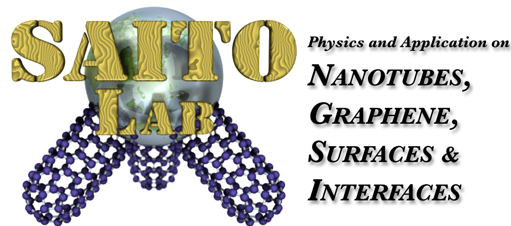
Applied Physics Laboratory II/III

(IV) Reflection High Energy Electron Diffraction (RHEED)
– Experimental Procedures –

Laboratory: Room 462, 4F, South Wing, Building 3 of Eng.
Staff Room: Room 465, 4F, South Wing, Building 3 of Eng.



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http://www.surf.nuqe.nagoya-u.ac.jp/

April 1, 2015

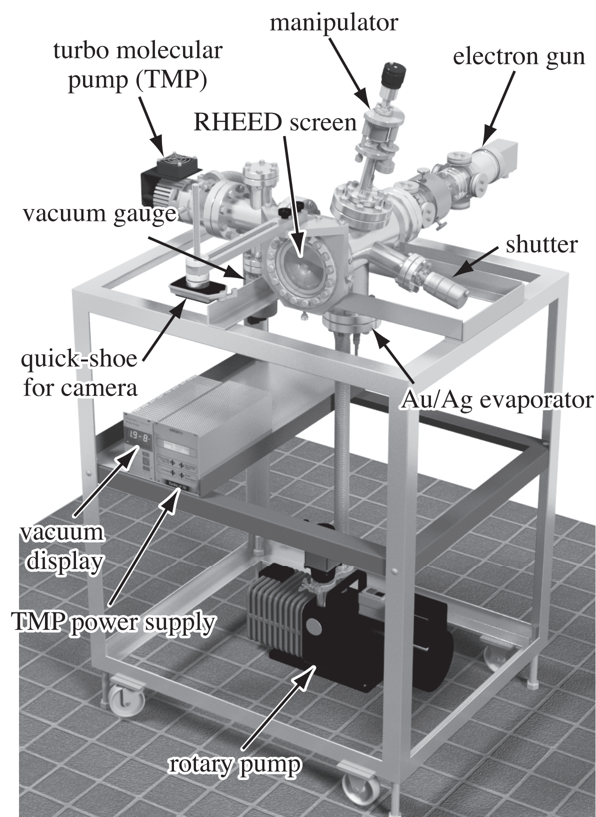
Strict Observance

1. **No eating or drinking shall be allowed** in the laboratory or adjacent hallway.
2. Wet umbrellas shall not be brought in the laboratory but shall be kept out in the hallway.
3. Absence and tardy are not allowed. **10 minutes or more late arrival will be treated as an absence**, and you can not take the class. If you were absent or late for a reason of illness, delay on public transport, bereavement etc., bring a voucher in written form so that you can take a makeup class. 4 attendances out of 5 classes is the least requirement for report submission.
4. **High-voltage equipment (max 12 kV) will be used** in the experiment. Handle the equipment with extreme care to avoid accidents. Be sure to check any unclear points with the instructor in charge.
5. Keep hands off instruments or equipment other than the student experiment equipment.

Cautions

- Carefully read these procedures and fully understand the work content before starting the experiment. Be sure to work with sufficient knowledge and care to avoid dangers such as electric shock.
- Depending on the experiment to be conducted, students may be divided into Groups A and B. We have only one set of laboratory equipment. Hence, the two Groups cannot conduct the experiment simultaneously. The Groups shall work in cooperation with each other.
- Experiment notebooks are important records. One notebook shall be issued to each group. Group members shall take turns to keep records.
- Experiment work shall be divided among the Group members. Each member shall carry out the role assigned to them. While having hands free, they shall watch other members working.
- Experiment data shall be analyzed later in the day. The data thus obtained shall be consulted for work in the next session.
- Depending on the actual experiment schedule, the experiment may not be conducted according to the schedule prescribed in these procedures. In such a case, follow the instructions given as necessary by the instructor in charge.

Components of the equipment



Note: Though omitted in the above image, a cylinder for shielding external light is installed around the fluorescent screen and a DSLR camera is installed on the quick shoe.

Session 1

1.1 Observing an existing Si(111) surface

First, perform the RHEED observation of the surface of the Si(111) substrate already set in vacuum. This sample is either of the following: the one that is left over from the last session when the experiment group observed a clean surface; or a newly introduced silicon wafer for the last time. Listen to the explanation of the type of sample by the instructor in charge.

In the first session, the instructor in charge will explain, among other things, how to handle the electron gun while actually operating it. From the next time on, each member will operate the electron gun and must memorize the steps given below.

1. **Starting the electron gun**

    - Ensure a vacuum of $1{\times}10^{-4}$ Pa or less. Then, turn ON the high-voltage power supply switch and the switches of the five coil power supplies.
    - Slowly turn the filament current up to the specified value written on the whiteboard. (Carefully avoid an overcurrent).
    - Turn the Wehnelt voltage up to the specified value (which is a temporary value and should be fine-adjusted later).
    - Set the electron gun high voltage coarse adjustment knob (Coarse) to 10 kV. Then, turn the fine adjustment knob (Fine) slowly up to the specified high voltage value (10.000 kV) (This value determines the electron beam wavelength and hence must be set accurately).
    - Raise the coil power supply currents AX and AY up to their respective specified values.
    - Turn the DY knob slightly (approx. 0.3 A) so that an electron beam will hit the fluorescent screen. Then, adjust the DX knob to bring the spot near the center in the left-right direction.
    - While looking at the spot on the fluorescent screen, adjust the Wehnelt voltage until the whole spot appears uniform and brightest.
    - Adjust the lens so that the spot will appear smallest.
    - Operate the DX and DY knobs so that reflection diffraction from the sample will be visible.

