Epitaxial Growth of Ag on Si(111)-4×1-In Surface
Studied by RHEED, STM, and Electrical Resistance Measurements

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We investigated the initial stages of Ag growth on a Si(111)-4×1-In surface at room temperature (RT) and low temperatures (LT, 70–120K) in situ by reflection-high-energy electron diffraction (RHEED), scanning tunnelling microscopy (STM) and electrical resistance measurements in ultrahigh vacuum, together with on a Si(111)-7×7 clean surface for comparison. It has been revealed that the 4×1-In surface actually acts as a highly anisotropic template for Ag growth at RT, but not at LT. The Ag islands grown on the wetting layers were elongated along the 4×1-stripe at RT, while they were isotropic and round in shape at LT. This is due to a difference in migration ability of the arriving Ag adatoms. The anisotropic growth has been found to affect the formation of percolation paths for electrical conduction among the Ag islands; the growth conditions (deposition rate and temperature) do not affect the critical coverage for percolation so much on the 4×1-In surface, compared with in the case of isotropic growth on the 7×7 surface. The initial deposition of submonolayer-thick wetting layers has been found to induce some structural changes of the substrate surface; at RT, the 4×1-In structure changes into a 4×2’ structure, while the 8×2’-In structure at LT changes into a 4×1 structure. The former change induces a resistance increase, while the latter induces a resistance drop. These changes may be caused by the induced changes in surface states and band bending beneath the surface. [DOI: 10.1380/ejssnt.2003.72]

Keywords: Scanning tunneling microscopy, Reflection high-energy electron diffraction(RHEED), Electrical transport (conductivity, resistivity, mobility, etc.), Epitaxy, Percolation, Silver, Indium, Silicon

I. INTRODUCTION

The formation of Ag/Si(111) interfaces has been a subject of quite large number of studies, resulting in the huge accumulation of knowledge, which in turn triggers new investigations from different points of view[1]. The lattice mismatch between Ag and Si crystals amounts to 25%, and the resultant interface energy is very large. The lattice strain and large interface energy greatly affect the Ag growth, eventually leading to formation of three-dimensional (3D) islands with widely varying island density and size depending on the growth conditions. Moreover, the high mobility of Ag atoms on the Si surface at room temperature (RT) is known to accelerate the 3D island growth. When Ag is deposited onto a clean Si(111)-7×7 surface, the growth proceeds in the Stranski-Krastanov mode at RT (3D island growth on a wetting layer) [2–5], but in quasi-layer-by-layer growth mode at low temperatures (LT) [4–8]. The growth mode and morphology of Ag thin films on Si(111) substrates also sensitively depend on the adsorbate termination of the surface [2, 4–11]. On H, Ag, and Sb-terminated surfaces, a Volmer-Weber growth mode (3D island growth without wetting layers) is observed with different island density and size distributions. The layer-by-layer growth mode occurs under some favorable conditions at LT (see Table 1).

For achieving continuous and atomically flat metal films on Si surfaces, several ways are employed such as choosing special regimes of the growth conditions and an introduction of surfactants to modify the surface energy and nucleation density[12, 13]. In general, when island spacing is larger than some value, the islands will nucleate a second layer on top of them before coalescence, giving multiplayer (3D) growth [14]. Therefore, the reduced island spacing, or high nucleation density of islands, can enforce layer-by-layer growth, which is realized at low temperatures or by introducing surfactants.

Additionally, a novel ‘electronic growth’ mechanism has recently been developed for metal/semiconductor systems, in which the energy contribution of the quantized electrons confined in the metal overlayer can actually affect the morphology of the growing film, prevailing over the strain, surface and interface energies [15–19].

Here we investigated Ag growth on a Si(111)-4×1-In surface at RT and LT (70-120K) by in-situ reflection-high-energy electron diffraction (RHEED), scanning tunneling microscopy (STM) and electrical resistance measurements in ultrahigh vacuum (UHV). Because of the highly anisotropic nature in atomic arrangement and surface electronic structure of the 4×1-In surface [20–23], we expected anisotropic epitaxial growth of metals on this surface. It will be interesting to compare the results with the growth on an isotropic substrate of Si(111)-7×7 clean surface. Furthermore, the 4×1-In structure is known to transform into a 8×2’-In phase below ~130K accompanied by charge-density-wave (CDW) formation due to Peierls instability of its quasi-one-dimensional metallic surface state[23]. It is also interesting to investigate the influence of such a phase transition upon the initial stages of epitaxial growth on it.

