Beam Rocking Auger Electron Spectroscopy of a Si(111)$\sqrt{3} \times \sqrt{3}$-Ag Surface

Yoshimi Horio, a, † Hitoshi Nakahara, b Junji Yuhara, c Yuji Takakuwa d

a Department of Electrical and Electronic Engineering, Daido University, 5-10 Takiharu-cho, Minami-ku, Nagoya 457-8530, Japan
b Department of Energy Engineering, Nagoya University, Furo-cho, Chikusa-ku, Nagoya 464-8603, Japan
c Department of Applied Physics, Nagoya University, Furo-cho, Chikusa-ku, Nagoya 464-8603, Japan
d Institute of Multidisciplinary Research for Advanced Materials, Tohoku University, 2-1-1 Katahira, Aoba-ku, Sendai 980-8577, Japan

† Corresponding author: horio@daido-it.ac.jp

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The Auger electron intensities of Si-LVV and Ag-MNN from a Si(111)$7 \times 7$ and a Si(111)$\sqrt{3} \times \sqrt{3}$-Ag surfaces were measured for varying glancing angles of the incident electron beam in reflection high-energy electron diffraction, known as the beam rocking Auger electron spectroscopy (BRAES). The energy of incident electron beams used was 10 and 5 keV. The intensity anomalies observed in these BRAES profiles on the surface wave resonance condition were more pronounced for the 5-keV incident beam than for the 10-keV beam. It was found from BRAES profiles of the Si(111)$\sqrt{3} \times \sqrt{3}$-Ag surface by the 5-keV incident beam that the intensity enhancements of Si-LVV differed from those of Ag-MNN at the [11\_2] incidence, but were similar at the [10\_1] incidence. This result can be interpreted qualitatively by the wave field periodicity parallel to the surface.

Keywords Reflection high-energy electron diffraction; Si(111)$\sqrt{3} \times \sqrt{3}$-Ag; Auger electron spectroscopy

I. INTRODUCTION

Research on electronic device materials using two-dimensional (2D) thin films has been intensively pursued to achieve higher functionality. Recently, the formation and physical properties of not only graphene [1], but also silicene [2], germanene [3], and stanene [4], which are group IV 2D materials, have been investigated. Adsorbed surfaces such as an ultrathin film are also important in this regard, and these structures have been investigated via scanning tunneling microscopy and reflection diffraction.

In particular, the reflection high-energy electron diffraction (RHEED) is widely used as a surface structure analysis method because it is simple and can be performed in situ. With this method, we can not only study the conventional reflection diffraction electrons but also the secondary electrons emitted from the surface. We have measured the incident glancing angle dependence of the Auger electron intensity, known as the beam rocking Auger electron spectroscopy (BRAES). The primary feature of the BRAES is that an increase in the Auger intensity is observed when the incident condition satisfies the surface wave resonance (SWR) conditions. For several surface structures [5–12], we have investigated the correlation between the Auger intensity anomaly and the wave field (incident electron density) distribution formed by incident electrons around the crystal surface. However, because the incident electron energy of RHEED is too high for Auger excitation, inelastically scattered electrons generated in the crystal are also involved in Auger excitation. As the wave field distribution obtained from dynamical calculations is for elastically scattered electrons, the correlation between the Auger intensity anomaly and the wave field is expected to be weakened. In this study, we present experimental BRAES profiles for Si(111)$7 \times 7$ and Si(111)$\sqrt{3} \times \sqrt{3}$-Ag surfaces. These results were obtained by reducing the incident electron energy from 10 to 5 keV and thus, reducing the contribution of inelastically scattered electrons.
II. SURFACE WAVE RESONANCE CONDITION

A SWR condition appears when the specular reflection is near a condition for the emergence of a non-specular beam; for example, both sides of $m$ and $\tilde{m}$ beams. On this condition, these beams cannot overcome the barrier of the inner potential and cannot leave the sample surface. The range of the glancing angle, $\theta$, at which the SWR condition is satisfied is calculated by the following formula. The SWR condition is associated with the 2D rods of $m$ and $\tilde{m}$.