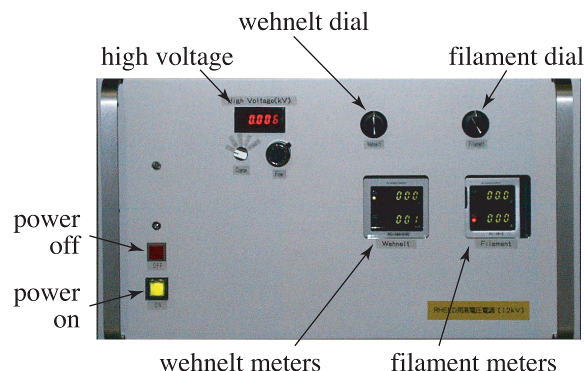


図 1.1: Components of RHEED high-voltage power supply control panel

    - After finishing the adjustments, turn up the filament current up to 1.2 A (This may be performed before the adjustments described immediately above).
    - Recheck the electron gun voltage, which may have fluctuated, and correct it if shifted (Adjust the electron gun high voltage as necessary).

2. **Adjusting the diffraction conditions**
   Using the manipulator and the DX and DY coils, make adjustments until the RHEED pattern is clearly visible. Fine-adjust the DX coil here so that the pattern will be positioned as symmetrically as possible. The diffraction conditions can be shifted easily due to sample heating or energization and should be readjusted each time the pattern is checked or photographs are taken.

3. **Photographic recording**
   A DSLR camera is used to record RHEED patterns. Basic adjustment and setup of the camera will be carried out by the instructor as well as its connection to the computer. Photographing can be performed via the computer, and the captured images can be checked and retouched immediately. Before photographs are taken, the shutter speed must be set appropriately on the camera side. An image file name consists of the date and time of photographing. Hence, record the photographing time in the experiment notebook and add specific descriptions as well. Print as many copies of each captured image as the number of members on the spot.
   Detailed descriptions of the photographing and printing procedures are given separately. Copies of measurement data files (JPEG) will be issued upon request. Bring a USB memory or similar medium and request the instructor to that effect.

4. **Stopping the electron gun**

    - Record the operating conditions of the electron gun (acceleration voltage, Wehnelt voltage, fila-

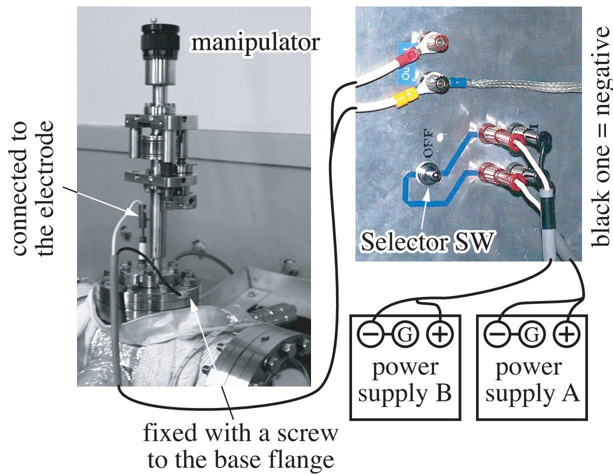


図 1.2: Wiring between sample holder and sample heating power supply.

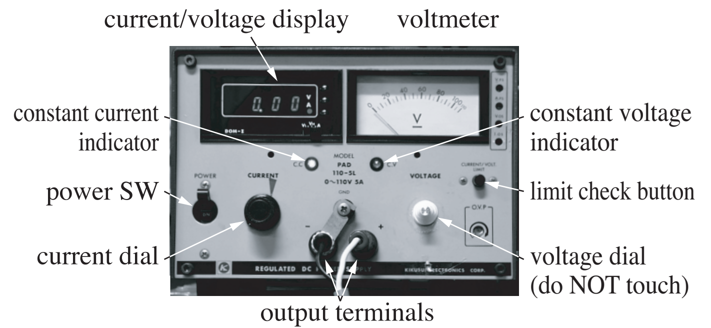


図 1.3: Components on the sample heating power supply control panel

ment current, and current in each coil) for reference for work in the next and subsequent sessions.

- Set the high voltage to 0 V (first set the Fine knob to 0 and then the Coarse knob to 0), then the Wehnelt voltage to 0 V, and finally the filament current to 0 A.
- Turn all the coil power supply current knobs back to 0 A.
- Turn OFF the high-voltage power supply and five coil power supplies.

1.2 Calibrating the Si substrate temperature

Check here the temperature-resistance dependence of semiconducting silicon and create a substrate temperature prediction graph from the resistance value. The graph created here provides the reference for temperature throughout this experiment.

To minimize measurement errors, perform the same work twice (or once per group when the students are divided into two groups) and determine the average value of the two measurements for use as the temperature calibration data for the subsequent sessions. If the band gap energy evaluation (explained later) differs largely from known Si value, try using one of the datasets for temperature calibration.

