Reflection High Energy Electron Diffraction (RHEED)

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1 Introduction

A crystal surface (or interface with foreign substances) is generally known to have an atomic arrangement different from that of an internal crystal structure. The properties of substances depend more largely on the arrangement (or crystal structure) of constituent elements than on constituent elements *per se*. The recent high-degree integration of electronic devices has greatly increased the importance of knowing the exact crystal structures of surfaces and interfaces or controlling them into desired structures. Additionally, nanostructures such as carbon nanotubes can be considered as substances consisting entirely of surfaces, thus showing the close relationship between nanotechnology and surface science.

This experiment is intended to investigate the surface structures of a crystal (Si(111) surface) placed in an ultra-high vacuum using reflection high-energy electron diffraction (RHEED). Also to be studied are the structures of the crystal surfaces with other substances (Ag/Au) deposited on it.

2 Outline of the Experiments

- You should bring your own **calculator, ruler, compass** for data analysis.
- Students will be divided into two groups, Group A and Group B (unless there are only 5 or less persons).
- We have only one set of laboratory equipment. Hence, the two groups cannot conduct the experiment simultaneously. The groups must work in good cooperation with each other.
- Each group must be well prepared for the discussion so that they can give confident answers to questions on the obtained experimental data. It is also important to understand results of the other group for report preparation.
- For precautions and experiment procedures, refer to the experiment procedure manual issued separately.

The 5-session experiment plan is as shown below.

- Session 1
  - Lecture 1 (ultra-high vacuum, crystal surface structure, reciprocal lattice)
  - Observation of existing Si (111) surface
  - Temperature calibration of Si substrate
- Session 2
  - Observation of clean Si (111) surface
  - Substrate temperature dependence of clean surface
  - Lecture 2 (electron diffraction, RHEED pattern analysis technique)
  - Determination of camera length and RHEED pattern analysis
- Session 3
  - Observation of Ag-deposited Si (111) surface (room temperature, 500°C)
  - Observation of Au-deposited Si (111) surface (room temperature, 500°C)
- Session 4
  - Interim discussion on experiments so far
- Session 5
  - Free experiments
    The combination of deposits, the substrate temperature, and other parameters are changed for each group to investigate changes to the surface.

3 Lectures

3.1 Basics of ultra-high vacuum

3.1.1 Definition and significance of ultra-high vacuum

Under ordinary temperatures and pressures, the number of gaseous molecules per mole (22.4 liters) is the Avogadro's number N_A of 6.02×10^{23} molecules. Accordingly, under atmospheric pressure (1.013×10^5 Pa), a gas exists with a molecular density as high as 2.7×10^{19} cm^{-3}.

With gaseous molecules in the environment, a crystal surface is constantly collided with gaseous molecules. Consequently, such gaseous molecules will be adsorbed onto or react with the surface. To correctly grasp the state of a crystal surface, the crystal surface must be clean. In other words, a vacuum environment is required in which other molecules will not reach the surface. The vacuum is divided by level into low vacuum (1×10^2 to 1×10^{-2} Pa), high vacuum (1×10^{-2} to 1×10^{-6} Pa, ultra-high vacuum (UHV: 1×10^{-6} to 1×10^{-10} Pa), and extreme-high vacuum (XHV: 1×10^{-10} Pa or less).[1] As explained in the next section, a vacuum level approximately equal to a ultra-high vacuum is necessary to keep a surface sufficiently clean during an experiment. The degree of vacuum relatively easily achievable with the currently available laboratory scale technology is limited to around 1×10^{-9} Pa. To achieve extreme-high vacuum, special equipment and technology are required.

3.1.2 Number of molecules hitting a wall under vacuum

The number n of molecules hitting a wall under a vacuum per unit area per unit time for when the vacuum pressure is P is given as:

$$n = \frac{P}{\sqrt{2\pi mkT}}, \tag{1}$$

where m is a mass of a molecule, k is the Boltzmann constant, and T is temperature. When the gaseous molecules are nitrogen molecules, $m = 4.64 \times 10^{-26}$ kg. Hence, when the vacuum pressure is 10^{-4} Pa, for example, the approximate number n of nitrogen molecules colliding into the wall will be determined to be 3×10^{14} cm^{-2} from $k = 1.38 \times 10^{-23}$ J/K and $T = 300$ K. Here, let us take an Au(100) surface for example. The surface contains two atoms per unit lattice with a lattice constant of 0.4 nm, which means that there are $2/(4 \times 10^{-8})^2$ to 1.3×10^{15} atoms in the surface. This shows that, when the vacuum pressure is 10^{-4} Pa, the number of particles reaching the surface per unit time per unit area is approximately on the same order of the number of atoms present in the surface. When all collided molecules are adsorbed onto the surface (adsorption probability of 100%), one to two molecular layers will be formed on the surface per second under a vacuum pressure of 10^{-4} Pa.

