Electrical Conduction on Various Au/Si(111) Surface Superstructures *

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Submonolayers of Au on Si(111) exhibit various surface superstructures, \(5 \times 2, \alpha\sqrt{3} \times \sqrt{3}, 2\sqrt{2}T \times 2\sqrt{2}T, 6 \times 6, \) and \(\beta\sqrt{3} \times \sqrt{3}\), depending on the Au coverage and heat treatments. We have succeeded in detecting the clear difference in temperature dependence of electrical conductivity among these Au/Si(111) surface superstructures by using the micro-four-point probe method. The conductivity of \(6 \times 6\) is higher than that of the \(\beta\sqrt{3} \times \sqrt{3}\) in the whole temperature range (130-300 K), though both surfaces have the same Au coverage, but are different in long-range order of atomic arrangement. The surface conductivity of both surfaces exhibit semiconducting temperature dependence of conductivity, while that of the \(\alpha\sqrt{3} \times \sqrt{3}\) show a metallic character, which is consistent with surface-state bands revealed by previous photoemission spectroscopy. [DOI: 10.1380/ejssnt.2005.497]

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I. INTRODUCTION

Through the past decades, surface superstructures on bulk crystals have been employed as a new platform for exploring low-dimensional physics. Dramatic transitions of states like various kinds of phase transitions frequently occur in the superstructures due to the low dimensionality. It is an advantage of surface physics that these transitions of states can be observed directly by means of scanning tunneling microscopy (STM) and photoemission spectroscopy (PES) with high spatial/energy/momentum resolutions. However the electrical conductivity measurement for such low-dimensional systems has not been fully performed until recent years. Advanced development of experimental techniques, such as micro-four-point-probes (\(\mu\)APP) and independently driven four-tip STM (4TSTM), make it possible to measure the conductivity of superstructures directly [1–3]. And important results, such as detection of anisotropy in surface conductivity due to anisotropic superstructures, resistance caused by a monatomic step, and metal-insulator transition in surface states of superstructures, have been derived from these methods [4–7]. However there is no systematic report about surface conductivity which depends on the superstructures with the same adsorbate on a crystal surface.

Extensive studies have been carried out on Au-induced surface superstructures on Si(111). Various superstructures, \(5 \times 2, \alpha\sqrt{3} \times \sqrt{3}, 2\sqrt{2}T \times 2\sqrt{2}T, 6 \times 6, \) and \(\beta\sqrt{3} \times \sqrt{3}\) have been found by reflection-high-energy electron diffraction (RHEED) and other techniques [8]. The formation of these superstructures depends on the Au coverage and delicate thermal treatments. Studies by STM and PES have clarified that the surface with about one monolayer Au coverage exhibits a reversible two-dimensional (2D) glass-crystal transition between the \(\beta\sqrt{3} \times \sqrt{3}\) (glass phase) and \(6 \times 6\) (crystal) phase just by controlling thermal treatments [9–12]. The \(6 \times 6\) phase has a long-range order in atomic arrangement, while the \(\beta\sqrt{3} \times \sqrt{3}\) phase does not because of high-density out-of-phase domain boundaries. The PES studies have also clarified that the \(6 \times 6\) and the \(\beta\sqrt{3} \times \sqrt{3}\) have semiconducting surface states, while the \(\alpha\sqrt{3} \times \sqrt{3}\) has a metallic surface-state band [11]. These properties should be closely related to electrical conduction through the surface states.

In the present study, we have performed in-situ surface electrical conductivity measurements on these surface superstructures of Au/Si(111) in ultrahigh vacuum (UHV) by using the micro-four-point probe method in variable-temperature mode ranging from room temperature (RT) to 130 K. We have also performed STM and RHEED observation at RT and 80 K in a separate chamber to confirm the surface structures. We have succeeded in detecting the clear difference of conductivity among the superstructures. The conductivity of \(6 \times 6\) (crystal phase) is higher than that of the \(\beta\sqrt{3} \times \sqrt{3}\) (glass phase) in the whole temperature range used, showing the influence of long-range order of atomic arrangement in surface superstructures. The temperature dependence of conductivity for both of the \(6 \times 6\) and \(\beta\sqrt{3} \times \sqrt{3}\) is semiconducting, while the \(\alpha\sqrt{3} \times \sqrt{3}\) is metallic. This result is understood in terms of character of the surface-state bands revealed by previous PES studies. These results clearly show that the electrical conductivity is very sensitive to the atomic structure in the topmost atomic layer on a surface.