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TABLE I: Ag growth modes on clean and adsorbate-terminated Si(111) surfaces.

| Temperature | Termination | Growth mode          | Techniques                        | References |
|-------------|-------------|----------------------|-----------------------------------|------------|
| RT          | Clean       | Stranski-Krastanov   | STM, LEED, Ion scattering         | [2–5]      |
| LT          | Clean       | Layer-by-layer       | RHEED, STM                        | [4–8]      |
| 300°C       | H           | Volmer-Weber         | STM                               | [9]        |
| RT          | $\sqrt{3} \times \sqrt{3}$-Ag | Volmer-Weber | RHEED | [4] |
| LT          | $\sqrt{3} \times \sqrt{3}$-Ag | Volmer-Weber | RHEED | [4, 10] |
| RT          | Sb(1ML)     | Stranski-Krastanov   | RHEED, STM                        | Present work* |
| LT          | 4 $\times$ 1-In | Stranski-Krastanov   | RHEED, STM                        | Present work* |

* The size and shape of islands are different depending on the substrate temperature.

FIG. 1: RHEED patterns taken during the Ag deposition on the 4$\times$1-In surface at RT (a, b, c) and at 100K on the 8$\times$2$\times$2-In surface (d, e, f). (a), (d) - the initial surface before the Ag deposition. (b), (e) - after 0.3 ML of Ag deposition on (a) and (d), respectively. (c) and (f) - after 10 and 7 ML of Ag deposition on (a) and (d), respectively. (c') RHEED pattern (c) with supplementary lines showing streaks.

II. EXPERIMENTAL

The experiments were carried out in a UHV chamber with a base pressure of about $2 \times 10^{-10}$ Torr, equipped with LT-STM (UNISOKU USM 501 type) and RHEED systems. The substrates used were B-doped Si(111) wafers with a resistivity of 0.001 $\Omega$cm. Atomically clean Si(111) surfaces were prepared in situ by direct Joule heating at 1500 K after outgassing at 800 K for several hours. After this treatment, a sharp $7 \times 7$-RHEED pattern was observed at RT, and the STM images corresponded to a well-ordered Si(111)-7$\times$7 clean surface. The Si(111)-4 $\times$ 1-In surface was produced by In deposition onto the 7 $\times$ 7 substrate kept at 700 K by direct current heating. Then, the sample was cooled down to RT and transferred to the cold STM stage. The Ag atoms were additionally deposited on top of the surface at RT on the RHEED sample stage or at 70 K on the LT-STM stage. For STM observations, electrochemically etched W tips were employed after cleaning by in-situ heating in UHV. All STM images shown here were taken in constant-height mode with slow feedback.

For electrical resistance measurements, another UHV chamber was used, equipped with RHEED and a sample holder for cooling by liquid nitrogen. An n-type Si(111) wafer of 50-100 $\Omega$ cm resistivity was used. The surface preparations were the same as for STM observations mentioned above. During Ag deposition under isothermal conditions at LT or RT, the electrical resistance of the wafer was measured as a voltage drop between a pair of Mo wire contacts (0.3 mm in diameter) about 5 mm apart each other, pressed on the front face of the wafer, with constant current fed through the substrate clamps at the both ends [24]. The quality of the electrical contact be-
The Ag coverage and deposition rate were determined by deposition duration with constant evaporation rates, assuming that 1 ML of Ag was needed for the complete conversion in RHEED pattern from the 7 × 7 to the Si(111)−√3 × √3-Ag structures. The RHEED pattern was continuously observed during the Ag deposition at elevated temperatures without interruption, so that we could accurately determine the completion point and coverage for the structural conversion. The reproducibility in determining Ag coverage by this method was estimated to be around 10%.

