimate the intensity of S, a Gaussian fit was performed, as shown in the inset in Fig. 2. An additional Gaussian component was included to account for the contribution from the occupied dangling bond state, S_{up} , 0.17 eV below E_F . A good fit was obtained by keeping the energy separation of the S and S_{up} components fixed at 0.24 eV and only allowing their widths to vary. To achieve a good fit for spectra above 625 K, a shift of 30 meV toward higher binding energy was introduced to the two components. The solid curve in Fig. 2 shows the intensity of S vs temperature.

In the 185 K LEED pattern in Fig. 1(b), the vertical arrow indicates a $(\frac{3}{4}, \frac{1}{2})$ spot, characteristic of the $c(4 \times 2)$ surface phase. At higher temperatures, it turns into a streak and fades out. The intensity of the $(\frac{3}{4}, \frac{1}{2})$ spot, dotted curve in Fig. 2, have dropped close to zero when state S, solid curve, starts to increase. This demonstrates that the surface phase transition is almost complete before the onset of S. There is no change of the surface as detected in LEED at higher temperatures, i.e., the 2×1 diffraction spots remain. Noting that the intensity of S in Ref. 5 was about half of the maximum value that we find, one can conclude that the two studies are consistent, except for a temperature offset. If one only considers the temperature range up to 585 K, where S has reached about 50% of the maximum intensity (see Fig. 2), it is easy to get the impression that S is related to the $c(4 \times 2)$ to 2×1 transition. However, the further increase of S beyond that temperature changes the picture and provides clear evidence that S is not coupled to the phase transition.

We can make a small remark regarding the streaks in the LEED patterns in Fig. 1(b) by noting that they fade out very slowly with temperature. It has not been possible to reproduce the streaks using Monte Carlo calculations¹¹ and it is believed¹² that a strong short range order along the dimer rows, induced by the anisotropic displacement of second-layer atoms, is responsible for the streaks appearing in LEED. Our results suggest that there may be a correlation between the streaks and the surface state that appears 3 eV below E_F around $\bar{J}'_2$. This state was identified as a back-bond resonance¹³ with maximum state density at the second layer.

FIG. 3. Photoemission intensity near E_F at different points in the SBZ. (a) Photoemission spectra (raw data) normalized to the photon flux obtained at different emission angles Θ_e corresponding to points a-c in the SBZs. The dotted lines are visual aids and represent the base level for each spectrum. The inset shows the rapid drop in the intensity of S with emission angle near normal emission. (b) Solid and dotted lines indicate the $c(4 \times 2)$ SBZs (left) and 2×1 SBZs (right) of the two domains that differ by a 90° rotation. Points a-c mark points probed with ARPES. Point a represents normal emission.

Measurements on this surface state, in the study by Landemark *et al.*,¹ showed no major change up to RT. We found a similar behavior but could at higher temperatures, in the temperature range where the LEED streaks completely fade out, observe a sudden shift of almost 0.1 eV toward lower binding energy with increasing temperature. These observations indicate that a change in the dimer back bond, and consequently in the second layer, could be related to the disappearance of the streaks in the LEED patterns.

The photoemission intensity near E_F was also probed at various emission angles in the $[010]$ and $[\bar{1}10]$ directions. Figure 3(a) shows spectra from three different points (a-c) in the SBZs. In the $[010]$ direction, the two domains on the surfaces are degenerate, as illustrated in Fig. 3(b). Point a corresponds to the $\bar{\Gamma}$ point. The inset in Fig. 3(a) shows how the S intensity drops rapidly with increasing emission angle. According to calculations,^{6,9} the π^* band is low in energy at $\bar{\Gamma}$ in both the $c(4 \times 2)$ and the 2×1 models and at $\bar{J}'$ in the 2×1 model but not at the $\bar{J}$ point in either of the models. At point b, the S intensity is close to zero, as shown in Fig. 3(a), but at point c, $\bar{J}'_2$ and $\bar{J}'_2$ in the 2×1 cell and $\bar{J}_2$ and $\bar{J}_2$ in the $c(4 \times 2)$ cell, there is again an increase in intensity. This supports the assignment^{1,6} of S to the ordinarily empty π^* band. Furthermore, it supports the assignment of S to the

FIG. 4. Valence band spectra (raw data) at different temperatures from the $\bar{J}'_2$ point, see point c of Fig. 3(b). At temperatures below 365 K, a state S_2 is visible that is characteristic of the $c(4 \times 2)$ surface phase. Above 425 K, intensity above E_F , marked $S(\bar{J}'_2)$, starts to increase due to filling of the π^* band.