II. EXPERIMENTAL AND ANALYSIS METHODS

A. Apparatus and Sample

The experiments were performed using a home-made UHV chamber for the micro-four-point-probe measurement [1–3] and a commercial STM chamber, both of which were equipped with RHEED system with base pressure of \(1 \times 10^{-10}\) Torr. The probe spacing in the four-point probe was 20 \(\mu\)m. The Si(111) substrate was \(n\)-type (P-doped) with the resistivity of 2-15 \(\Omega\)cm at RT. First, to obtain the clean Si(111)-\(7 \times 7\) superstructure, the Si sample was rinsed in ethanol and acetone repeatedly in an ultrasonic bath, then flashed repeatedly at 1200 \(^\circ\)C by Joule heating in UHV. RHEED was used to confirm the superstructures in this study. Then Au atoms were de-
posited from an Alumina-coated-tungsten basket by Joule heating to the Si substrate which was kept at 700°C. The deposition rate of Au was estimated by a phase diagram of Au/Si(111) superstructures [8]. After the deposition of proper amounts of Au, the sample was annealed for 5 minutes at 650°C, then cooled down to RT. The cooling rate and Au coverage are crucial to prepare the aimed surface superstructures. We chose 13 different surfaces as samples for conductivity measurements, as summarized in Table I.

Table I shows various combinations of the Au coverage (0.44, 0.52, 0.76, 0.84, 0.92, 1.0 ML) and cooling rate (Quench, Standard, Slow) in preparing the surface superstructures. The cooling rates of “Quench”, “Standard” and “Slow” mean that it takes few seconds, 1 minute and 30 minutes to decrease the sample heating current down to zero, respectively. The last column “1.0 ML (RT depo.)” means that Au was deposited on the clean 7×7 substrate at RT and no post annealing after that. Downward arrows indicate the annealing at 650°C for 5 minutes and subsequent quench cooling. An upward arrow indicates the annealing and subsequent slow cooling. ‘A’ and ‘B’ in 1.0 ML column indicates different runs of sample preparations under the same conditions. The surface structures of these 13 surfaces were identified by RHEED. Some of them showed the same RHEED patterns. The 13 surfaces are called hereafter by the name listed in Table I; 5×2(0.44), 5×2(0.52), α-√3×√3(α), α-√3×√3(β), 2√2Γ, 6×6(0.92), 6×6(α), 6×6(β), 6×6(γ), β-√3×√3(0.92), β-√3×√3(α), β-√3×√3(β), and “1×1”, respectively.

For these 13 samples, we measured the surface conductivity using the micro-four-point-probe at temperatures ranging from RT to 130 K [3]. We observed RHEED patterns in situ after the conductivity measurement to confirm the surface structures. The sample temperature below RT was measured by two pairs of gold-iron/chromel thermocouples which were attached close to the sample. We also took STM images in a separate chamber for several surfaces in Table I at RT and 80 K.

### B. Analysis

In general, the measured conductivity (σ_{exp}) includes three contributions,

\[ \sigma_{exp} = \sigma_{SS} + \sigma_{SC} + \sigma_B, \]

where σ_{SS} is the conductivity of surface superstructure which we aim to measure, σ_{SC} is the conductivity of surface space-charge layer caused by band bending below the surface, and σ_B is the conductivity of substrate bulk. When we use the micro-four-point probe [1–3], a large fraction of the measuring current flows near the surface region so that the measurement becomes sensitive to σ_{SS} and σ_{SC}. Furthermore, it is known from previous PES measurements that the Au-induced surface superstructures on Si(111) cause band bending to form p-type (hole-accumulation) layer below the surface [12, 13]. Then, since we chose an n-type substrate, a p/n junction is formed between the surface region and bulk. Because the p/n junction prevents the measuring current from penetrating into the bulk region, we can ignore the σ_B contribution [7]. Therefore the measured conductivity is a sum of σ_{SS} and σ_{SC} only. Furthermore, the thicknesses of the surface state and surface space-charge layer are much thinner than the probe spacing (20 µm), the sample can be regarded as a 2D conductor.

The σ_{SC} can be calculated theoretically if we know the Fermi-level positions at the surface and in bulk. The Fermi energy (E_F) in the bulk is known from the resistivity, and the surface E_F with respect to the valence-band-maximum energy at surface (E_{SVB}) of each surface superstructure is already measured by core-level PES [12–14].