1. **Wiring the sample heating power supplies**
   Two types of power supplies are selectively used to heat the sample. Ensure that the power supply switch is turned OFF. Then, wire the power supplies as instructed by Fig. 1.2.

2. **Heating the sample**
   Fig. 1.3 shows the power supply control panel used for heating the sample.
   When the sample heating current is 5 A or less, set the selector switch to (LO) and heat the sample only with Power Supply A. When the sample is cool, an attempt to pass current immediately will fail and the constant voltage indicator lamp (green) will illuminate. In this case, wait for several seconds without turning the current control knob. (Do not continue turning the knob; failure to comply will result in a sudden flow of high current.) If the constant current indicator lamp (red) fails to illuminate after 30 seconds elapsed, it is most likely that the selector switch has been forgotten to be set to Power Supply A.

3. **Measuring the resistance and temperature of the sample**
   Subject the sample to currents of 0.1, 0.2, 0.3, 0.4, 0.5, 0.6, 0.7, 0.8, 0.9, 1.0, 1.2, 1.4, 1.6, 1.8, 2.0, 2.2, 2.4, 2.6, 2.8, 3.0, 3.5, 4.0, 4.5, and 5.0 [A] and record the voltage and the substrate temperature for each case (measuring method to be explained later). Note, however, that the substrate temperature cannot be measured when the current is small. The voltage changes over time. Do not measure the voltage until more or less stable. Constantly monitor the vacuum pressure during measurement. When 1×10^{-5} Pa is exceeded, interrupt the heating of the sample and wait until the vacuum pressure is back to the normal level.

4. **Method of measuring the substrate temperature**
   Measure the substrate temperature using a radiation thermometer. Follow the measurement procedure explained by the instructor in charge. The radiation thermometer is an optical device and must be handled with care.

5. **Organizing the measurement data**
   Based on the measurement data, draw graphs as shown in Fig. 1.4.[1] (be careful with the unit of temperature!) These graphs will be used for temperature calibration

[1] No current vs. temperature graph has to be created for the following reason. While a linear relationship theoretically exists between the reciprocal of the temperature and the logarithm of the resistance value, no theoretical formula exists between the current and resistance value, making the graph extrapolation difficult.

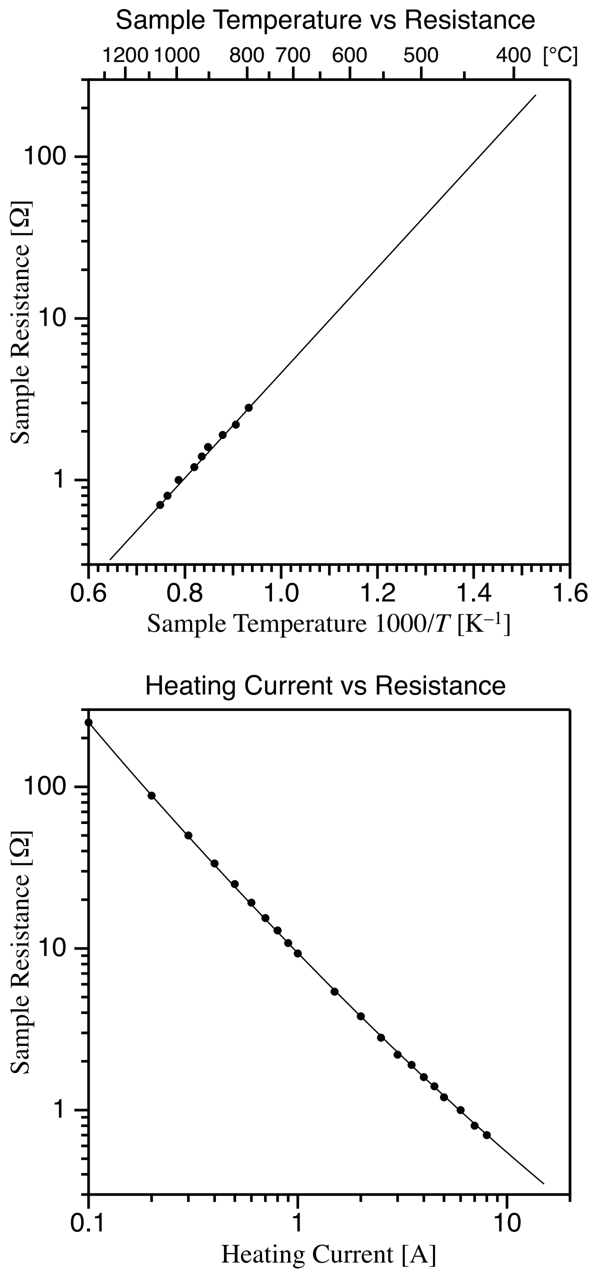


図 1.4: Typical sample temperature calibration graph

in the following sessions. Note that a resistance vs. temperature graph should be fitted using the least-square method to a linear equation and the resistance vs. current graph should also be fitted using the least-square method to a quadratic equation.[2]

1.3 Assignments of the day

Complete the following tasks by the next session:

- Create the substrate temperature calibration graph that covers the range from 400 °C to 1300 °C.
- Determine the band gap (unit: eV) of this substance from the gradient of the R-T graph and compare it with the reference data (See below).