### III. RESULTS AND DISCUSSION

#### A. RHEED observations

First we monitored variations in RHEED pattern during Ag deposition on the 4×1-In surface (Fig. 1). RHEED patterns of the initial surface are shown in Fig. 1(a) (4×1-In at RT) and Fig. 1(d) (8×2′-In at 100 K) with the same In coverage. At the early stage of Ag deposition at RT (Fig. 1(b)), the characteristic streaks appeared, indicating epitaxial Ag islands growing. At the same time, the fundamental and fractional-order spots of the 4×1-In reconstruction were weakened. At coverage above 5 ML, all diffraction spots of the 4×1-In superstructure disappeared and the streaks corresponding to epitaxial Ag(111) films were only observed (Fig. 1(c)). The streaks are without intensity modulation along them, indicating that the Ag film have atomically smooth and flat tops. Ag films grown on the 7×7 clean surface at RT showed similar streaks, but with some intensity modulation along the streaks[1], meaning more 3D shapes of Ag islands. So we can say that the 4×1-In substrate is more suitable than the 7×7 surface for making atomically flat 2D films at RT. We also notice that streaks in Fig. 1(c) split into two and connect the neighboring fundamental spots in particular directions on the 1st Laue zone (as schematically shown in (c′)), meaning some modulation in the direction parallel to the 4×1 stripes. In other words, there is some anisotropy in structure of Ag films. This is not observed on the 7×7 substrate[1].

During Ag deposition at 100 K, the initial 8×2′ (Fig. 1(d)) structure transformed to a 4×1 structure immediately at the beginning of Ag deposition (with less than 0.1 ML coverage), as reported in Ref. [25]. With further Ag deposition, the RHEED patterns looked slightly different from those at RT. Though the fundamental and fractional-order spots of the 4×1-In structure were weakened, the characteristic streaks seen in Fig. 1(b) were hardly observed in Fig. 1(e). With Ag coverage above 3 ML, streaks similar to those in Fig. 1(c) were also observed in Fig. 1(f), indicating the epitaxial growth of Ag films. But the streaks are much broader and have strong intensity modulations along them, meaning a tendency of 3D islanding with smaller sizes, compared with the RT case in Fig. 1(c). This pattern is very similar to that of Ag films grown on the 7×7 surface at 170K [4]. Since the RHEED intensity oscillation was observed during Ag deposition at the cold 7×7 surface[4], the growth proceeded similarly also on the 4×1-In substrate, that is, in a quasi-layer-by-layer mode on top of Ag islands, in spite of 3D shapes of islands. Thus, there seems no notable difference in growth structure between the 7×7 and 4×1-In at LT, while the island shape is different at RT between the two substrates as mentioned above.

In summary, these RHEED observations suggest that the Ag film grows at RT in a form of 2D flat islands with some anisotropy in structure, while it grows at LT in more 3D and isotropic way. In comparison with on the 7×7 surface, the Ag films are more atomically flat on the 4×1-In substrate at the growth of RT, while the film structure is similar to each other on the both surfaces at LT.

Figure 2 shows intensity decays of the fundamental (-1, 0) and fractional-order diffraction spots (-3/4, 0) of the 4×1-In structure during the Ag deposition at RT (a) and at 100 K (b). It can be seen that the fractional-order spots fade with the fundamental spots with similar rates up to about 0.6 ML coverage at both temperatures. This means that the 4×1-In reconstruction is preserved at the initial stages of Ag deposition. On the other hand, if the surface superstructure is disrupted, the fractional-order spots should, in general, fade more rapidly than the fundamental spot. This phenomenon is actually observed with more than 0.6 ML deposition of Ag, especially at 100 K (Fig. 2(b)). This indicates that the 4×1-In reconstruction is disrupted partially with more than 0.6 ML Ag deposition. Such a tendency is more pronounced at

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**FIG. 2:** Decays in intensity of the fundamental and fractional-order spots in RHEED during Ag deposition on the 4×1-In structure (a) at RT and (b) at about 100 K.
FIG. 3: A series of filled-state STM images during Ag deposition on the Si(111)-4×1-In surface at RT. (a) The initial 4×1-In surface (225×225Å², $V_t=1.3$ V). (b) Ag coverage $\Theta_{Ag}=0.2$ ML, (c) $\Theta_{Ag}=0.2$ ML (a different place on the same sample). (d) $\Theta_{Ag}=3$ ML, (e) $\Theta_{Ag}=12$ ML (450×450Å², $V_t=2$ V).