$$\tan^{-1} \left( \frac{B_m}{K_{0T}} \right) < \theta < \tan^{-1} \left( \frac{B_m}{K_{0T}} \right),$$

where $U = 2meV_0/h^2$ and $m$, $e$, and $h$ are conventional physical constants. Mean inner potential $V_0$ is 12 V for Si crystal and 21 V for Ag crystal, which are deduced from Doyle and Turner’s table [13] with an empirical correction of 86.2%. The SWR region width is derived from the mean inner potential of the Si crystal for the $7 \times 7$ surface and the tentative Ag crystal for the $\sqrt{3} \times \sqrt{3}$-Ag surface. $K_0$ is the surface parallel component of the incident wave vector in a vacuum. The 2D reciprocal lattice vector (rod vector), $B_m$, is normal to $K_{0T}$ and parallel to the sample surface. Its length, $B_m$, is $2\pi/d_m$, where $d_m$ is the spacing of $m$-th lattice plane of the crystal surface. The lower and higher limits of the glancing angle $\theta$ for the SWR region are called the internal threshold “$\theta_{IT}$” and the vacuum threshold “$\theta_{VT}$”, respectively. In this study, the SWR region associated with $1\bar{1}$ and $1\bar{1}$ rods ($2\bar{0}$ and $2\bar{0}$ rods) is focused on for the $[1\bar{1}2\_1]$ incidence (for the $[10\bar{1}]$ incidence).

For example, calculated RHEED patterns of the Si(111) $7 \times 7$ surface are shown in Figure 1. The SWR regions are represented by the blue parabola areas. In the case of the $[1\bar{1}2\_]$ incidence of the Si(111) $7 \times 7$ surface [or Si(111)$\sqrt{3} \times \sqrt{3}$-Ag surface], the SWR region associated with $1\bar{1}$ and $1\bar{1}$ rods is from $\theta_{VT} = 3.1^\circ$ (or $2.5^\circ$) to $\theta_{IT} = 3.7^\circ$ for the 10-keV incident beam as shown in Figure 1(a) and from $\theta_{VT} = 4.3^\circ$ (or $3.6^\circ$) to $\theta_{IT} = 5.2^\circ$ for the 5-keV incident beam as shown in Figure 1(b). In the case of the $[1\bar{1}0\_1]$ incidence of the Si(111) $7 \times 7$ surface [or Si(111)$\sqrt{3} \times \sqrt{3}$-Ag surface], the SWR region associated with the $2\bar{0}$ and $2\bar{0}$ rods is from $\theta_{VT} = 3.7^\circ$ (or $3.3^\circ$) to $\theta_{IT} = 4.2^\circ$ for the 10-keV incident beam as shown in Figure 1(c) and from $\theta_{VT} = 5.3^\circ$ (or $4.7^\circ$) to $\theta_{IT} = 6.0^\circ$ for the 5-keV incident beam as shown in Figure 1(d).

Figure 1: Kinematic simulations of RHEED patterns of the Si(111)$7 \times 7$ surface at a glancing angle of $4^\circ$ involving diffraction spots (red circles), Kikuchi lines (green curves) and the SWR regions (blue curves). (a) and (b) are patterns at the $[1\bar{1}2\_]$ incidence. (c) and (d) are patterns at the $[10\bar{1}]$ incidence. (a) and (c) are obtained by the 10-keV incident beam, and (b) and (d) by the 5-keV beam.
III. EXPERIMENTAL

In this study, a RHEED apparatus equipped with a cylindrical mirror analyzer (CMA), as shown in Figure 2, was used. The CMA was set above the sample surface with a working distance of approximately 10 mm and was used for the detection of Auger electrons emitted from the sample surface by the RHEED incident electron beam. The base pressure was $4 \times 10^{-7}$ Pa and the pressure was maintained at an order of $10^{-7}$ Pa, during the deposition of Ag. The sample holder was rotated around the axis normal to the sample surface to adjust the azimuth angle.

This apparatus included a mechanical tilting system for the electron gun. Thus, the glancing angle of the incident electron beam was finely altered. Recently, a new system using a stepping motor was constructed to enable smoother changes in the glancing angle without vibration that allowed using a stepping motor was constructed to enable smoother changes in the glancing angle without vibration that allowed the BRAES profiles to be measured with relatively less noise.