To obtain a clean surface, it is necessary to ensure that the surface is not covered with adsorbates for at least the duration of the experiment. Considering that n is proportional to P, it is expected that the surface can be kept clean under a vacuum pressure of 10^{-8} Pa for approximately 1 hour. If the residual gas consists of molecules of low reactivity (*i.e.*, low adsorption probability), the surface can be kept clean for a further longer time. The vacuum used for this time of experiment is approximately 10^{-6} Pa and this is sufficient to maintain a normal surface for several hours, considering the adsorption probability of the residual gas (consisting primarily of hydrogen). Precautions must be taken, however, because the surface may be rapidly contaminated under conditions contributory to reaction, for example, during heating of the sample.

3.2 Crystal surfaces and reconstructed structures

3.2.1 Structure of clean crystal surface

A typical bulk-truncated surface structure appears as shown in Fig. 1(a), which shows the face (001) of a diamond structure as viewed from the cross-sectional direction. Such a structure is, however, disadvantageous in terms of energy efficiency due to the excessively many unpaired electrons (dangling bonds) left behind. Therefore, in an actual surface, adjacent dangling bonds are bonded to each other, as shown in Fig. 1(b), to form a more stable structure (note, however, that this gives rise to strain in bonds with increased strain energy). This kind of surface structure is called a reconstructed surface structure. When surface reconstruction occurs, the original surface (before reconstruction) having a periodicity of a will have a periodicity of $2a$.

When viewed from above, this surface will appear as shown in Fig. 2. As is clear from the figure, new unit cells are formed, each having a periodicity of $2a$ in the x direction and the original periodicity of a in the y direction. Let us call such a surface structure as 2×1 structure. What should be noted here is that the reference length (1×1) is that of two-dimensional unit cells (dotted-lined squares in Fig. 2) in the surface as bulk-truncated but not that of three-dimensional unit cells of a crystal structure.

Fig. 3 shows the structural model of the 7×7-DAS (dimer-adatom-stacking-fault) surface known as a clean Si(111) surface (one to be used in this experiment). This structure is a unique structure in that not only the atoms in the first layer but the third layer of the surface are significantly shifted from their bulk sites.

3.2.2 Substance adsorption and surface structure

When a different kind of a material is adsorbed onto a crystal surface, the crystal surface will turn into a reconstructed structure different from a clean surface. Fig. 4 shows a structural model of a Ga-adsorbed Si(111) surface. In this structure, the unit cell of the reconstructed structure has a length $\sqrt{3}$ times greater than that of the primitive lattice of the Si(111) surface and the orientation

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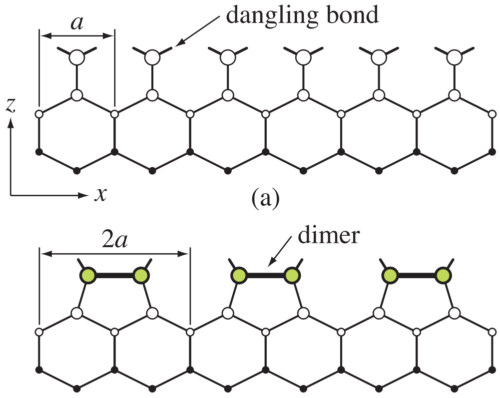
[1] The unit of vacuum for official use is Pa (pascal) in the SI unit system. Among vacuum scientists, however, 'Torr' (derived from Torricelli; same as 'mmHg') and 'mbar' is often used instead. The atmospheric pressure is 1.01325×10^5 Pa, 760 Torr and 1013.25 mbar in the respective unit. The relationship between Torr and Pa is approximately expressed as 1 Torr = 133 Pa. Note that these units of vacuum may be used intermixed in the subsequent lectures and references used therefor.

of the unit cell is rotated by 30 degrees. Therefore, this kind of surface structure is called $\sqrt{3}\times\sqrt{3}$-R30°. In addition to this, there are various structures resulting from adsorption of foreign substances. There are cases where the substrate element is substituted with an adsorbed element, causing the adsorbate to be embedded into the inside of the crystal. Moreover, many surface structures are semi-stable and their surfaces often allow simultaneous co-existence of multiple structures.



(a)

(b)

Figure 1: (a) Cross-sectional view of the (001) face of the diamond structure as bulk-truncated; and (b) cross-sectional view of the (001) face of the diamond structure reconstructed by dimer formation. To make it easier to see the correspondence with Fig. 2, the circles closer to the surface are rendered larger.

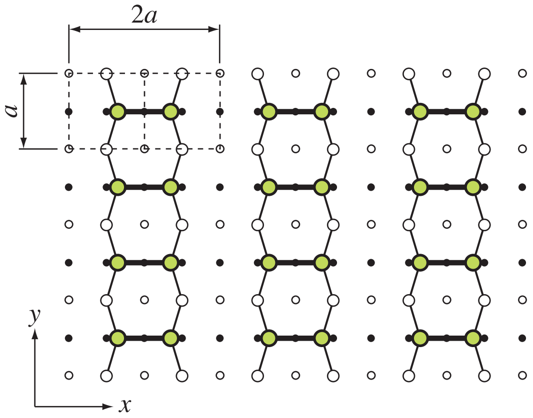


Figure 2: Top view of the (001) face of the reconstructed diamond structure. The circles closer to the surface are rendered larger. Only the bonds in the first layer of the surface are shown. The dotted-lined squares in the figure represent the surface unit cells (1×1 unit cells) as bulk-truncated. The figure shows that the unit cells have a periodicity of $2a$ in the x direction and a periodicity of a in the y direction.