[2]Quadratic relation is an empirical formula and not theoretical one.

Electrical resistance of intrinsic semiconductors

It is known that the electrical resistance of an intrinsic semiconductor (non-doped semiconductor) is expressed as:

$$R \propto \exp\left(\frac{E_g}{2kT}\right) \tag{1.1}$$

where E_g and T are respectively the band gap and absolute temperature of the semiconductor. A semiconductor band gap is a material-specific value and is one of the very important physical properties of a semiconductor. In most cases (including this experiment), semiconductors contain dopants so that they will be of n-type or p-type conductivity. Such a semiconductor can be regarded as an intrinsic semiconductor when it has a sufficiently high temperature (> approx. 300 °C). Note that a non-doped semiconductor shows at room temperature a resistance value almost equivalent to that of an insulator.

Reference data

The physical properties of typical semiconductor materials are as shown below:

- Physical properties of Si
  - Mass number: 28.086
  - Density: 2329 kg/m^3
  - Lattice constant: 5.4307 Å
  - Crystal structure: diamond structure
  - Melting point/boiling point: 1410/2355 °C
  - Band gap: 1.206 eV (indirect transition)
- Physical properties of Ge
  - Mass number: 72.59
  - Density: 5323 kg/m^3
  - Lattice constant: 5.65754 Å
  - Crystal structure: iamond structure
  - Melting point/boiling point: 937.45/2830 °C
  - Band gap: 0.785 eV (indirect transition)
- Physical properties of GaAs
  - Mass number: 144.64
  - Density: 5320 kg/m^3
  - Lattice constant: 5.653 Å
  - Crystal structure: zinc blende structure
  - Band gap: 1.43 eV (direct transition)
- Physical properties of GaN
  - Mass number: 83.72
  - Lattice constant: 3.189 Å
  - Crystal structure: Wurtzite structure
  - Band gap: 3.5 eV (direct transition)
- Physical properties of SiC
  - Mass number: 40.097
  - Density: 3160 kg/m^3
  - Lattice constant: 4.359 Å
  - Crystal structure: zinc blende (3C), hexagonal (6H, 4H), *etc.*
  - Band gap: 2.23/3.0/3.3 eV (3C/6H/4H, indirect transition)

Session 2

2.1 Observing the clean Si(111) surface

In this session, obtain a clean Si(111) surface in ultra-high vacuum to observe its reconstructed structure, a 7×7 structure, using RHEED.

1. **Starting the electron gun**
   Similarly as in the last session, operate the RHEED electron gun and adjust the electron gun and the sample manipulator so that the RHEED pattern will appear on the fluorescent screen.

2. **Cleaning the substrate (flushing)**
   From the graph created in the last session, determine the current value for when the substrate temperature is 1250 °C (not K!). Using a current value about 1 to 2 A lower than the obtained value as the target value, heat the substrate for approx. 5 seconds to confirm whether the RHEED pattern agrees with a clean-surface RHEED pattern given for reference (Fig. 2.1). If the former agrees with the latter, the flushing of the surface is completed. Repeat this operation until a clean pattern is obtained. Be sure to record the conditions of flushing (heating current, and duration and number of times of flushing) for reference for the subsequent sessions. Additionally, from the current used, determine the temperature during flushing. Be careful to avoid applying an overcurrent to Si, which has an approx. melting point of 1400 °C. Once the substrate has melted, not only the day ' s session cannot be continued but recalibration of the temperature will be required. The detailed procedure of the flushing operation is as follows:

   (a) Ensure that the two heating power supplies are wired to heat the sample and both of the current control knobs are set to 0.

   (b) Ensure that the power supply selector switch is set to Power Supply A (5 A max.).

   (c) Turn ON the switches of the two power supplies.

   (d) While pressing down the current check button of Power Supply B (see Fig. 1.3), adjust the current control knob to approx. 1 A.

   (e) Using Power Supply A, apply a current of approx. 1 A to the sample.

   (f) Turn the selector switch straight to Power Supply B (No stopping in the middle!).

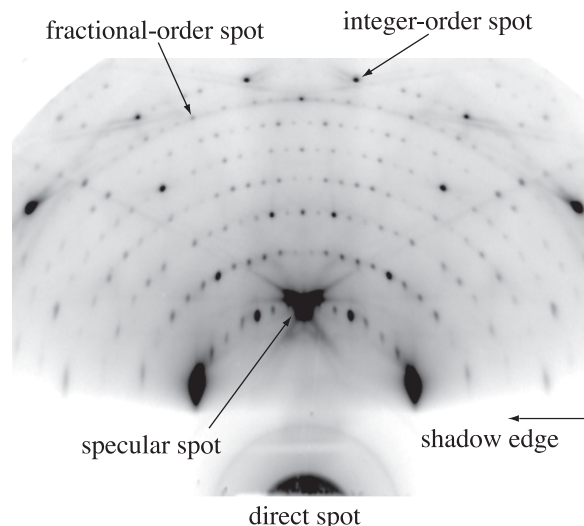


図 2.1: Typical RHEED pattern of clean Si(111) 7×7 surface

   (g) Using Power Supply B, pass a current of up to the target value quickly without exceeding it. Wait for five seconds and then turn the current down to 0.