100 K than at RT. This may be because the area fraction on the surface covered by Ag films is smaller at RT than at LT due to a smaller number density of islands at RT, which originates from a longer migration distance of Ag adatoms. Since the migration distance is comparable to the island separation, the migration distance can be estimated from the STM images shown below to be about 20 nm at RT and 2 nm at LT. From the results in Fig. 2, we speculate that a wetting layer of about 0.6 ML thick is first formed by Ag deposition, and then Ag islands begin to nucleate on top of it, meaning a Stranski-Krastanov (S-K) growth mode at both temperatures. The wetting layer does not destroy the 4×1 structure of the substrate, while the Ag islands grown on top of it destroy the 4×1 structure beneath them.

B. STM observations

Figure 3 shows filled-state STM images of the 4×1-In surface after different doses of Ag adsorption at RT. The initial 4×1-In surface is shown in Fig. 3(a). At Ag cov-
average about 0.3 ML, the two types of islands are formed; elongate (Fig. 3(b)) and round ones (Fig. 3(c)); the elongate islands are the majority. The characteristic streaks in Fig. 1(b) correspond to these islands. With closely looking at the image, we notice that a double-periodicity modulation along the stripes of the 4×1-In substrate is seen at some areas, e.g., indicated by a white arrow in Fig. 3(b). This is a ‘defect-induced 4×2’ structure’ as reported in Ref. [26]. The area fraction of the 4×2 domains is so small that RHEED does not detect it. Its details will be reported elsewhere [27]. At Ag coverage about 3 ML (Fig. 3(d)), the islands begin to coalescence. Figure 3(e) is a typical STM image for a thick Ag film, representing the continuous and smooth films grown with elongated domains along the 4×1 stripes. An interesting point is that we can see the 4×1-stripes on top of the Ag films, as indicated by a white arrow in Fig. 3(e). This can be a quantum size effect similar to that observed for Pb films[28]. This anisotropic modulation corresponds to the faint streaks connecting particular fundamental spots in Fig. 1(c). There are some atomic and/or electronic anisotropies in the grown Ag film, which originates from the substrate.

After cooling the substrate down to 100 K, the 4×1-In
C. Electrical resistance measurements

Figure 5(a) shows the change in resistance (normalized with the initial resistance $R_0$) of the Si wafer during Ag deposition on the clean Si(111)-7×7 surface at RT, with various deposition rates. At the initial stage of Ag adsorption, the resistance is hardly changed. When Ag coverage exceeds a critical coverage $\theta_{cr}$ (roughly indicated by arrows in Fig. 5(a)), the resistance begins to drop rapidly. The $\theta_{cr}$ decreases as the Ag deposition rate is increased, which is consistent with the previous report [29]. This phenomenon is associated with a process of island coalescence and formation of percolation paths for electrical conduction. With higher deposition rates, the probability of island nucleation is higher, resulting in a larger number density of islands with smaller sizes. As the island density is higher, we need a smaller coverage for making percolation paths by island coalescing. The $\theta_{cr}$ thus strongly depend on the deposition rates, ranging from 1.8 ML (at 3 ML/min) to 6 ML (at 0.3 ML/min).

In the case of Ag growth on the Si(111)-4×1-In surface at RT (Fig. 5(c)), we also observed similar behaviors in resistance change as for the 7×7 case; the initial slow decrease in resistance, followed by rapid drop after the critical coverage $\theta_{cr}$ (indicated by arrows). But the $\theta_{cr}$ did not change so much by changing the deposition rate.
This can be attributed to the anisotropic island growth; elongate islands along the 4×1 stripes may make the percolation paths more easily in the direction of stripes, and the $\theta_{cr}$ is more insensitive to the island density than in the case of isotropic growth of islands on the 7×7 surface. We of course need theoretical supports for this speculation. In general, the percolation threshold could be sensitively influenced by island density and size in two-dimensional case, while it would be less sensitive in one-dimensional case, because of limited freedom of path formation.