3.3 Electron diffraction

3.3.1 Diffraction conditions and reciprocal space/reciprocal lattice

When a single-wavelength plane wave is incident into the periodic structure, scattered waves in a particular direction will result. This is a phenomenon caused by wave diffraction and interference. As shown in Fig. 5, when the wave is scattered, the path difference must be of integral multiple of the wavelength to ensure agreement in phase between waves scattered from different reflection planes. In other words, the Bragg condition given by Eq. 2 must be met:

$$2d\sin\theta = n\lambda, \tag{2}$$

where λ is the wavelength of the wave, d is the spacing to the reflection plane, θ is the grazing angle (supplement of the angle of incidence) to the reflection plane, and n is an arbitrary integer. When considering the Bragg condition, note that it is tacitly assumed that the incident wave and the diffracted wave have the same wavelength, that the crystal surface is an infinite periodic continuum, and that multiple scattering (re-scattering of diffracted waves) is ignored.

The above expression of the Bragg condition is suitable for the intuitive understanding of diffraction phenomena but is unsuitable for application to complex systems. Accordingly, the concept of reciprocal space is introduced here to facilitate the mathematical handling of complex systems. A reciprocal space is a periodic space and mathematically corresponds to the Fourier-transformed world

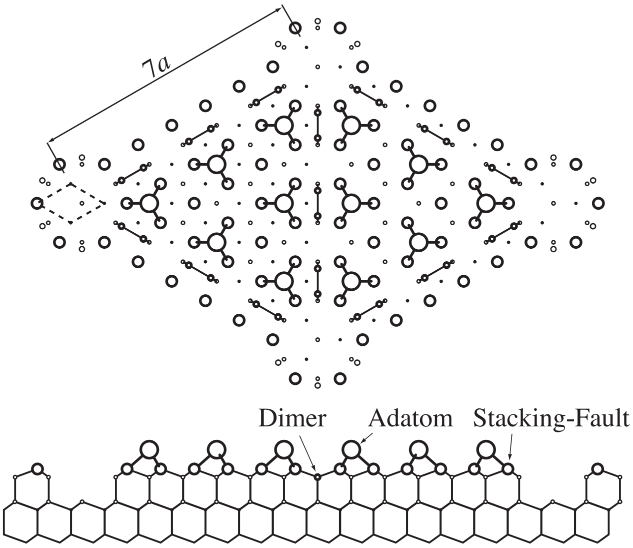


Figure 3: Structural model of Si(111) surface. The above figure shows a top view of the surface, while the below figure shows a side view of the surface. This structure contains dimmers, adatoms, and stacking-faults and hence is called a 7×7-DAS structure. (K. Takayanagi et al., Surface Science **164** (1985) p.367.)

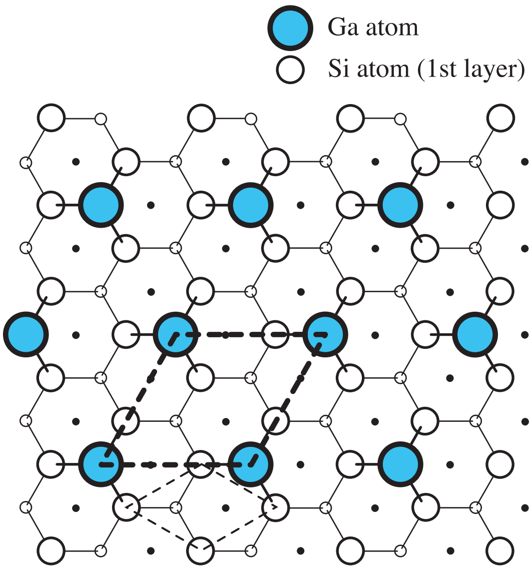


Figure 4: Structural model of Si(111) surface adsorbed with 1/3 monolayer of Ga. The rhombus in thin dotted lines in the figure is a primitive lattice of the Si(111) surface, while the rhombus in thick dotted lines is a Ga-adsorbed unit lattice. (J. Nogami et al., J. Vac. Sci. Technol. B **6** (1988) p.1479.)

of a real space (one in reality). Let us take sounds (1-dimensional wave) for example. Many may know that audio equipment, etc., uses frequency axes to represent sounds. Fourier transformation is the process of breaking down sound waves into frequencies and a Fourier transformed space corresponds to an acoustic reciprocal space. In a 3-dimensional reciprocal space, one periodicity of a certain direction is given as a vector, *i.e.*, in case of a crystal having periodic structure in real space, reciprocal lattice vector $\boldsymbol{a}^*$ can be defined as $|\boldsymbol{a}^*| = 2\pi/d$, where d is a lattice spacing in real space and the vector direction corresponds to normal direction of the lattice planes. While for a plane wave with wavelength λ, the wave number vector $\boldsymbol{k}$ can be determined as $|\boldsymbol{k}| = 2\pi/\lambda$ and its direction is oriented to the wave travel direction. Using these concept of reciprocal space, the Bragg condition of Eq. 2 can be rewritten as follows.

