Lack of temperature dependence for the filling of the metallic-like bands in Si(553)-Au

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We study the temperature dependence of the metallic-like bands of Si(553)-Au by angular-resolved photoemission spectroscopy (ARPES). First, we define a protocol for dealing with the short-term stability due to surface contamination and the effect of photo-voltage. After that, we extract the changes in the band-filling and Fermi-velocity. We conclude that both quantities change only weakly with temperature. This lack of temperature dependence can be compared to currently proposed models for the temperature evolution of the electronic structure of Si(553)-Au.

I. INTRODUCTION

The Si(553)-Au surface system is a prototype example for a low-dimensional surface system\cite{4,5}. A structural model for this surface system was refined by experiment\cite{6,7} and density functional theory (DFT)\cite{8,9}, with details still ongoing\cite{10,11}. Fig. 1(a) sketches the structure of this system, consisting of Au-decorated Si(111)-terraces. In a simplified view, there are two electronic subsystems on these terraces: one subsystem is by the structural motive of an Au-double-stranded chain. The other subsystem is located at the zig-zag edge of a Si-honeycomb chain and by 1/3-hole-filled Si-dangling bonds. Both subsystems exhibit distinct periodicities at low temperatures, $\times 2$ for the Au-chain and $\times 3$ for the Si-step edge. The $\times 3$ is only visible at low temperatures in scanning tunneling microscopy\cite{12,13} and electron diffraction\cite{14,15}.

Fig. 1(b) shows the Fermi surface at $T=50K$ in the $1\times 1$ surface Brillouin-zone measured by ARPES\cite{16}, while Fig. 1(b) shows a cut displaying the bands. The metallic-like bands in the system are one Rashba-split band pair\cite{17,18}, called S1 and S2, and another band S3. The bands S1 and S2 have a filling of about 1/2; band S3 is mostly near 1/3 but varies much stronger due to its higher sensitivity, e.g., to dopants or defects\cite{19}. The orbital origin of the S1/S2-bands is from hybridization of the Au-atomic chain and the Si-atoms within the Au-ladder and to the lower right (direction in respect to Fig. 1), S3 comes from the hybridization of the left Au-atomic chain with the Si-atoms to the upper left (in reference to Fig. 1) of the Au-ladder\cite{20,21}. The zone-folding at the $\times 2$ Brillouin-zone (BZ) at low-T\cite{22,23} was explained in Ref. 17 by us as originating from a many-body effect involving a partial-gapping of the bands.

A key feature of the Si-step edge chain is the unexplained $\times 3$ periodicity, long known from STM studies. Studies by SPA-LEED\cite{24,25} found the transition temperature to be around 100K for the $\times 3$ periodicity. Naively, one expects the underlying physics from textbook phenomena like the Peierls-transition built from single-particle states. Then, the expectation is a gap-opening at the Fermi surface. However, while this physics is easy to realize for a strictly 1D system at even fractional fillings, it is much harder for odd fillings. Experiments also suggest that charge density formation by standard Peierls-like mechanisms can be excluded (see Refs. in Ref. 2). Instead, a spin-chain scenario driven by magnetic exchange interactions was proposed\cite{26}. Follow-up efforts\cite{27} described such phase transition using purely classical methods\cite{28}. Recently, there was the proposal of an order-disorder phase transition\cite{29}. This idea was also advocated by recent experimental results\cite{30,31} where Raman spectroscopy detects two variants of local lattice arrangements\cite{32}. In the order-disorder phase-transition model of Ref\cite{33} a charge transfer from the step-edge to the metallic-like bands is expected. Such change in carrier-density of the step-edge can then be discussed to have an effect on the dimerization of the Au-ladder \cite{34} and, therefore could act as an explanation alternative to the partial-gapping
FIG. 1: (a) Structural model of Si(553)-Au. (b) Fermi-surface at $T=50$ K with the $1\times1$ surface Brillouin-zone. (c) ARPES measurement as energy vs. momentum cut showing the Rashba-splitted band S1/S2 and band S3. $k_x$ is along the [110]-direction, along the blue line $\Gamma_{0,0}'-K_{0,0}$ in (b).