Another feature that was different from on the 7×7 substrate was observed at the very beginning of the deposition; the resistance increases by 5~10% with ~0.2 ML deposition in Fig. 5(c), which is not observed for the 7×7 surface in Fig. 5(a). This slight change in resistance corresponds to the formation of the defect-induced 4×2’ structure as mentioned in Fig. 3(b). This is a wetting layer formation before Ag islands growing.

Figures 5(b) and (d) show the changes in resistance during Ag depositions on the clean 7×7 and 8×2’-In surfaces at about 100 K, respectively. In both cases, the $\theta_{cr}$ was around 1 ML and did not depend on the deposition rate. At LT the thermal mobility of arriving Ag atoms is suppressed, so that the nucleation of islands occurs with higher densities and smaller island sizes, leading to coalescence with a smaller coverage than at RT. This does not depend on the deposition rate so much because the nucleation density is so high at LT that the migration of arriving Ag atoms does not play significant roles.

Thus the critical coverage for formation of percolation paths depend on the growth condition as well as the initial surface reconstruction structures, as summarized in Fig. 6. At RT, the growth structure depends on the growth condition in different ways between on the two surfaces because of the different kinetic properties of arriving atoms. At LT, the growth structure does not depend on the growth condition or the initial substrate surface structures, because the kinetics plays negligible roles.

On the 8×2’-In substrate at LT (Fig. 5(d)), one can notice a slight drop in resistance at the beginning of Ag deposition. This change corresponds to a change in RHEED pattern from the 8×2’ to 4×1. This phenomenon contrasts with the initial slight increase in resistance at RT in Fig. 5(c), accompanied by the structural change from the 4×1 to 4×2’. A small amount of Ag adsorption induces characteristic changes in atomic and electronic structures of the substrate depending on the temperature.

**IV. CONCLUSION**

*In-situ* observations and measurements presented here have revealed that the Si(111)-4×1-In surface actually acts as a highly anisotropic template for Ag growth at RT, but not at LT. At RT, after formation of a submonolayer-thick wetting layer, three-dimensional (3D) islands grew with large separations among them, a majority of which was elongated along the 4×1-stripes of the substrate. At LT where the 4×1 transformed into a 8×2’ structure on the substrate, such an anisotropic island growth was suppressed. This is due to a difference in migration ability of the Ag adatoms; the adatoms migrate along the 4×1-stripes preferentially at RT, while the migration is suppressed at LT. Continuous Ag films were formed with increasing deposition, which were composed of large islands (10~30 nm in size) with flat tops of Ag(111) face at RT, while much smaller islands (a few nm in diameter) at LT.

When the Ag coverage exceeded critical values $\theta_{cr}$, the resistance decreased rapidly both at RT and LT, which corresponded to the island coalescence, leading to the formation of percolation paths for electrical conduction. The anisotropic growth on the 4×1-In surface was revealed to affect the formation of percolation paths for electrical conduction among the Ag islands. The $\theta_{cr}$ at RT was 2~6 ML, depending on the Ag deposition rate (0.3~3 ML/min) on the 7×7 surface, while it was less sensitive to the deposition rate on the 4×1-In substrate. The $\theta_{cr}$ was reduced to 1 ML at 100 K irrespective of the deposition rate and the substrate surface structure. This is because the migration of arriving Ag adatoms play negligible roles in nucleation of islands at LT.

The initial deposition of submonolayer-thick wetting layers was found to induce some structural changes of the substrate surface; at RT, the 4×1-In structure changed into a 4×2’ structure, while the 8×2’-In structure at LT changed into a 4×1 structure. The former change induced a resistance increase, while the latter made the resistance drop. These changes may be caused by the adsorption-induced changes in surface states and band bending beneath the surface due to charge transfer between the adatoms and substrate, which should be confirmed by photoemission spectroscopy studies in the future.

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**Fig. 6:** Critical coverages $\theta_{cr}$ for steep drop in resistance versus deposition rate of Ag on the 7×7 and 4×1 (or 8×2’) surfaces at RT and LT.
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