$$2\frac{2\pi}{|\boldsymbol{a}^*|}\sin\theta = n\frac{2\pi}{|\boldsymbol{k}|}, \tag{3}$$

thus,

$$2|\boldsymbol{k}|\sin\theta = n|\boldsymbol{a}^*|. \tag{4}$$

The above equation ls called Laue condition. Fig. 6 is a schematic illustration of the Laue condition, which corresponds to reciprocal representation of the Bragg condition illustrated in Fig. 5. As shown in the figure, left member of Eq. 4 corresponds to the base length of isosceles triangle with oblique side length of $|\boldsymbol{k}|$ and apex angle of 2θ. When the base length matches an integer multiple of the reciprocal lattice vector, reflected waves are in phase.

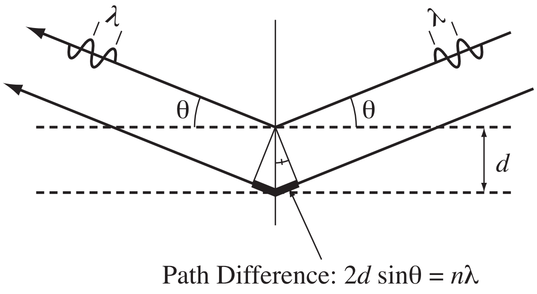


Figure 5: Schematic illustration of Bragg condition (real space)

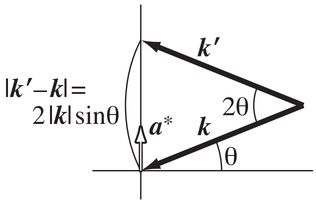


Figure 6: Schematic illustration of Laue condition (reciprocal space)

The upper oblique side of the isosceles triangle in Fig. 6 ($\boldsymbol{k}'$) corresponds to the reflected (diffracted) wave, so that Eq. 4 can be expressed as

$$\boldsymbol{k}' - \boldsymbol{k} = n\boldsymbol{a}^*. \tag{5}$$

The difference vector $\boldsymbol{k}' - \boldsymbol{k}$ is also called scattering vector.

Figs. 5 and 6 each show a case where only a single period (spacing d) exists. An actual crystal has a structure periodic in 3-dimensional directions. Therefore, the reciprocal lattice vectors that provide the basis for the period exist in the respective axial directions ($\boldsymbol{a}^*$, $\boldsymbol{b}^*$, and $\boldsymbol{c}^*$). When considering a 3-dimensional crystal, the Laue condition should be written as

$$\boldsymbol{k}' - \boldsymbol{k} = h\boldsymbol{a}^* + k\boldsymbol{b}^* + l\boldsymbol{c}^*, \tag{6}$$

where h、k、l are integers and their combination hkl is called an index number.

3.3.2 Reciprocal lattice of surface (two dimensional)

RHEED is a method for obtaining diffraction from surfaces, *i.e.*, 2-dimensional crystals. Fig. 7 shows as an example the atomic arrangement in the real space and the distribution of reciprocal lattice points in the reciprocal space for a 2-dimensional lattice containing rectangular unit cells. In the case of rectangular unit cells, as shown in the figure, the reciprocal unit cells are also rectangular. The reciprocal lattice points constituting the reciprocal lattice are distinguished by index numbers. In the case of a two dimensional lattice, the origin is indexed as 00, the point distant by $\boldsymbol{a}^*$ from the origin as 10, the point distant by $2\boldsymbol{b}^*$ from the origin as 02. Negatively positioned

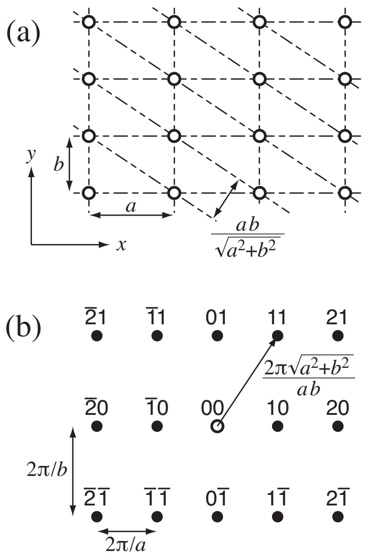


Figure 7: Two-dimensional crystal and its reciprocal lattice. (a) Real space and (b) Reciprocal space.

points are denoted by upper-barred indices as with $\overline{1}0$ (See Fig. 7(b)). A vector from the origin to a reciprocal point indexed hk is expressed as $h\boldsymbol{a}^* + k\boldsymbol{b}^*$, which is the right member of the Laue condition of Eq. 6. So that, if this vector coincides with a scattering vector $(\boldsymbol{k'} - \boldsymbol{k})$, diffraction from hk planes occur. Any reciprocal lattice points that are newly produced in a reconstructed surface structure and have a double periodicity for example are denoted by fractional order indices such as $^1\!/_2\,{}^1\!/_2$.