The four probes are arranged on a line on the sample surface (linear four-point method). The outer pair of probes are for current sources, and the inner pair of probes are voltage probes. By using the four-probe method, in general, the contact resistance between the probes and sample is avoided. For infinitely large 2D conductors, the measured resistance (R_{exp}) and sheet conductivity (σ_{exp}[S/C]) have a relation,

\[ \sigma_{exp} = \frac{\ln 2}{\pi R_{exp}}. \]

This is independent of probe spacing, which is a unique character of 2D conductors [2]. This relation can be derived by solving Poisson’s equation for a infinitely large 2D conductor [15]. Since, in this experiment, a fixed probe spacing of 20 µm was used, the sample (about 4 × 15mm² in lateral size) can be regarded as infinitely large compared with the probe spacing.

### III. RESULTS AND DISCUSSION

#### A. Structures

Figure 1 displays RHEED patterns taken from various Au/Si(111) superstructures, (a) 5×2, (b) α-√3×√3, (c) 2√2Γ×2√2Γ, (d) 6×6, (e) β-√3×√3, and (f) “1×1”.

Figure 2 displays the corresponding STM images, (a) 5×2, (b)(c) α-√3×√3, (d) 6×6, (e) β-√3×√3, and (f) “1×1”.

Figure 1(a) is a nearly single-domain 5×2 pattern. The clear five-fold superspots are observed, and the arrow in (a) indicates streaks of two-fold periodicity. Figure 2(a) is a STM image of the 5×2 surface. Stripes correspond to the five-fold periodicity. The two-fold periodicity along the stripes is hardly seen in this STM image. The bright protrusions are Si adatoms [16]. We confirmed that the 5×2 RHEED pattern appeared in the range of 0.44 ML and 0.52 ML coverage of Au.

Figure 1(b) is a RHEED pattern of the α-√3×√3 which has streaky fractional reflections, meaning that the √3×√3 domains are small in size. Figures 2(b) and (c) are STM images of the α-√3×√3. The bright zigzag structure in (b)(filled-state image) is out-of-phase domain boundaries between different √3×√3 domains, called domain walls [10]. In Fig. 2(c)(empty-state image), bright √3×√3 regions are easily seen, and they are divided into small domains by dark domain walls.

Figure 1(c) is a RHEED pattern of the 2√2Γ×2√2Γ surface superstructure. The 13 fraction-order Laue rings are observed between the zero-th and the first Laue zones,
FIG. 1: RHEED patterns taken from various Au/Si(111) superstructures. (a) 5 × 2, (b) α-√3 × √3, (c) 2√21 × 2√21, (d) 6 × 6, (e) β-√3 × √3, and (f) “1 × 1”. The electron beam energy was 15 keV. (a), (c) and (f) were taken at RT. (b),(d)and (e) were taken at 10 K. But, all patterns basically showed no changes by cooling below RT. The arrow in (a) indicate two-fold periodicity.

which are the feature of the 2√21 × 2√21 superstructure. The STM image of this surface shows the periodic domain wall configuration [17]. Figure 1(d) clearly displays the 6 × 6 pattern. Figure 2(d) has a long-range order in corrugation which is consistent with the clear RHEED pattern in Fig. 1(d). We have confirmed that this surface is formed at more than 0.9 ML of Au.

Figure 1(e) is a RHEED pattern of the β-√3 × √3, having sharper fractional-order spots compared with those of the α-√3 × √3 pattern in (b). Figure 2(e) is the corresponding STM image. It is not easy to find the √3 × √3 periodicity in this image. But, the Fourier transformation of this STM image clearly shows sharp √3 × √3 spots and ring-like structure around them, which is consistent with the previous high-resolution electron diffraction study [18]. With more than 0.9 ML coverage of Au, the β-√3 × √3 is formed by quench cooling, while the 6 × 6 is formed by slow cooling. Both surfaces are transformed into an apparently same √3 × √3 structure above 500°C, which have a long-range periodicity without domain walls. The β-√3 × √3 and 6 × 6 structures are reversibly prepared via the high-temperature √3 × √3 phase [10] by controlling the cooling rate from the high-temperature phase. According to the PES and STM studies, the local atomic structure in these two surfaces is the same, but only the long-range order is different. It means that the β-√3 × √3 and 6 × 6 can be identified as a glass phase and a crystal phase in 2D, respectively [10–12].

Figure 1(f) is a RHEED pattern from the surface which is made by 1 ML deposition of Au at RT on the 7×7 clean surface and no additional heat treatments. This pattern has only fundamental spots, which means no superstructure formed. We call this surface by ”1 × 1” in this paper. Figure 2(f) is the STM image of this surface, in which islands (probably Au or Au silicide) are connected to each other by obeying the 7×7 periodicity on the substrate.