The sample used was a mirror-polished n-type Si(111) single crystal with resistivity of $8-10$ $\Omega$ cm and dimensions of $15 \times 4 \times 0.5$ mm$^2$. A clean Si(111) $7 \times 7$ surface was obtained by repeated flash annealing at approximately 1200°C with current heating in an ultra-high vacuum apparatus. A Si (111)$\sqrt{3} \times \sqrt{3}$-Ag surface was obtained by evaporating 1 ML of Ag on the Si(111)$7 \times 7$ substrate surface while the substrate was maintained at approximately 450°C. The evaporator was a tungsten filament hooked with Ag wires, and 1 ML of Ag was evaporated for approximately 20 s by current heating.

In the BRAES measurements, the incident electron beam irradiates a point, which must match the focal point of the CMA above the sample surface. A slight shift in the irradiation point causes a large change in the Auger intensity, thus the focus of the incident electron beam, obtained by an electromagnetic lens, was relaxed slightly, resulting in a slight expansion of the irradiation area.

The BRAES measurements were performed by repeatedly scanning the energy range that included the Auger peak-to-peak energy in the energy differential spectrum, while changing the glancing angle at a rate of $2^\circ$ min$^{-1}$. The scanning energy ranges were 13 eV from 83 to 96 eV for the Si-LVV Auger peak and 20 eV from 348 to 368 eV for the Ag-MNN Auger peak. The measured data were stored in a data logger, and the peak-to-peak intensity was automatically extracted by a program developed in-house.

The rocking curve of the specular spot was obtained from stocked RHEED patterns by the KSA-400 system (k-Space Associates). The integrated intensity of the specular spot was automatically measured from the stocked patterns. Since the spot position gradually shifts for changing the glancing angle, it was estimated by linear interpolation between the initial and the final positions.

IV. RESULTS AND DISCUSSION

Rocking curves of the specular reflection spot and the BRAES profiles of Si-LVV and Ag-MNN were obtained by using a conventional 10-keV and a low 5-keV incident electron beams. Figures 3 and 4 are the experimental results at [112] and [10$\bar{1}$] incident directions, respectively. The bottom two graphs and the top three graphs in both Figures 3 and 4 were obtained from the Si(111) $7 \times 7$ and the Si(111)$\sqrt{3} \times \sqrt{3}$-Ag surfaces, respectively. Figures 3(a) and 3(b) [or Figures 4(a) and 4(b)] show the results by the 10-keV and 5-keV incident beams, respectively. The top bars in Figures 3 and 4 show the indices of Bragg reflections for the Si(111) surface considering the mean inner potential of Si. Shadows in Figures 3 and 4 represent the glancing angle range which satisfies the SWR condition. The lower and the higher angle limits of the SWR region correspond to the internal threshold, $\theta_{IT}$, and the vacuum threshold, $\theta_{VT}$, respectively. At the [112] incidence of Figure 3, attention was paid to the SWR region associated with the $1 \bar{1} \bar{1}$ and $\bar{1} \bar{1} \bar{1}$ rods and at the [10$\bar{1}$] incidence of Figure 4, attention was paid to the SWR region associated with the $2 \bar{0}$ and $\bar{2} \bar{0}$ rods. The former is denoted as SWR$_{1 \bar{1} \bar{1}}$ and the latter as SWR$_{2 \bar{0}}$. These SWR regions were calculated using the mean inner potential of 12 V for the Si(111) $7 \times 7$ surface and the tentative 21 V of the Ag crystal for the Si(111)$\sqrt{3} \times \sqrt{3}$-Ag surface. All the results were measured under room temperature. In Figures 3 and 4, the bottom graph shows the rocking curve of the specular spot and the next graph above it shows the BRAES profile of Si-LVV. These two curves were obtained from the $7 \times 7$ surface. The top two graphs show the BRAES profiles of Ag-MNN and Si-LVV, and the next graph below it shows the rocking curve of the specular spot. These three curves were obtained from the $\sqrt{3} \times \sqrt{3}$-Ag surface.

In Figure 3(a) of the 10-keV incident beam at the [112] incidence, enhancements of Si-LVV Auger intensity are observed at the both ends ($\theta_{IT}$ and $\theta_{VT}$) of the SWR$_{1 \bar{1} \bar{1}}$ region as indicated by arrows A and B. The intensity anomaly is reconfirmed to be the same as the previous study.
results \cite{5}. For the $\sqrt{3} \times \sqrt{3}$-Ag surface, the intensity anomaly of Si-LVV becomes slightly weaker and that of Ag-MNN is not clear due to the relatively high noise. On the other hand, the rocking curve of the $\sqrt{3} \times \sqrt{3}$-Ag surface is modulated from that of $7 \times 7$ surface.