Where the fundamental vectors do not intersect orthogonally with each other, some calculation will be required. In a two-dimensional crystal that has vectors $\boldsymbol{a}$ and $\boldsymbol{b}$ as fundamental vectors[2], the two fundamental vectors $\boldsymbol{a}^*$ and $\boldsymbol{b}^*$ in the reciprocal space can be given respectively as follows:

$$\begin{cases} \boldsymbol{a}^* = \dfrac{2\pi(\boldsymbol{b} \times \hat{\boldsymbol{z}})}{\boldsymbol{a} \cdot (\boldsymbol{b} \times \hat{\boldsymbol{z}})} \\[2ex] \boldsymbol{b}^* = \dfrac{2\pi(\hat{\boldsymbol{z}} \times \boldsymbol{a})}{\boldsymbol{b} \cdot (\hat{\boldsymbol{z}} \times \boldsymbol{a})} \end{cases} \tag{7}$$

where $\hat{\boldsymbol{z}}$ is the unit vector in the vertical direction to the surface. From Eq. (7, it is shown that vector $\boldsymbol{a}^*$ is oriented orthogonally to vector $\boldsymbol{b}$ and its length is $2\pi/(|\boldsymbol{a}|\cos\theta)$ (where θ is the angle formed by vectors $\boldsymbol{a}$ and $\boldsymbol{a}^*$).

The reciprocal lattice points shown in Fig. 7(b) constitute the reciprocal space as viewed from the vertical direction to the surface. Though being a two-dimensional lattice, the reciprocal space exists in three-dimensional space and must be considered three-dimensionally. Because a two-dimensional crystal has no thickness in the z direction, its reciprocal lattice is $2\pi/0$ in the z direction or extends infinitely. Consequently, the three-dimensional view of the reciprocal space of the two-dimensional lattice appears as shown in Fig. 8 where the reciprocal lattice consists of rods. The rod passing through the origin of the reciprocal lattice is called 00-rod.

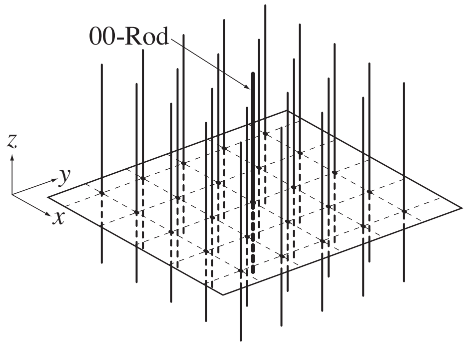


Figure 8: Reciprocal lattice of two-dimensional crystal in three-dimensional space

There is one point that requires care regarding the handling of reciprocal and real spaces. In a real space, the origin can be choose arbitrary, *i.e.* it may be placed on an atomic site or interatomic site. A reciprocal space is, however, a periodic space and its origin is a special point with a periodicity of 0 (= infinite wavelength). Hence, the origin cannot be placed at a desired site. This can be confirmed by the fact that the origin remains unchanged to changes in the location and orientation of the sample during a diffraction experiment. This means that a diffraction pattern does not affected by sample vibration, so that diffraction method is useful to determine crystal structure (atomic positions) exactly (for microscopy, you need to get rid of vibration perfectly (much less than atomic scale) to do the same).

3.3.3 Wave nature of electron beam

An electron beam is both a particle beam and a wave with a wavelength, the wave given as:

$$\lambda = \frac{h}{p}, \tag{8}$$

where h is the Planck constant and p is the momentum of electrons. The relationship between the wavelength and the kinetic energy E_k of electrons is given as below in the range where a non-relativity theory holds:

$$E_k = \frac{p^2}{2m_e}, \tag{9}$$

where me is the mass of electrons. Eq. refequ-Ek is substituted into Eq. 8 to obtain:

$$\lambda = \sqrt{\frac{h^2}{2m_e E_k}} = \sqrt{\frac{150.4}{E_k[\text{eV}]}}\ [\text{Å}]. \tag{10}$$

Hence, when $E_k = 150$ eV, $\lambda = 1$ Å; and when $E_k = 15$ KeV, $\lambda = 0.1$ Å.

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[2] Assume here that a unit lattice contains one atom only (and ignore the extinction rule).