3. **Observing and recording the RHEED patterns of the clean surface**
   Rotate the sample manipulator to observe changes in the pattern. Observe that the reciprocal lattice rods are cut by the Ewald sphere under the RHEED conditions (All members must observe the patterns). Adjust the diffraction conditions and record the RHEED patterns.

2.2 Substrate temperature dependence of the clean surface

Observe here how the clean Si(111) surface structure changes in response to the substrate temperature. Experiments should be carried out to evaluate phase transition temperatures.

1. **Observing and recording changes in the pattern**
   While observing the RHEED pattern, turn up the substrate temperature slowly and maintain the substrate heating current at a point where the RHEED pattern has shown a significant change. With this state maintained, record the RHEED pattern and the heating current and the vacuum pressure at the same time. Repeat the same operation each time a change occurs within the heating current range up to 5 A. Finally, leave the substrate to cool down to the room temperature and record the pattern again.
   When the students are divided into Groups A and B, the above-mentioned work should be performed by each group.

2. **Stopping the electron gun**
   Similarly as in the last session, stop the electron gun and turn OFF the sample heating power supply.

2.3 Determination of camera length and RHEED-to-reciprocal lattice transformation

Assuming that the sample used is Si(111) substrate, calculate the camera length of this experiment from obtained RHEED pattern. Consult a instructor in charge for more detail.

2.4 Assignments of the day

Complete the following tasks by the next session:

- Perform a RHEED-to-reciprocal lattice transformation of the spot selected from the RHEED patterns captured today. Then, obtain the geometry and lattice constant of the unit lattice.
- Determine whether the lattice constant calculated as above agrees with the value predicted for the Si(111) surface.

Session 3

3.1 Wiring and degassing the evaporation source

The Ag and Au evaporation sources each consist of a boron nitride (BN) tube of 3 mm diameter (and 2 mm inner diameter) wrapped around with a tungsten wire and containing the vapor deposition material inside. Pass heating current through this tungsten wire to cause the deposition material to vaporize and vapor-deposit on the surface (Fig. refEvaporator).

Prior to vapor-deposition, the evaporation source must be preheated and degassed to eliminate impurities around the evaporation source. Insufficient degassing may result in vapor-deposition of impurities.

1. **Wiring the evaporation source**
   The Ag evaporation source is designed to pass current between the far-side electrode and the right-side electrode. The power supply for evaporation source is Power Supply B, which is also used for sample heating. Hence, disconnect the gray wire connected to the sample and connect the black wire connected to the evaporation source (**Ensure that the power switch is turned OFF during work!**).

2. **Setting the power supply**
   The evaporation source is driven in the constant voltage (CV) mode. Therefore, set the **voltage control knob of the power supply to 0 V** and the **current control knob approximately to the half position (mark position)**. Be sure to do this. Otherwise, the evaporation source may be destroyed when the power is turned ON!

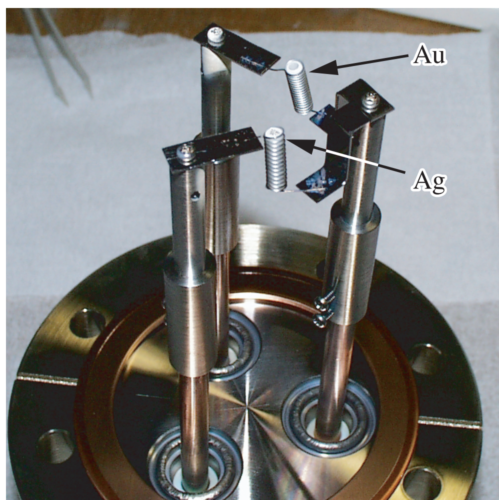


図 3.1: Ag and Au evaporation sources

3. **Degassing the Ag evaporation source**
   Ensure that the shutter is closed. While watching the vacuum gauge, turn the voltage up to the specified value slowly so that the vacuum pressure will not exceed 5×10^{-5} Pa. With this state maintained, wait for one minute. If the vacuum deteriorates in the middle, set the voltage back to 0 V once and wait until the vacuum stabilizes. Then, repeat this operation. When the vacuum pressure remains at or below 5×10^{-5} Pa despite one minute of heating, the degassing process is completed.

4. **Degassing the Au evaporation source**
   Perform the above-mentioned procedure to the Au (left-side electrode). **When changing the wiring, use provided leather gloves to prevent burn injury.** Be careful with the degassing voltage, which differs from that for the Ag.

5. **Starting the electron gun**
   Similarly as in the last session, start the electron gun so that a RHEED pattern will be visible.

3.2 Observation of Ag/Au deposited surface

Observe here how the surface structure changes when Ag or Au is deposited on the clean Si(111) surface at two kinds of substrate temperatures (room temperature and 500 °C). When the students are divided into two groups, A and B groups are in charge of Ag and Au experiments respectively.

1. **Starting the electron gun**
   Similarly as in the last session, start the electron gun so that a RHEED pattern will be visible.

2. **Cleaning the substrate**
   Disconnect the heating Power Supply B from the evaporation source and reconnect it to the sample. Then, set the power supply **current control knob to 0 A** and the **voltage control knob to Max**. Similarly as in Section 2.1, subject the Si substrate to flushing to obtain a clean surface. At the same time, record the resulting pattern.

3. **Ag deposition and recording of pattern changes during the deposition**
   - Disconnect the heating Power Supply B from the sample and reconnect it to the evaporation source. Set the power supply **voltage control knob to 0 V** and the **current control knob approximately to the half position (mark position)**.
   - Heat the sample at desired temperature (only for 500 °C of substrate temperature).