2×1 surface phase based on the calculated surface band structures.^{6,9} Point c was chosen for further investigations since a state that is characteristic of the $c(4 \times 2)$ phase is visible at low temperature 0.55 eV below E_F , as indicated by S_2 in Fig. 4 (compare with S_2 in Ref. 1). At a temperature of around 365 K, it disappears, and around 425 K, intensity starts to build up above E_F , labeled $S(\bar{J}'_2)$ in Fig. 4. The temperature evolution is similar to that observed at the $\bar{\Gamma}$ point, as shown in Fig. 2. The decrease of S at temperatures above 625 K may be due to smearing of the bands in $\bar{k}_{\parallel}$ space. This would make the electrons less localized as the band minima become less pronounced.

We now have three different factors that indicate that S should be assigned to the 2×1 but not to the $c(4 \times 2)$ phase. First, the LEED patterns show no trace of the $c(4 \times 2)$ phase when S has its maximum intensity. Second, photoemission intensities at symmetry points agree with minima of the π^* band calculated for the 2×1 model. Third, the surface state S_2 at $\bar{J}'_2$ that is characteristic of the $c(4 \times 2)$ band structure disappears before the onset of S. This implies that S should be assigned to the 2×1 band structure.

Several suggestions have been made of possible explanations for the appearance of S in the photoemission spectra. These include symmetrization of the dimers,^{4,8} flipping of the dimers,⁴ defects,⁵ doping by adatoms released from step edges,^{6,7} and thermal filling.¹

Symmetrization of the dimers would make the surface band structure metallic.¹⁴ Instead of having one empty π^* band and one occupied π band formed by the dangling bond orbitals, there would be two partially filled bands crossing E_F . ARPES data do not show any signs of such metallic

bands; hence, we cannot find any support for the idea of symmetric dimers giving rise to S. Furthermore, the hypothetical symmetric dimer structure would lead to a spectral feature at or slightly below E_F . This is in qualitative disagreement with the experimental finding that S is actually located above E_F . Neither can we, in our results, find any support for S being a metallic state induced by the flip-flop motion of the dimers since the surface stays semiconducting even when S appears.

The n -doped sample was prepared several times at several different occasions always showing virtually the same behavior. The onset of S, the maximum intensity, and the disappearance of the $c(4 \times 2)$ streaks in the LEED pattern occurred within just a few 10 K for all preparations. The intrinsic sample also gave very similar temperatures for the onset and maximum intensity of S. We therefore conclude that S is not very sensitive to the sample preparation. After being exposed to residual gas in the chamber for several hours, we found the drop in the S_{up} and S intensities to be very similar, contrary to the report by Jeon *et al.*⁷ who found S to be more sensitive.

Kevan⁵ associated the intensity to a state in the band gap distributed over three dimers, 10–12 Å, created by a single dimer flip defect. This interpretation should result in a structure at, or below, E_F in contrast to our result and the results of Refs. 1, 6, and 7.

Doping by the release of adatoms from step edges at elevated temperatures has been suggested as an explanation to S.^{6,7} We have not been able to find any support for such a phenomenon. Adatoms acting as donors would, in analogy with alkali metals on Si(001) (Ref. 15) and Ag on Si(111)- $(\sqrt{3} \times \sqrt{3})R30^\circ$ -Ag,¹⁶ result in a shift of E_F into unoccupied bands in order to accommodate the additional electrons. This would not give a state above E_F and hence we find this explanation unlikely.