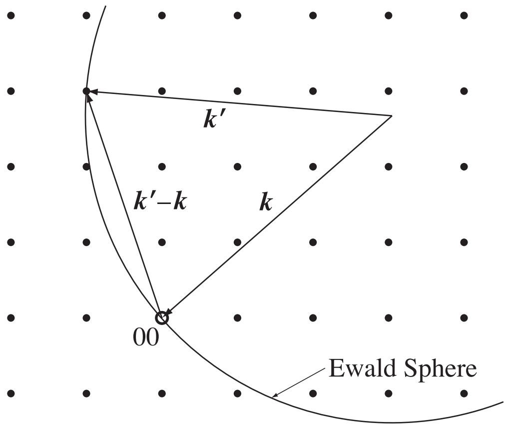

Figure 9: Relationship between reciprocal lattice, incidence vector, scattering vector, and diffraction vector in reciprocal space

In the case of a high-speed electron beam, the speed of electrons enters the domain of the special theory of relativity. In this case, the total energy E of electrons is expressed as below from the Dirac Relation and the Einstein Relation:

$$E^2 = m_0^2c^4 + p^2c^2 = m_e^2c^4, \tag{11}$$

where m_0 and c are the static mass of electrons and the speed of light, respectively. Because the kinetic energy E_k is given as:

$$E_k = m_ec^2 - m_0c^2, \tag{12}$$

Eq. 8 can be transformed as below, using Eqs. 11 and 12:

$$\lambda = \sqrt{\frac{150.4}{E_k\{1 + (E_k/2m_0c^2)\}}}. \tag{13}$$

Considering that the condition under which the effects of the special theory of relativity can be ignored is $E_k \ll m_0c^2 \sim 511$ KeV, it follows that Eq. 10 can be used if the energy of the electron beam is lower than several tens of KeV.

Eq. 8 is transformed, using the wave number $|\boldsymbol{k}| = 2\pi/\lambda$, into the following vector expression:

$$\boldsymbol{p} = \hbar\boldsymbol{k}, \tag{14}$$

where $\boldsymbol{p}$ and $\boldsymbol{k}$ are the momentum vector and the wave number vector, respectively ($\hbar = h/2\pi$). From this, it is shown that the direction of the momentum vector of electrons (*i.e.*, the flowing direction of electrons) agrees with that of the wave number vector.

3.3.4 Reciprocal lattices and diffraction

In Subsection 3.3.1, the Laue condition has been shown as the reciprocal space expression of the Bragg condition. When the incident condition to a crystal is determined, the direction in which a diffracted wave is observed can be easily determined from the Laue condition. Let the wave number vector of the incident electron beam be $\boldsymbol{k}$ ans the wave number vector of the diffracted wave be $\boldsymbol{k}'$. The incident condition (wave number and travel direction of incident wave) and the period of the crystal (positions of reciprocal lattice points) are given (see Fig. 9). First, draw $\boldsymbol{k}$ toward the origin in the reciprocal space. Because the Laue condition assumes $|\boldsymbol{k}'| = |\boldsymbol{k}|$, *i.e.*, the elastic scattering condition, $\boldsymbol{k}'$ must be a vector connecting the origin of $\boldsymbol{k}$ and a point on a sphere whose radius is $|\boldsymbol{k}|$. This sphere is called the Ewald sphere. As explained above, the Laue condition requires that a scattering vector $\boldsymbol{k}' - \boldsymbol{k}$ agrees with a reciprocal lattice point. Therefore, an actually observed diffracted wave $\boldsymbol{k}'$ will be the vector drawn from the center of the Ewald sphere to the point where the Ewald sphere overlaps with a reciprocal lattice as shown in Fig. 9. This figure is called the Ewald sphere construction. Though Fig. 9 contains only one overlapping point, a plurality of diffracted waves can be observed when a plurality of reciprocal lattice points overlap with the Ewald sphere.

3.3.5 Reflection electron diffraction from two-dimensional crystals

Reflection electron diffraction from two-dimensional crystals can be explained by the application of Ewald construction method to reciprocal lattice rods shown in Fig. 8. Fig. 10 is the sideways view of the surface that shows the relationship between the reciprocal lattice rods and the Ewald sphere. Assume that the incident electron beam $\boldsymbol{k}$ is incident at a grazing angle θ to the surface. When there is a fluorescent screen placed on the left side in the figure, points A, B$\cdots$ indicated in the figure will appear bright. Of the diffracted waves, the downward ones cannot be observed because they propagate from the surface into the inner part of the crystal and get absorbed into the crystal. Therefore, no diffracted wave can be observed downward from position S in the figure. This position S is called the shadow edge. Additionally, the (00 spot) of wave A diffracted from the 00-rod exactly corresponds to the location of the specular reflection of the incident wave and hence is also called a specular reflection spot. Point D is a location on the fluorescent screen directly reached by the incident wave and is called a direct spot (which is induced not by the electron beam that passed through the sample but by the one that reached the fluorescent screen without hitting the sample). Note that the distance from the point of incidence of the electron beam (location of the sample) to the fluorescent screen is called the camera length (corresponding to the length L in the figure) and provides the parameter that determines the scale factor of the diffraction pattern. In practice, the camera length is determined by using a standard sample which has known crystal structure.