Figure 3(b) shows the corresponding experimental results obtained by the 5-keV incident beam. Because the magnitude of the incident wave vector changes short, the glancing angle of the SWR$_{\pm1\pm1}$ condition shifts to higher. The glancing angles at which the Si-LVV intensity anomalies arise are shifted to higher angles as indicated by arrows C and D compared with the case of the 10-keV incident beam. It should be noted that these enhancements by the 5-keV incident beam are stronger than those by the 10-keV incident beam. The reason considered is that the ratio of inelastically scattered electrons involved in Auger excitation is reduced in the case of the 5-keV incident beam compared with the 10-keV case. The Si-LVV Auger electrons become excited if the incident beam has energy of at least approximately 100 eV that corresponds to the ionization energy of L-shell electrons. Therefore, both elastically scattered electrons and inelastically scattered electrons are involved in the Auger excitation. The contribution of inelastically scattered electrons, which are diffusely distributed, weakens the correlation between the intensity anomalies in the BRAES profile and the diffraction conditions. The ratio of inelastically scattered electrons involved in Auger excitation to elastically scattered electrons is smaller for the 5-keV incident beam compared to the 10-keV one. Thus, the intensity enhancement is more pronounced. Since the excitation probability of the L-shell electrons is higher for the 5-keV incident beam than for the 10-keV one, the Auger sensitivity is higher for 5 keV than for 10 keV, which reduces the background noise level of the BRAES profiles and produces clear profiles, as shown in Figure 3(b).

The intensity anomalies of Si-LVV for the $\sqrt{3} \times \sqrt{3}$-Ag surface differ from those for the $7 \times 7$ surface but correspond to the SWR$_{\pm1\pm1}$ region of the $\sqrt{3} \times \sqrt{3}$-Ag surface as shown by arrows E and F in Figure 3(b). Furthermore, the intensity anomalies in the BRAES profiles of Ag-MNN are different from those of Si-LVV as shown by arrows G, H, and I. These anomalies in the BRAES profiles are considered to be attributed to the wave field distribution. The rocking curve of the $\sqrt{3} \times \sqrt{3}$-Ag surface is also modulated from that of the $7 \times 7$ surface.

In Figure 4(a) of the 10-keV incident beam at $[10\bar{1}]$ incidence, enhancements of the Si-LVV Auger intensity are also observed in the SWR$_{\pm2\pm0}$ region of $7 \times 7$ surface as indicated by arrows A and B. For the $\sqrt{3} \times \sqrt{3}$-Ag surface, the interval of the two anomalies of Si-LVV becomes slightly wider as shown by arrows C and D, and that of Ag-MNN is further wider as shown by arrows E and F in the SWR$_{\pm2\pm0}$ region. On the other hand, the rocking curve of $\sqrt{3} \times \sqrt{3}$-Ag surface is modulated from that of the $7 \times 7$ surface. Especially, the split of the 444 Bragg peak is characteristic.

Figure 4(b) shows the corresponding experimental results obtained by the 5-keV incident beam. The glancing angles at
which the Si-LVV intensity anomalies arise for the $7 \times 7$ surface are shifted to higher angles as indicated by arrows G and H comparing with arrows A and B of the 10-keV incident beam case in Figure 4(a). The intensity anomalies of Si-LVV for the $\sqrt{3} \times \sqrt{3}$-Ag surface are similar to those for the $7 \times 7$ surface as shown by arrows J and K. Additional small peak seems to appear at the end ($\theta_{IT}$) of the SWR$_{\pm 20}$ region as shown by arrow I. Intensity anomalies of Ag-MNN are similar to those of Si-LVV for the $\sqrt{3} \times \sqrt{3}$-Ag surface as shown by arrows L, M, and N. The rocking curve of the $\sqrt{3} \times \sqrt{3}$-Ag surface is also modulated from that of the $7 \times 7$ surface such as the split of 444 Bragg peak.

Figures 3 and 4 show that the BRAES profile obtained with the 5-keV incident beam has a lower noise level than the 10-keV beam. It is considered that the excitation probability of the inner shell electrons of the target atom is higher for the 5-keV beam. Also, the 5-keV incident beam is more sensitive to the surface because it has a shallower penetration depth. If the incident beam has higher energy than the binding energy of the inner shell electrons of the target atom, then both the elastic scattered electrons and inelastic scattered electrons excite the Auger electrons. The proportion of elastic scattered electrons among the electrons that can excite Auger electrons is higher for the 5-keV incident beam. For this reason, it is considered that the correlation between the Auger intensity anomaly and diffraction condition such as the SWR condition is more pronounced for the 5-keV incident beam than for the 10-keV beam.