Therefore, it is interesting to experimentally determine how the S1/2 and S3 band-filling changes with temperature. This aim is followed in this work. However, the Si(553)-Au surface system is experimentally a pretty complex system for ARPES; it suffers from short-term stability of the surface and the effect of photo-voltage changing with temperature. We sense that these issues are incompletely accessed in preceding experimental works. To succeed, we have to address them first. Therefore, the following experimental section II should not merely be a repetition of standard procedures but an attempt to define a standard protocol. Only after that can the band-filling extraction succeed in the following results section III. We find only a minimal change of the band-filling of the metallic-like bands.

II. EXPERIMENTAL

A. Preparation

The preparation was similar as in Ref. [23]. We used n-doped (phosphorus) Si(553) substrates. First, the protective photoresist was removed with standard-grade acetone. Then, the samples were cleaned in an ultrasonic bath (2 min. for each solvent) with acetone, isopropanol, and methanol of the highest purity. To avoid residual solvents, the samples were blown off with dry nitrogen. The base pressure of the UHV chamber used for the in situ preparation was below $5\times10^{-10}$ mbar. After degassing the samples, we heated up to $1250^\circ$C via direct current heating to remove the protective oxide layer. Gold evaporation of 0.48 ML was performed while the substrate was held at a temperature of $650^\circ$C, followed by a short post-annealing at $850^\circ$C. The quality of the preparation was controlled by LEED.

B. Refresh

After a particular time of residual gas exposure, a significant loss of sample quality is observed in Si(553)-Au. This degradation was simulated by putting a well-prepared Si(553)-Au sample, see Fig. 2(a), into the fast entry load-lock ($p=5\times10^{-8}$ mbar) for 25 min. STM and LEED experiments evidence the adsorption of residual gas atoms, see Fig. 2(b). The LEED pattern shows an increase in the diffuse background intensity accompanied by a loss of spot intensities. In particular, the $\times2$ streaks are only slightly visible. An STM overview scan discloses a surface completely covered with adsorbates. To recover the sample, the following two-step direct current heating sequence was applied:

Step #1: $RT \xrightarrow{t=5s} 600^\circ C (5s) \xrightarrow{t=5s} RT$
FIG. 2: LEED patterns (52 eV, T =RT) and STM overview scans (39 nm × 39 nm, U =+0.5 V, I =5 pA,T =77 K) of a Si(553)-Au sample after each step of the recovery sequence: (a) directly after preparation, (b) after storage in the load lock at a base pressure of p=5 ×10⁻¹⁰mbar for 25 min., (c) after the first step of the recovery sequence, (d) after the complete sample recovery. No significant difference between the fresh and the refreshed sample can be identified.

Step #2: RT(60s) → 850°C (5s) → RT

The first step aims to desorb residual gas atoms and is accompanied by a short pressure boost. The second step ensures a reassembly of the surface after adsorbate desorption. During the second step, no further pressure boost occurs. The sample refresh is analyzed by LEED and STM experiments after step #1 and step #2 of the recovery sequence, see Fig. 2(c) and (d). After step #1, no Si(553)-Au spots but rather (1×1) spots are observed. The STM overview scan reveals a rough, disordered sample surface. After step #2, LEED and STM images indicate a well-ordered Si(553)-Au atomic wire array. No significant difference is visible when comparing the LEED image to the freshly prepared sample; cf. Fig. 2(a) and (d). There is only a slight increase in the number of defects visible in the STM image.

This process can be repeated many times without significantly losing sample quality visible in LEED and STM. Later on, we will see only a small irreversible component in our ARPES result. Nevertheless, the quality is very high after many repetitions. This contrasts the one-step process suggested in Refs. 24 and 25 which can only be repeated several times without sample degradation. A plausible explanation for this difference may be a considerable loss of Au-atoms when combining adsorbate desorption and surface reordering in one step.

C. Temperature-dependent ARPES

Photoemission measurements were performed with a non-monochromatized duo-plasmatron He-discharge lamp. The base pressure of the analysis chamber was ≈7×10⁻¹¹ mbar at low temperatures. The operational pressure in the analysis chamber was p=6×10⁻¹⁰ mbar to 1×10⁻⁹ mbar (due to the Helium gas and evaporates from the cryostat depending on the temperature) and a spot size of about 1.5×2mm². The operation conditions were optimized for emission of the He-I line at hν = 21.22 eV. The energy resolution of the spectrometer was 25 meV. Despite a residual gas filter for the He-gas-line, the sample is prone to degradation under the pressure of the He-lamp. To guarantee defect-free samples, for measuring the temperature dependence, we, therefore, performed a thermal refresh procedure as described above before each ARPES measurement.