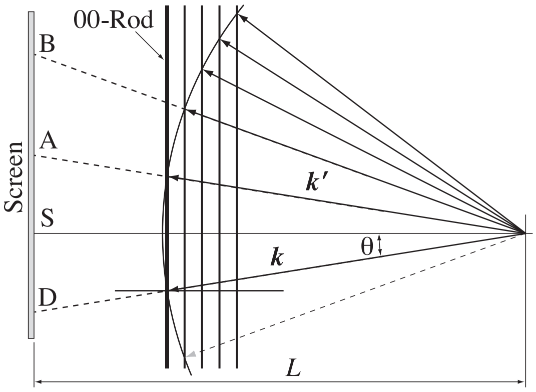

Figure 10: Reciprocal lattice rods and Ewald sphere

When an electron beam is incident from a direction of high symmetry, the diffraction spot is rendered as a pattern consisting of points arranged around several concentric circles. Of these concentric circles, the one passing through the 00 spot is called the 0th-order Laue zone and another one passing through the 10 or 01 spot lying outside the 00 spot is called the 1st-order Laue zone. Additionally, the zone between 0th and 1st order, having the reconstructed surface structure with a double periodicity, is the $^1/_2$-order Laue zone.

3.3.6 Reflection electron diffraction in actual crystals

The electron diffraction shown above assumes an ideal 2-dimensional crystal. Actual crystals, however, may differ variously from the ideal. What follows provides brief summaries of typical differences and their influences on diffraction. For more details, consult the materials listed in the Bibliography.

> **The actual crystal surface is not two-dimensional.**
Under the general condition of reflection electron diffraction (incident grazing angle of ~4°), electron beams penetrate from the substrate surface to the depth of 10-odd Å. Therefore, the z-direction intensity distribution of reciprocal lattice rods occurs, reflecting the near-surface three-dimensional structure (z-direction periodicity). Consequently, when the incident grazing angle of the electron beam is changed, the point at which the rod is cut by the Ewald sphere will be shifted to change the diffraction intensity.

> **The crystal has a finite periodicity.**
No crystal can have an infinite periodicity. Any phase shift zone (phase boundary) in the middle will break the periodicity. A single solid perfect-crystal area is called a domain and its size is called the domain size. When the domain size is A, the reciprocal lattice points (rods) will have a spread of $2\pi/A$. In a RHEED pattern, the spread of reciprocal lattice points is observed as a streak.

> **An electron beam has a finite coherence length.**
An electron beam is not a wave consisting of an infinite succession of phases and hence has a finite coherence length. Generally, an electron beam has a coherence length of several thousand Å. Therefore, if a crystal is perfect exceeding this range, the spread of reciprocal lattice points due to coherency limit will be unable to be ignored. Moreover, it is not easy to keep the spread angle of the electron beam within 1 mrad. Therefore, the origin of the Ewald sphere has a width, consequently resulting in an enlarged spot.

> **Not all crystals are necessarily oriented in one direction.**
If a surface consists of a plurality of oriented crystals, the reciprocal lattice will also consist of those of the respective crystals. Hence, diffracted waves from a plurality of domains will look overlapped with each other. If there are a large number of mosaic crystals variously rotated around the z-axis, the reciprocal lattice will be concentrically tubular with reciprocal lattice rods rotated around the origin. In this condition, the reflection electron diffraction pattern will remain unchanged to the rotation of the incidence azimuth of the electron beam.

> **The surface is not necessarily flat – Case 1 (vicinal surface)**
If a surface is slightly inclined from, for example, the (111) face, the surface will be stepped due to the inclination (this kind of surface is called a vicinal surface). If the steps are almost uniformly spaced, it follows that there is a lattice equal in width to the steps, thereby resulting in diffraction. Generally, the spaces between the steps are considerably greater than those between atoms and hence can be observed as an internal microstructure of the reciprocal lattice rods.

> **The surface is not necessarily flat – Case 2 (irregular surface)**
If a surface contains minute convexities and concavities, transmission electron diffraction will occur due to electron beams passing through the peaks of such convexities and concavities. Unlike reflection electron diffraction, transmission electron diffraction carries the information of the convex and concave parts of the bulk crystal (*i.e.*, 3-dimensional information). The significant distinction from a reflection electron diffraction pattern is that the transmission electron diffraction pattern is a lattice pattern that appears like a lookdown view of a reciprocal lattice. In addition, when passing through a convexity or concavity having a particular inclined face, an electron beam

will have a spot shape reflecting the shape of the convexity or concavity due to refraction effects.

> **The surface is not necessarily made of crystal.** When a surface is in amorphous state, the reciprocal lattice will show a broad distribution of intensity with a peak around the origin. Therefore, an obscure reflection electron diffraction pattern will occur around specular reflection. When this amorphous is as thin as several Åthick or less, an obscure pattern of diffraction from the subsurface crystal may show through the amorphous layer.

> **Inelastic scattering also occurs.** Inelastic scattering occurs along with energy exchange. In most cases, however, the amount of energy to be exchanged is no more than several eV. In the case of a high-speed electron beam, the influence of inelastic scattering will occur clearly as the effects of the loss of the memory of the incidence direction. The loss of the memory of the incidence direction means that, with the wave number maintained, the origin of the Ewald sphere can be moved to an arbitrary location that meets the diffraction conditions. Consequently, numerous paired lines (called Kikuchi lines) will appear all over the electron diffraction pattern. In the case of RHEED, Kikuchi lines will be caused by the 3-dimensional structure near the surface. Kikuchi lines enable the observer to obtain information on the bulk crystal near the surface. The types of inelastic scattering clearly observed in RHEED patterns include thermal diffuse scattering induced by thermal vibration (phonon scattering) in addition to Kikuchi lines.