The RHEED patterns of 5 × 2, α-√3 × √3, 6 × 6 and β-√3 × √3 did not change by cooling from RT to 130 K. Also no qualitative difference was found in STM images between at liquid N2 temperature (Figs. 2(b)-(f)) and at RT [10], which was consistent with the RHEED results. So we can say that there are no structural transitions in the Au/Si(111) surface systems in this temperature range.

This feature is important to investigate the temperature dependence of surface conductivity, and makes it easy to interpret the results.

B. Electrical Conduction

Figure 3 shows sheet conductivities (σₑₑₑₑ) measured as a function of temperature ranging from 130 K to RT for the respective surfaces listed in Table 1. The filled and open circles in Fig. 3(a) are for the 5 × 2 surfaces with 0.44 and 0.52 ML of Au, respectively, which show basi-
cally no difference. The $5 \times 2$ is known to be formed in the Au coverage range between 0.44 ML and 0.52 ML. Figure 3(a) means that the conductivity is not sensitive to the variance of Au coverage in the range where the same superstructure is fabricated.

In Fig. 3(b), the filled and open circles indicate the conductivity of $\alpha\sqrt{3}(q)$ and $\alpha\sqrt{3}(s)$, respectively. The $\alpha\sqrt{3}(s)$ was fabricated by slow cooling and $\alpha\sqrt{3}(q)$ was made from $\alpha\sqrt{3}(s)$ by annealing it at 650°C for 5 min and subsequent slow cooling. But the $\alpha\sqrt{3}(s)$ and $\alpha\sqrt{3}(q)$ show no difference in conductivity. This is reasonable by recalling that the RHEED patterns and STM images are the same for the two surfaces.

In Fig. 3(d), the filled circle, open circle and open triangle indicate the conductivities of $6 \times 6(\alpha)$, $6 \times 6(\beta)$ and $6 \times 6(0.92)$, respectively. The former two have a 1.0 ML Au coverage and the latter has a 0.92 ML coverage of Au. Again, there is no essential difference among the three $6 \times 6$ surfaces.

In Fig. 3(e), the filled circle, open circle and open triangle indicate the conductivities of $\beta\sqrt{3}(A)$, $\beta\sqrt{3}(B)$ and $\beta\sqrt{3}(0.92)$, respectively. They were fabricated from the $6 \times 6(\alpha)$, $6 \times 6(\beta)$ and $6 \times 6(0.92)$, respectively, by annealing them at 650°C and subsequent quench cooling, of which sequences are indicated by solid arrows between Figs. 3(d) and (e). In Fig. 3(d), the crosses indicate the conductivity of $6 \times 6(\beta')$ which was fabricated from the $\beta\sqrt{3}(B)$ by annealing it and subsequent slow cooling, as indicated by a dotted arrow between (d) and (e). In other words, three measurements for the $6 \times 6(\beta) \rightarrow \beta\sqrt{3}(B) \rightarrow 6 \times 6(\beta')$ were done in series with the same sample by using the reversible phase transition between the $6 \times 6$ and $\beta\sqrt{3}$ superstructures. The data points for different samples in (d) and (e) indicate good reproducibility and insensitive to the Au coverage (0.92 ML and 1.0 ML).

As mentioned in the previous section, the $\sigma_{\text{exp}}$ includes the contributions from $\sigma_{SC}$ and $\sigma_{SS}$ in principle. The solid curves in Fig. 3 show the contribution of $\sigma_{SC}$ calculated theoretically from the energy difference $\Delta E \equiv E_F - E_{SVBM}$, where $E_{SVBM}$ is the energy of valence-band maximum at surface [14]. We chose the following values as $\Delta E$ from the literature (though the values are scattered from literature to literature), 0.13 eV and 0.3 eV for the $5 \times 2$, 0.065 eV and 0.13 eV for the $\alpha\sqrt{3} \times \sqrt{3}$, 0.31 eV and 0.34 eV for the $6 \times 6$, 0.31 eV, and 0.3 eV for the $\beta\sqrt{3} \times \sqrt{3}$ [12, 13]. The contributions of $\sigma_{SC}$ are not shown for Figs. 3(a), (d) and (e) because they are smaller than 0.1% of $\sigma_{\text{exp}}$. There is no report for the $"1 \times 1"$ and $2\sqrt{21} \times 2\sqrt{21}$, so the contributions are not shown in Figs. 3(c) and (f). Since $\sigma_{\text{exp}}$ is larger than the $\sigma_{SC}$ for all surfaces, there must be a contribution of $\sigma_{SS}$. Especially the $\sigma_{SS}$ is dominant for the $6 \times 6$ and $\beta\sqrt{3} \times \sqrt{3}$ surfaces.