The measurement procedure is illustrated in Fig. 3(a). The sample was thermalized for about 1h at 10K before the measurement. A resistive heater cartridge controlled the temperature ramp on the manipulator by a PID-controller using sensor B. See the lower panel of Fig. 3(a). Sensor A is a silicon diode near the cooling head, sensor B about
FIG. 3: (a) The entire ARPES measurement cycle for some selected variables of interest. The upper panel shows the power supply’s current, performing the sample’s DC heating with the refresh procedure described above. The current was adjusted to the temperatures by a pyrometer. The middle panel shows the change in pressure, and the baseline is by the He discharge lamp. The pressure was \(6 \times 10^{-10}\) mbar at low temperatures. The cryostat temperature was ramped at a constant rate and controlled by the liquid helium flux and a resistive heater. The pink arrows mark the position where the helium flux was changed manually. (b) The upper panel shows the change in surface photo-voltages (SPV) during the measurements, following overall linearly the temperature change (temperature B with the scale on the right). The lower panel shows the sample temperature as extracted from the SPV and compared to the readings of the temperature sensors.

10 mm away from the sample. The refresh described above was applied after each measurement step. The upper panel of Fig. 3 (a) shows the read-back of the current source doing the refresh by direct current heating. In the middle panel of Fig. 3 (a), one also sees a slight rise in pressure, especially for low T, when the refresh is done. After the direct current heating sequence, the temperature reached the base temperature after around 2 mins, as inferred from the low temperatures (see Fig. 3 (a), inset of the lower panel). The sample was then given 5 min. for reaching equilibrium before starting the ARPES acquisition at the read temperature. For analysis, the normalization is by the photon-dose: each spectrum was obtained with the same parameters, i.e., the same current of the He-discharge lamp and the same counting time.

Experiments showed that switching the light on and off in the laboratory had a large effect on the surface photo-voltages (SPV). Also the day-night cycle was visible. During the measurements, we therefore covered all viewports to prevent uncontrollable changes on the SPV due to environmental light pollution. As stated above, we performed under a stable photocurrent of the He-discharge lamp. Because of the SPV changing with temperature, cf. Refs. 26 and 27, we have to align the photo-electron kinetic energies by a line-fit of a Fermi function. The reference is the Fermi-energy of an Au(111) sample. The obtained SPV shows clearly a linear relation with temperature. Compared to a Schottky barrier-like situation (cf. Ref. 27, see there especially Fig. 2), the visible T-linearity is likely caused by the high bulk P-doping combined with the intense helium discharge lamp. This is also obvious from the relatively large magnitude of the shift in SPV. However, despite overall linearity, we notice some weak changes of curvature of the SPV, see pink arrows in the upper panel of Fig. 3 (b), but not in the temperature reading (red and pink arrows). These points coincide with substantial changes either in the helium flux or resistive heating. Measuring the temperature of a sample is a very delicate task for metrology. We, therefore, use the sample itself, as a built-in Schottky-diode, for temperature calibration. We assume that temperature B represents at start condition (after long thermalization) the sample temperature and that the curves of temperature B and sample temperature coincide on average, have, therefore, the smallest \(\chi^2\). These both assumptions result in the formula

\[
SPV(T) = (-1.3734\,eV \pm 6.3\,meV) + (3.5\,meV/K \pm 0.05\,meV/K) \times T
\]
FIG. 4: (a) The temperature dependence of the MDC at $E_F$. (b) ARPES map at $T=9K$ as an aid for understanding the band topology as well as the labels on the top. A small shift of the Fermi-wavelength $k_F$ is visible in the MDCs of (a) showing a reduced number of carriers at high $T$.

The inverse function, $T(SPV)$, gives the sample temperature displayed in the lower panel of Fig. 3 (b). Note that the temperature correction does not strongly alternate the final results. However, the dependence of the $SPV$, as a direct probe of the samples’ temperature, proves that we followed a very systematic heating/cooling cycle.