The behavior of the Auger intensity anomalies by the 5-keV incident beam is generally similar to that by the 10-keV beam, but there are some differences. This may be mainly due to the difference in the penetration depth. It is interesting for the $\sqrt{3} \times \sqrt{3}$-Ag surface structure to compare the BRAES profiles of the 5-keV incident beam in the [112] and [101] directions. It should be noted that Si-LVV profile shows a dip at the angle corresponding to the peak H in Ag-MNN profile in Figure 3(b). This is contrasted to the profiles in Figure 4(b) in which three peaks I, J, and K appear at the same angles with L, M, and N, respectively. It can be seen that the Si-LVV and Ag-MNN Auger intensity anomalies are different in the [112] incidence, but similar in the [101] incidence.

For the Si(111)$\sqrt{3} \times \sqrt{3}$-Ag surface, Takahasi et al. presented a honeycomb chained-triangle (HCT) structure [14, 15] by X-ray diffraction (XRD). Aizawa et al. predicted existence of an inequivalent-triangle (IET) structure [16] by the first-principles calculation, which is an energetically stable structure. The HCT structure has $p31m$ symmetry with a rotation angle 0° of the topmost Ag triangle, while the IET structure has $p3$ symmetry with a rotation angle of ±6°. There were several reports [16–18] that IET to the HCT phase transition was an order-disorder transition depending on the temperature. Nakahara et al. reported that the IET structure transfers to a rotating honeycomb chained-triangle (r-HCT) structure [19] at temperatures above 150 K by using the RHEED rocking curve. The r-HCT structure has

Figure 4: Experimental results by the 10-keV (a) and 5-keV (b) incident beams. All these results were measured at the [101] incidence. In both graphs of (a) and (b), the bottom two dot curves are the rocking curve (black curve) of the specular spot and the BRAES profile of Si-LVV (red curve) for the Si(111)$7 \times 7$ surface. The top three dot curves are the rocking curve (black curve) of the specular spot and two BRAES profiles of Si-LVV (red curve) and Ag-MNN (blue curve) for the Si(111)$\sqrt{3} \times \sqrt{3}$-Ag surface.
$p31m$ symmetry in which Ag triangles thermally rotate between two stable rotation angles of $+6^\circ$ and $-6^\circ$. For qualitative interpretation of the Auger intensity anomalies, the r-HCT model is assumed as that the Ag triangles rotate randomly in the range of $\pm6^\circ$.

Both the plan and the side views of the r-HCT surface structure are shown for [112] and [101] incidences in Figures 5(a) and 5(b), respectively. Adsorbed Ag atoms and substrate Si atoms are indicated by blue and red circles, respectively. Here, characteristic three moments of the surface structure whose rotation angles of the Ag triangles are $0^\circ$ and $\pm6^\circ$ are overlaid. The difference in height between the Ag layer and the top Si layer is $0.57$ Å $[19]$, which are rather close. Therefore, attention is paid to the periodicities of the Ag and the top Si atomic rows parallel to the incident direction. The wave fields on the SWR conditions associated to a pair of $\tilde{1}$ and $\tilde{1}$ rods and a pair of $2 \bar{0}$ and $2 \bar{0}$ rods are also drawn by wavy green shadows in Figures 5(a) and 5(b), respectively. White and green areas show high and low densities of the incident electrons, which correspond to “on” and “off” of the wave field. For a strict discussion, these “on” and “off” situations are also represented by straight and broken lines, respectively. The periods of the wave fields in the SWR $[\tilde{1}\tilde{1}]$ [Figure 5(a)] and the SWR $[20 \bar{0}]$ [Figure 5(b)] are 1.92 and 1.66 Å, respectively. The latter period is half of that in the SWR $[1\bar{1}0]$ because of the second order condition. A rhombus in the plan view means the $\sqrt{3} \times \sqrt{3}$ unit mesh. Here, three representative Ag and Si atoms, which are denoted as A–C and D–F, respectively, are paid attention. The centers of these atoms are indicated by small white points.