In an actual experiment, analysis taking into account the influence of unavoidable impurities as well as that of noises from various external factors will also be necessary in addition to the above-mentioned considerations.

4 Reporting

4.1 Instructions for report preparation

- **Write in YOUR OWN words**. Copying (including emulating) sentences without permission or credit is called plagiarism, and is strictly prohibited act in paper preparation. It is important to read and study with other's writings, however, you should comprehend these contents and create your own for your report. **If your report is judges as a plagiarism**, it may be **expelled from the evaluation**.

- Reports must be submitted **in PRINTED FORM** with a provided cover page. Electronic versions are NOT acceptable.

- A report must contain at least the following parts:
  1. Background and purpose
  2. Method and procedure
  3. Results and analysis
  4. Discussion

- Ensure clear distinction between facts (*i.e.*, what can be asserted directly from experiment results), your own opinion (observation), and those of others. If you use other's words, never forget to cite their sources.

- Errors must be evaluated quantitatively.

- Regarding the method of the experiment
  1. **State the procedural steps actually used**. Do not copy the procedure prescribed by the procedure manual.
  2. State the specific experimental conditions (heating temperature, image capturing conditions, *etc.*).

- Regarding the results of the experiment
  1. Avoid simply listing the results. Instead, **organize and graphically represent the results** (by means of tables, graphs, etc.) so that the content will be properly understood by third parties.
  2. Temperature calibration graphs must be created by each member and added with interpolation and extrapolation curves. Be sure that **a logarithmic plot has a logarithmic scale**. Do not forget to include axis labels and their units.
  3. RHEED to reciprocal lattice transformation can be divided within the group. In this case, it is OK to attach copies of data collected by the other group members.
  4. Ensure that **reciprocal lattice diagrams have an aspect ratio of 1:1**.
  5. Reciprocal lattice diagrams must clearly **show the unit cells of the reconstructed structure** with **main points indexed**. (Standardize the method of index reference setting.)
  6. In the case of RHEED to reciprocal lattice transformation, the spot intensity should be categorized into three or so levels and indicated on reciprocal lattice diagrams accordingly, using differently sized circles. Any reciprocal lattice point added for the convenience of analysis should be represented as such to ensure distinction from other points.

- In addition to the following points, analysis and discussion can be based on interim discussion during experiments.

  - What the initial substrate was like?
  - What resulted from flushing?
  - Lattice constant (is the substrate truly Si? How does it change as deposited with Ag/Au?)
  - How is the clean Si(111) surface at high temperature?
  - Comparison of deposition temperature of Ag/Au.
  - What are common and what are different between Ag and Au?
  - Estimate the domain size from the streak pattern.
  - Comparison of earlier predictions and results of the free experiments.

- Others

  1. Questions on experiments, analysis, *etc.*, are accepted at any time by the instructor in charge (Room 465, 4F, South Wing, Building 3 of Eng.).
  2. In principle, experiment reports should be handed in directly to the instructor in charge or should be submitted into the specified post box in front of the Room 465 in the absence of the instructor in charge. **You must receive a receipt of the report** to ensure your report was accepted.
  3. Failure to submit reports will result in receiving zero points for this experiment. Students are not allowed to submit reports if they have failed to attend at least the half of the number of sessions.

Bibliography

- A. Ichimiya and P.I. Cohen, *"Reflection High Energy Electron Diffraction"*, Cambridge University Press, 2004 (ISBN 0-521-45373-9).
- S. Hasegawa, X. Tong, S. Takeda, N. Sato and T. Nagao, *"Structures and Electronic Transport on Silicon Surfaces"*, Prog. Surf. Sci. **60** (1999) 89.
- N. Sato, T. Nagao and S. Hasegawa, *"Two-Dimensional Adatom Gas Phase on the Si(111)-$\sqrt{3}\times\sqrt{3}$-Ag Surface Directly Observed by Scanning Tunneling Microscopy"*, Phys. Rev. B **60** (1999) 16083.
- S. Yamazaki, I. Matsuda, H. Okino, H. Morikawa and S. Hasegawa, *"Electrical Conduction on Various Au/Si(111) Surface Superstructures"*, e-J. Surf. Sci. Nanotech. **3** (2005) 497.
- A. Ichimiya, H. Nomura, Y. Horio, T. Sato, T. Sueyoshi and M. Iwatsuki, *"Formation of $\sqrt{21}\times\sqrt{21}$ Structure by Gold Deposition on Si(111)$\sqrt{3}\times\sqrt{3}$-Ag Surface and the Wavering Behaviour"*, Surf. Rev. Lett. **1** (1994) 1.