III. RESULTS

A. Change of Fermi-wavelength and band filling with temperature

During the temperature scan, we notice a slight change of the Fermi-wavelengths ($k_F$’s) of the metallic bands. The momentum distribution curves (MDC’s) at $E_F$ (integrated over ± 25 meV) are displayed in Fig. 4 (a), where the MDC’s of the cycle are vertically arranged; the start is on the bottom and the end on the top. Fig. 4 (b) shows an ARPES map at $T=9K$ (start of the temperature cycle) to understand the band topology and the MDC’s. In Fig. 4 (a), all the left bands bend towards lower filling when starting from the bottom at low $T$ and going to higher $T$. This bend reverses about in the middle with cool-down. An effect of sample movement can be ruled out by the inverse moment of S3 (bright white on the right side, label -S3) bending in the opposite direction. The effect is not significant (below 1%) but visible. Note that with increasing temperature, holes (and not electrons) are added to the system.

1. Irreversible and reversible components

We performed a line-fit of the MDC’s using three Lorentzians (ignoring contributions of the replica-bands S1*, S2*). The changes in Fermi-wavelength $k_F$ are shown in Fig. 5 (a)–(c). Instead of being completely reversible, we
FIG. 5: (a)-(d) Results of the line-fit of the MDC’s shown in Fig. 4. Displayed are the extracted Fermi-wavelengths for bands S1-S3. The changes in $k_F$ are considered to have an irreversible (surface aging) and a reversible component. (d) A similar two-component view applies to the photoemission intensity, either integrated over the full range or in a background region. The irreversible component can be also seen in (f)-(h) where the results of (a)-(c) are plotted against (e) the sample temperature. Please note that in (f)-(h) the uncorrected S1-S3 are shifted up by 0.035 Å$^{-1}$ (see purple arrow) to improve the visibility of the corrected $k_F(T)$. The corrected $k_F(T)$ for the bands S1-S3 is achieved by assuming the irreversible component being linear and subtracting it.

notice an asymmetry indicating irreversibility. We, therefore, consider the changes consisting of two components: One component is irreversible and the other reversible. The irreversible component stems from an aging/deterioration of the surface. The irreversible and reversible component can also be seen by the intensity integrated over the whole ARPES map or a background region. In Fig. 5(d), we compare that for the two regions. The inset specifies these regions which intensity was integrated (red for the whole ARPES map, blue for a selected background region). In all cases, the irreversible component of surface aging is characterized by a linear increase with measurement time. A correction, by subtracting this linear-T surface aging, restores the reversible behavior, see Fig. 5(f)-(h). Note that this correction does not directly depend on the temperature but assumes that a similar amount of defects is added at every refresh, which causes the irreversible component. Nevertheless, this correction requires that the temperatures at begin of the cycle (T=9K) and end of the cycle (T=16K) are almost equal. If this were not given, we would add a reversible component to the irreversible component we want to eliminate.

We now turn to the reversible behavior of the band-filling. The reversible behavior is mainly a linear T-dependent change in the filling of the metallic bands; see corrected curved in Fig. 5(f)-(h). The $k_F$ of band S3 deviates from the linear T-dependence for high temperatures. It shows a saturation. Concluding the change in filling, by considering all the metallic bands and comparing the lowest and highest temperature, our data displays a change of about 0.03 holes per 1×1 surface cell (0.18 holes per 1×6).
B. Fermi-velocity with temperature

FIG. 6: Fermi-velocity of the three bands vs. temperature. For details see text.

We determined the Fermi-velocity as follows: The dispersion of each band was obtained by the maximum position of the MDC’s. For this, each MDC was fitted with three Lorentzian for the bands S1-S3 (ignoring the replica bands S1*/S2*). After that, the Fermi-velocity $v_F$ (more precisely: the Fermi-velocity projected along $k_x$) could be extracted by a line-fit with the function $E(k)-E_F=v_F(k-k_F)$ using the obtained dispersion in the range of $E-E_F=-0.1$ eV to 0 eV. The results of this analysis in dependence on temperatures are displayed in Fig. 6. The average values are $v_F=(0.15 \pm 0.05) \text{ eV}\AA \approx (0.23 \times 10^5 \text{ m/s})$ for S1, $v_F=(0.12 \pm 0.06) \text{ eV}\AA \approx (0.18 \times 10^5 \text{ m/s})$ for S2, and $v_F=(0.12 \pm 0.05) \text{ eV}\AA \approx (0.18 \times 10^5 \text{ m/s})$ for S3. The Fermi-velocity of all the bands stays almost constant with temperature.