We find that the most plausible explanation for S is the occupation of the π^* band minima due to thermal broadening of the Fermi-Dirac distribution function. Figure 5(a) shows the Ge valence spectra after that the temperature dependent Fermi-Dirac distribution function has been divided out. In principle, such an operation should give a density of states (DOS)-like curve. A small vertical offset of 3% was added to the Fermi-Dirac function in order to avoid divergence due to division by small numbers. The temperature evolutions of the DOS-like structure and S in the raw spectra are very similar. Division by the Fermi-Dirac distribution function results in a 60 meV energy shift of the peak position. The peak now appears 0.13 eV above E_F and no shift is detectable in the temperature range where it is visible.

Photoemission intensity above E_F has also been observed on Si(001).¹⁷ Figure 5(b) shows normal emission valence band spectra from an n -type Si sample at three different temperatures. From the raw spectra, dotted curves, it is evident that the intensity above E_F is very weak compared to the Ge sample. In the temperature range up to 875 K, no decrease in intensity can be observed on the structure in the Fermi-Dirac divided spectra, solid curves, only a saturation. Once the intensity becomes significantly higher than the noise level, the DOS-like structure stays at 0.24 eV above E_F . The π^* states on Ge and Si have been probed previously using in-

FIG. 5. Valence band spectra at different temperatures after the Fermi-Dirac distribution function has been divided out. (a) Results from the Ge(001) spectra in Fig. 1(a). The DOS-like structure is marked by a dotted line at 0.13 eV. (b) Si(001) raw spectra (dotted curves) and after division by the Fermi-Dirac distribution function (solid curves). A DOS-like structure is marked by a dotted line at 0.24 eV.

verse photoemission.³ In that study, Ge showed a large and sharp π^* state 0.6 eV above the VBM at $\bar{\Gamma}$, while that of Si was somewhat less distinct 0.72 eV above the VBM. For Ge, we can determine the VBM to π^* separation to be 0.5 eV which is in reasonable agreement with Ref. 3. On Si(001), the VBM is ~ 0.4 eV below E_F on a recently annealed sample¹⁸ and, consequently, we have a VBM to π^* separation of ~ 0.64 eV, also in agreement with Ref. 3.

In contrast to Ge(001), Si(001) is known to exhibit a shift of the entire spectra after annealing. The π state is found 0.45–0.5 eV below E_F at $\bar{\Gamma}$ on both a recently annealed and an actively heated sample. After cooling down to RT, it has moved down to ~ 0.7 eV. Such a shift is consistent with the energy separation of 0.24 eV between E_F and the DOS-like structure in Fig. 5(b). On the RT surface, E_F is pinned by the π^* state at $\bar{\Gamma}$. Since the position of E_F is very close to the π^* state, a shift of E_F toward the VBM is necessary at higher temperatures in order to preserve the number of electrons. On Ge(001), E_F is positioned 0.17 eV above S_{up} and 0.13 eV below π^* , i.e., E_F is positioned almost in the center of the 0.3 eV surface band gap. This gives a much more stable position of the spectra with temperature for Ge(001).

IV. SUMMARY

We have investigated the origin of the surface state, S, that appears above E_F in ARPES of Ge(001). Based on three key observations, we conclude that S is not related to the $c(4 \times 2)$ to 2×1 phase transition, as suggested in the initial study.⁵ Compelling experimental evidence has been presented that connects S solely to the 2×1 phase. Although much weaker, also Si(001) shows a structure above E_F . Similar to Ge(001), it appears well above the temperature of the $c(4 \times 2)$ to 2×1 phase transition. Of the various explanations that have been proposed for S in the literature, we find that only thermal occupation of the empty π^* band is consistent with our results. By dividing the normal emission photoemission spectra by the Fermi-Dirac distribution function, a DOS-like structure was found 0.13 and 0.24 eV above E_F for Ge(001) and Si(001), respectively. As a consequence of the difference in the positions, the thermal occupation on Si(001) is much smaller, as observed in the photoemission experiment.

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