From Figs. 3(a), (d) and (e), slight differences in Au coverage and heat treatments do not affect the conduc-

![FIG. 2: STM images taken from various Au/Si(111) superstructures. Au coverage ($\theta$), tip bias ($V_{\text{TIP}}$), tunneling current ($I$) and temperature ($T$) at STM observation for each surface are (a) $5 \times 2$, $\theta=0.5$ ML, $V_{\text{TIP}}=2.0$ V, $I=0.26$ nA, $T=RT$; (b) $\alpha\sqrt{3} \times \sqrt{3}$, $\theta=0.82$ ML, $V_{\text{TIP}}=-1.0$ V, $I=0.3$ nA, $T=80$ K; (c) $\alpha\sqrt{3} \times \sqrt{3}$, $\theta=0.82$ ML, $V_{\text{TIP}}=2.0$ V, $I=0.3$ nA, $T=80$ K; (d) $6 \times 6$, $\theta=1.1$ ML, $V_{\text{TIP}}=1.5$ V, $I=0.4$ nA, $T=80$ K; (e) $\beta\sqrt{3} \times \sqrt{3}$, $\theta=1.1$ ML, $V_{\text{TIP}}=1.5$ V, $I=0.4$ nA, $T=80$ K; (f) "1 \times 1", $\theta=1.0$ ML, $V_{\text{TIP}}=-2.0$ V, $I=0.2$ nA, $T=80$ K. All images are filled-state topographic STM images except for (c) (empty-state current image). All images are in the same scale and crystal orientation, and the common scale is indicated under (c). The unit cell is indicated in each image. The unit cell in (f) is $7 \times 7$ unit.]

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http://www.sssj.org/ejssnt (J-Stage: http://ejssnt.jstage.jst.go.jp)
activity as long as the surface superstructures are the same. In other words, the surface conductivity is determined by the surface superstructures themselves. Thus we have confirmed good reproducibility in conductivity measurements.

In Fig. 4 the measured sheet conductivities of the respective surfaces are plotted on a single graph as a function of temperature ranging from 130 K to RT for the respective surfaces listed in Table I. (a) Filled circles and open circles are for the $5 \times 2_{(0,44)}$ and $5 \times 2_{(0,52)}$, respectively. (b) Filled circles and open circles are for the $\alpha-\sqrt{3} \times \sqrt{3}$ and $\alpha-\sqrt{3} \times \sqrt{3}$, respectively. (c) Filled circles for the $2\sqrt{21}$, (d) Filled circles, open circles, crosses and open triangles are for the $6 \times 6_{(A)}, 6 \times 6_{(B)}, 6 \times 6_{(B')}$, and $6 \times 6_{(0,92)}$, respectively. (e) Filled circles, open circles and open triangles are for the $\beta-\sqrt{3} \times \sqrt{3}$, $\beta-\sqrt{3} \times \sqrt{3}$ and $\beta-\sqrt{3} \times \sqrt{3}$, respectively. (f) Filled circles are for the "1 x 1". Solid curves indicate the conductivity through the surface space-charge layer $\sigma_{SC}$. Solid arrows between (d) and (e) indicate annealing and subsequent slow cooling, and dotted arrow indicates annealing and subsequent quench cooling in the sample preparation.

IV. CONCLUSION

We have performed the surface electrical conduction measurements for various surface superstructures of Au/Si(111); $5 \times 2, \alpha-\sqrt{3} \times \sqrt{3}, 2\sqrt{21} \times 2\sqrt{21}, 6 \times 6, \beta-\sqrt{3} \times \sqrt{3}$ and 1 ML Au film, at temperatures ranging from RT to 130 K by the micro-four-point probe method. We have succeeded in detecting the clear difference in surface electrical conductivity among these superstructures with good reproducibility. The conductivity of $6 \times 6$ (crystal phase) is higher than that of the $\beta-\sqrt{3} \times \sqrt{3}$ (glass phase) at the whole temperature range. The temperature dependence of $\alpha-\sqrt{3} \times \sqrt{3}$ is metallic, while those of the $6 \times 6$ and $\beta-\sqrt{3} \times \sqrt{3}$ are semiconducting, which is consistent with previous PES results. The most notable point is that the surface conductivity is quite different even if the Au
coverage is the same; the conductivity is very sensitive to the surface superstructures (and their surface states). The transport mechanism will be clarified through quantitative analysis of the temperature dependences with aid of information about surface electronic structures from PES.

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