There are slight changes in S2 (and maybe some systematic derivations of S1/S3 around 100K) which we attribute to the systematic error of our fit-model neglecting the replica bands.

IV. DISCUSSION

We see that the photovoltage has a large effect on the shift of the ARPES spectra. Any low-temperature gap values obtained by ARPES, i.e. Ref. 11 should be therefore seen with caution as every considerable change in light intensity changes the photovoltage at low temperatures, and therefore also the ARPES spectra. In seeing this, we think that more work has to be done to measure real gap-values of the metallic-like bands by ARPES. Nevertheless, the existence of solitons in the Au-double chain proves experimentally the existence of a (partial) gap which is theoretical accessed to be of the order of 14 meV.

We found here that the filling of the metallic-like bands changes by about 0.03 holes per $1\times1$ surface cell (0.18 holes per $1\times6$) when going from $\sim$10K to 260K. The change is mainly linear for S1/2, there is a slight non-linearity in S3 at high temperatures. Because of the T-linear changing surface photovoltage, we assume that mostly the surface photovoltage generates the experimentally observed T-linear changes in band-filling.

Alternatively, we can compare to other mechanisms causing a change of filling, for example the order-disorder phase-transition of Ref. 8. In agreement to SPA-LEED results, we can assume that the phase transition of the $\times3$ edge state is completely covered within our measurement range. Therefore, we probe the full charge transfer possible. In
comparison to the order-disorder model\cite{11}, therefore, our results give quite low limits on the charge transfer as well as a change in Fermi-velocity.

About the found T-non-linearity of the $k_F$ in S3, cf. Fig. 5 (f). Compared to Ref. 22 we think it is too small to be an effect on the dimerization of the Au-ladder. It may instead be related to a many-body description of the bands. In Ref. 17 we showed that a transverse many-body gap opens below 150K. This gap locks the difference of the Fermi-momenta of the two bands, $k_{F3} - k_{F1,2} = \text{const.}$ Therefore, below $\approx 150K$, the Fermi momenta are bound to follow the same linear trend. Above this temperature, the Fermi momenta are not any more constrained, and $k_{F3}$ (or the chemical potential of this band) can relax to the value that minimizes its own energy.

The spin-chain scenario of Ref. 3\cite{11} shows no change in filling of the metallic-like bands. In that sense, our interpretation of the T-linear change in band-filling, caused by the photovoltage, complies with that. However, it would be complicated to explain the slightly different behavior of S3 with temperature. We are also skeptical if that model\cite{11} would be an adequate description because of the following arguments: While this scenario is very intriguing, it is, in essence, a single-particle, mean-field picture at energy scales of a few tenths of meVs. Thus, for a phase transition at around 100K, one needs a parameter in the Hamiltonian of the same order of magnitude. This poses a problem because there are striking differences of magnitudes: if the step-edge is treated within a one-dimensional framework, then the anisotropy of the spin-exchange $J$ needs to be $k_BT \approx 10\text{meV}$; if the 2D spin system is spatially anisotropic, then the gap would be proportional to the weakest exchange among the two directions. However, experiments indicate\cite{11} that all spin-exchange and spin-orbit terms are substantially smaller than $10\text{meV}$; the anisotropic terms are below $2\text{meV}$. This is because the physics takes place on light Si-atoms (not Au), with no direct overlap between orbitals from neighboring edges (hence: no Fock terms).

V. CONCLUSIONS

In conclusion, we established a procedure for probing temperature effects in the electronic structure of Si(553)-Au by ARPES. We found the filling and the Fermi-velocity of the metallic-like bands not strongly changing with temperature. Our results put strict margins on models for understanding the electronic structure of Si(553)-Au.

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28. The FS and the ARPES measurement of the same measurement was already presented by us in Fig.1 of Ref. 17.

29. A decay in light intensity is especially common in 2nd. generation synchrotron sources and starts to be again an issue in 4th. generation synchrotron sources.

30. Using a doubtful assumption of a frozen spin-manifold, which leads them to the dubious conclusion of violating the Mermin-Wagner theorem.

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