- While watching the vacuum gauge, turn the voltage control knob of the evaporation source power supply slowly up to the specified value over 30 seconds to 1 minute. If the vacuum deteriorates beyond 5×10^{-5} Pa during this operation, perform the above-mentioned degassing work again. (After degassing, clean the substrate once more.)
- Holding the specified value, wait for 30 seconds and then open the shutter (*i.e.*, set the dial to $-30°$). When doing this, be sure to check the shutter dial to avoid turning the shutter excessively.
- Open the shutter to perform vapor deposition. The duration of vapor deposition will be specified separately. During vapor deposition, take a photograph every 30 seconds (Adjust the shutter speed and filament current of the electron gun appropriately because the evaporation source is glowing bright; maximum filament current is 1.27 A).
- After the elapse of the specified time, close the shutter (*i.e.*, set the dial to the "0" position) and stop heating the evaporation source and the substrate. When doing this, be sure to check the shutter dial to avoid turning the shutter excessively.
- After completing vapor deposition, let the substrate temperature go down to room temperature. Then, take photographs again. Additionally, observe how the pattern changes when the sample manipulator is operated to change the incidence azimuth of the electron beam (and take notes as necessary).
- Subject the substrate to flushing again to give it a clean surface again. Then, change the substrate temperature to repeat the experiment.

4. **Au deposition and recording of pattern changes during the deposition**
Similarly as in the Ag case, observe here how the surface structure changes when Au is deposited on the clean Si(111) surface at two kinds of substrate temperatures (room temperature and 500 °C). Before starting the experiment, calculate Ag and Au deposition rates so as to coincide deposition amount of both elements. Calculation method of deposition rate is described below.

5. **Stopping the electron gun**
When completing the experiment with the two different substrate temperature settings, turn OFF the electron gun, the sample heating power supply, and the evaporation source power supply. Be careful that no switches are forgotten to be turned OFF.

3.3 Preparation for discussion

The next session is intended for discussion. Prepare for the discussion session with the following points in mind:

- Correctly understand the content of the experiment conducted by the Group. (What was done under what conditions?)
- Calculate the substrate temperature, the deposition rate, and other parameters.
- Transform each characteristic RHEED pattern into a reciprocal lattice.
- Use the reciprocal lattice as the clue to consider the surface structure.
- Observe the changes in the surface structure to consider what occurred to the surface and why such changes occurred.

Method of calculating the deposition rate

The deposition rate can be calculated from the vapor pressure of the vapor deposition material. When the vapor pressure of the deposit is $P(T)$ at the source temperature T, the number n of atoms vaporized per unit area per unit time is given as:

$$n = \frac{P(T)}{\sqrt{2\pi mkT}}, \tag{3.1}$$

where m and k are the atomic mass and the Boltzmann constant, respectively. When the area of the crucible mouth is A, the amount N_0 of atoms flying out from the crucible per unit time is given as:

$$N_0 = nA = \frac{PA}{\sqrt{2\pi mkT}}. \tag{3.2}$$

Here, assume that the crucible to substrate distance is r and the atoms flying out from the crucible are deposited evenly over a hemisphere with a radius of r. Since the number of atoms flying onto the hemisphere per unit time is N_0, the amount N of atoms flying per unit area of the hemisphere can be expressed as:

$$N = N_0/2\pi r^2. \tag{3.3}$$

When a monolayer (ML) is defined as deposition of an atom per unit cell area U of the substrate, the deposition rate R is given as:

$$R = NU \text{ [ML/s]}. \tag{3.4}$$

Physical properties of Ag and Au, as well as physical constants are given below for reference:

- Physical properties of Ag
  - Mass number: 107.868
  - Density: 10500 kg/m^3
  - Lattice constant: 4.08626 Å
  - Crystal structure: FCC
  - Melting point/boiling point: 961.93/2212 °C
- Physical properties of Au
  - Mass number: 196.9665
  - Density: 19320 kg/m^3
  - Lattice constant: 4.07833 Å
  - Crystal structure: FCC
  - Melting point/boiling point: 1064.43/2807 °C
- Physical constants
  - Boltzmann constant: 1.380662×10^{-23} J/K
  - Mass unit: $1.6605655 \times 10^{-27}$ kg
  - Elementary charge: $1.6021892 \times 10^{-19}$ C
  - Planck constant: 6.626176×10^{-34} Js
  - Light velocity: 2.99792458×10^{8} m/s
  - Avogadro ' s number: 6.0221415×10^{23}

Session 5

5.1 Free experiment

In this session, perform vapor deposition and heating, using freely set parameters such as the combination of evaporation sources, the deposition amount, and the substrate temperature, to investigate how the surface changes.

Based on the discussion in the last session, clarify the purpose or what to be ascertained. Give a rough prediction of the outcome of the experiment. Additionally, draw up the experiment plan, taking the estimated total experiment time into account.

Typical experiment

- After deposition on a low-temperature substrate, increase the substrate temperature slowly to observe the change.
- Deposit the substrate with Au and then with Ag or the other way around.
- Deposit Ag or Au on the substrate at high temperature to alter the surface structure. Then, continue with vapor deposition at room temperature.
- Investigate the dependence of the domain size on the substrate temperature.
- Investigate the dependence of the surface structure on the deposition amount.