In the case of the [112] incidence, when the wave field rides on the Ag atom denoted by D, the other two Ag atoms denoted by E and F are in the neutral situation of the wave field as shown in Figure 5(a). Then, total Auger intensity of Ag is considered to be increased. At that time, the wave field rides on the Si atom denoted by A, however, the other two Si atoms denoted by B and C of the Si trimer are in the off situation of the wave field as shown in Figure 5(a). Then, total Auger intensity of Si is considered to be decreased. This may make a difference in the Auger intensity anomalies of Si-LVV and Ag-MNN.

In the case of the [101] incidence, on the other hand, when the wave field rides on the Ag atoms denoted by D and F, the other Ag atoms denoted by E is off situation of the wave field as shown in Figure 5(b). Then total Auger intensity of Ag is considered to be increased. At that time, the wave field rides on the Si atom denoted by C and the other two Si atoms denoted by A and B are in the neutral situation of the wave field as shown in Figure 5(b). Then, total Auger intensity of Si is considered to be increased. It is considered that Si-LVV and Ag-MNN show the same

![Figure 5](image-url): Top and side views of r-HCT model for Si(111) $\sqrt{3} \times \sqrt{3}$-Ag surface; (a) [112] and (b) [101] incidences. Three characteristic moments of the surface structure whose rotation angles of Ag triangles are $0^\circ$ and $\pm6^\circ$ are overlaid. The periods of the wave fields on the SWR conditions attributed to a pair of $\tilde{1}$ and $\tilde{1}$ rods and a pair of $2 \bar{0}$ and $2 \bar{0}$ rods are also inserted as wavy green shadows in (a) and (b), respectively. Three representative Ag and Si atoms are denoted as A–C and D–F, respectively. Straight and broken lines indicate “on” and “off” situations of the wave field, respectively.
intensity anomaly.

This study focuses on the SWR due to the integer-order rods. Even if the out-of-phase domains which are shift by $\pm 1/3$ of the $\sqrt{3} \times \sqrt{3}$ unit cell exist, the intensity of the wave field is modified but the basic periodicity such as "on" and "off" situations may be maintained. Furthermore, since the coherent length of 5 keV incident electron beam is shorter than that of 10 keV, the influence of the domain boundary is considered to be weak. A detailed discussion requires the distribution of the wave field obtained from dynamical calculations, which is currently underway.

V. SUMMARY

By improving the system for tilting the electron gun, the glancing angle of the incident electron beam can be refined, thus improving the experimental accuracy of the BRAES. To more clearly elucidate the intensity anomaly appearing in the BRAES profile, the energy of the incident electron beam was lowered from 10 to 5 keV, which reduced the ratio of inelastically scattered electrons involved in Auger excitation with respect to the elastically scattered electrons. In this experiment, BRAES profiles from the Si(111)7 $\times$ 7 and Si (111)$\sqrt{3} \times \sqrt{3}$-Ag surfaces were measured. In general, Auger intensity anomalies appeared at both ends, $\theta_{V1}$ and $\theta_{V2}$, of the SWR region. To reduce the ratio of the inelastic scattering component that contributes to the BRAES profile, BRAES measurement using the 5-keV incident beam was performed. As a result, an increase in the Auger intensity anomalies was observed with noise reduction. It was found for the BRAES profile of the $\sqrt{3} \times \sqrt{3}$-Ag surface using the 5-keV incident beam that the Auger intensity anomalies of Si-LVV and Ag-MNN were different at the $\sqrt{3} \times \sqrt{3}$-Ag surfaces were measured. In general, Auger intensity anomalies appeared at both ends, $\theta_{V1}$ and $\theta_{V2}$, of the SWR region. To reduce the ratio of the inelastic scattering component that contributes to the BRAES profile, BRAES measurement using the 5-keV incident beam was performed. As a result, an increase in the Auger intensity anomalies was observed with noise reduction. It was found for the BRAES profile of the $\sqrt{3} \times \sqrt{3}$-Ag surface using the 5-keV incident beam that the Auger intensity anomalies of Si-LVV and Ag-MNN were different at the [112] incidence, and were similar at the [101] incidence. Such features can be qualitatively explained by the period of the wave field in the direction parallel to the surface based on the r-HCT model. In the quantitative discussion, the dynamical calculation of the wave field is necessary. A comparison between the BRAES profiles and the wave field calculated based on the surface structure will be further reported.

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