Just after the free experiments, discuss each other on the predictions and results of the experiments.

付 録A

A.1 Principle of typical vacuum pumps

A.1.1 Rotary pump

Fig. A.1 illustrates the principle of a rotary pump. The two vanes (moving vanes) turns together with the rotator to suck in air from the inlet and send exhaust air to the outlet. Generally, oil is applied to the moving parts to prevent air leaks. Though this type of pump can be used from atmospheric pressure, its maximum achievable vacuum pressure is limited to around 10^{-1} Pa.

A.1.2 Turbo molecular pump

Fig. A.2 shows the construction of a typical turbo molecular pump. Similarly to gas turbines and the like, the moving and stationary vanes differently oriented from each other are arranged in layers so that the moving vanes can turn at speeds of several tens of thousands rpm (revolutions per minute) (The one used in this experiment turns at 75000 rpm). When the vacuum pressure is low ($> 10^{-3}$ Pa), this type of pump operates in the same principle as an electric fan to pass gas through it. When the vacuum is so high that the gas cannot be handled as a viscous fluid, the vanes sputter incidental incoming molecules out to the outlet. Though usable from atmospheric pressure, a turbo molecular pump needs an auxiliary pump, such as rotary pump, to force exhaust air out of its rear stage. Its maximum achievable vacuum pressure is approximately 10^{-7} Pa.

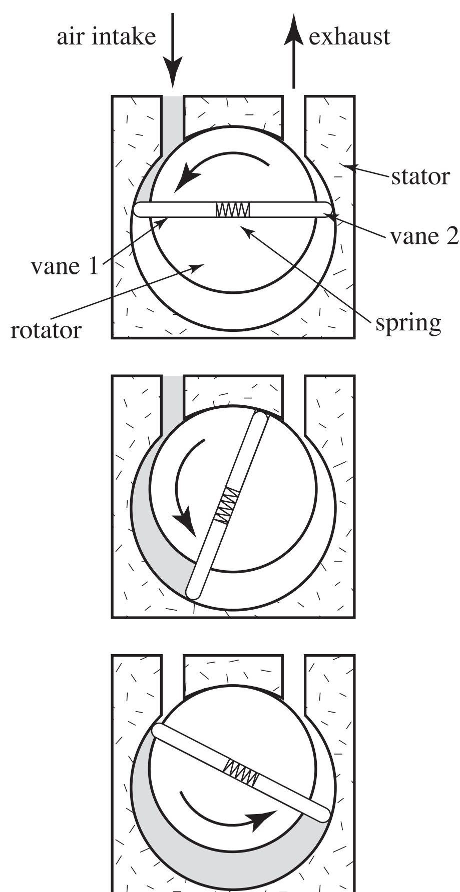


図 A.1: Operating principle of rotary pump

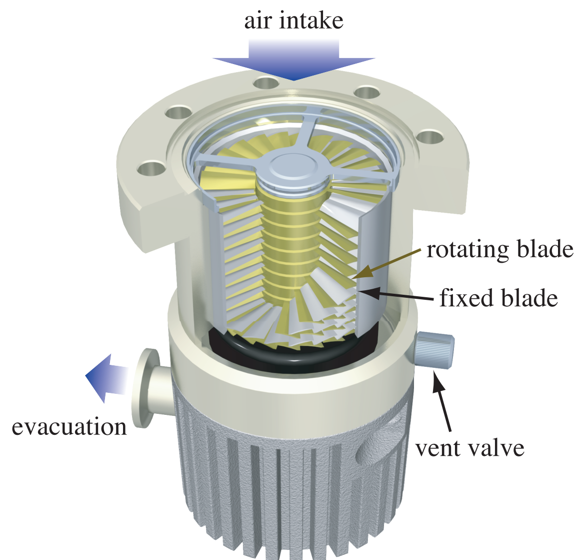


図 A.2: Operating principle of turbo molecular pump

A.1.3 Ion pump

Fig. A.3 illustrates the principle of a typical ion pump. Apply a voltage of several kV between the titanium cathode and the stainless steel honeycomb anode and apply a magnetic field of several K gauss in the same orientation as the electric field. Each electron emitted from the cathode follows a spiral path in the magnetic field toward the anode. On their way to the anode, electrons ionize the gas in the container. Such ionized gaseous molecules are accelerated by the electric field and hit hard on the titanium cathode to sputter titanium atoms from there. Sputtered titanium atoms deposit on the stainless steel anode to form a titanium layer. The surface of this titanium thin film is active and is characterized by its property of never releasing any gas adsorbed into it. Because titanium thin films continue to be deposited one on top of the other, the surface of the anode is always covered with active titanium so that adsorbed gaseous molecules will be confined between titanium thin films. This pump has a maximum achievable degree of vacuum as high as 10^{-9} Pa or more. A low vacuum pressure, however, leads to electric discharge between the electrodes. Hence, this pump cannot be started until its vacuum pressure is 10^{-3} Pa or less. This pump will have its life significantly reduced when used at a low vacuum pressure for a long time because it consumes the cathode for vacuum exhausting.

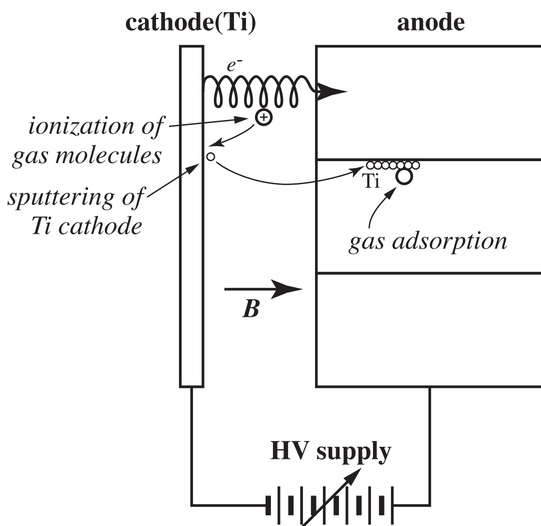


図 A.3: Operating principle of ion pump

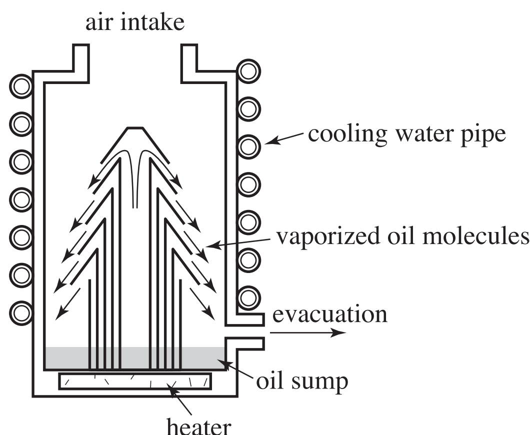


図 A.4: Operating principle of oil diffusion pump

A.1.4 Oil diffusion pump

Fig. A.4 shows the construction of an oil diffusion pump. The housing contains a stack of umbrella-shaped metal plates and oil inside. The built-in heater heats this oil to generate a flow of oil vapor along the metal plates. This flow carries gaseous molecules to the lower part of the housing so that the condensate will be sucked out from the outlet connected to, *e.g.*, a rotary pump. The housing is wrapped around with a cooling water pipe to turn vaporized oil back into liquid. This type of pump cannot be used until the vacuum pressure is increased by a rotary pump or the like to a certain degree ($\sim$ 1 Pa) to prevent oil from burning as heated. An ordinary pump of this type has a maximum achievable vacuum pressure of about 10^{-5} Pa. Ones featuring oil vapor leak prevention can achieve a vacuum pressure 10 or more times better. There used to be a time when diffusion pumps were used with mercury that vaporizes at a lower pressure than oil. Mercury diffusion pumps are no longer used for safety reasons. Following the advent of turbo molecular pumps that are easy to operate and free from oil contamination, more and more oil diffusion pumps are recently put out of use.

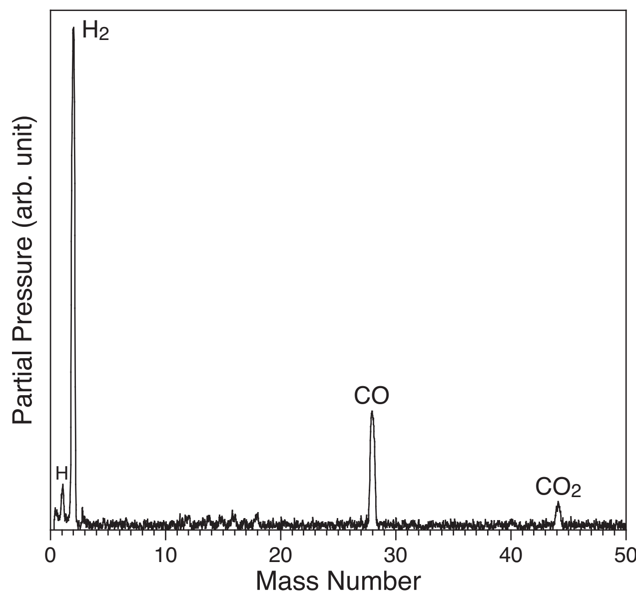


図 A.5: Components of residual gas in ultra-high vacuum

A.1.5 Other pumps

There are also additional types of vacuum pumps including diaphragm pumps, titanium sublimation pumps, sorption pumps, and cryogenic pumps among others. These various types of vacuum pumps are selectively used to suit different applications. Though no specific details are given here, those interested are advised to consult the source materials listed in the bibliography or search the Internet for relevant information.

A.2 Quality of vacuum

As is well known, atmospheric air consists mainly of nitrogen and oxygen. The residual gas vacuum-pumped out of the vacuum chamber, however, shows a compositional breakdown significantly different from that of atmospheric air. Fig. A.5 is a graph showing the results of a QMS (Quadrupole Mass Spectrometer) analysis of the residual gaseous components in ultra-high vacuum (1×10^{-7} Pa) (but not a graph based on data obtained using the student experiment equipment). The x-axis stands for the molecular mass number of gas, while the y-axis stands for the partial pressure of each component. As is clear from the graph, H_2 with the mass number of 2 occupies the most part of the residual gas. This is because hydrogen molecules cannot be pumped out easily using a vacuum pump. The second largest part is occupied by the gas with the mass number of 28, which is not N_2 but CO. If baking process of the vacuum chamber is insufficient, H_2O(=18) intensity becomes significant. Thus, not only the vacuum pressure but the components of the residual gas must be taken into account when conducting an experiment in vacuum.