

2026/4/28 update



Rules video



施設のルール  
動画

# Cryo Cross-section Polisher(CCP) Basic manual

Laboratory of XPS analysis

Contact K. Suzuki Ext. 6882

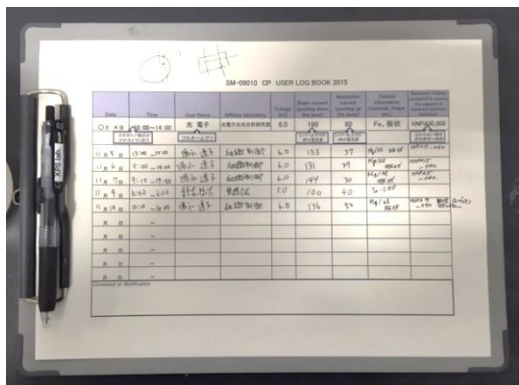
S. Yoshida Ext. 6882

# Caution

Please read carefully and follow these rules.

- Outdoor shoes are not allowed in the laboratory. Please keep the area clean and dispose of all waste properly.
- If you notice any equipment failure or malfunction, report it to the staff immediately.
- Do not handle any equipment roughly.
- Do not remove or take any laboratory items without permission, and do not lose them.
- Do not touch any items introduced into the vacuum chamber with bare hands. If contamination occurs, clean them yourself.
- Do not take out the CP sample stage or the air-free (non-atmospheric exposure) holder without permission.
- When using the precision lapping machine (Handy Lap), follow its manual and prepare your own polishing papers.
- For the use of the precision cutting machine, vacuum heating/deaeration system, or other equipment, obtain permission from the staff in advance.
- Users must make reservations and use the equipment according to the reserved time. Cancellations must be made by the day before.
- When using the equipment during late night, early morning, or holidays, all troubles must be handled at your own laboratory's responsibility. Emergency contact information is posted next to the laboratory entrance door.
- Inform your supervisor before using the equipment.
- When using the cryo function, contact the staff in advance to confirm the liquid nitrogen refill schedule.
- The cryo function cannot be used with the large sample rotation holder, position adjustment holder, or sample stage holder. Please select the correct holder.
- Shield plates (magnetic / non-magnetic) must be brought by users or purchased from the facility. If purchasing from the facility, contact the staff in advance.
- For the use of consumables such as G2 resin, please contact the staff in advance.

# Before using



Please fill in your name, usage time, and other required information in the usage log. If your usage extends beyond the reserved time, you must update your reservation in advance.



Necessary tools are stored in the CCP box. Holders and related items are located on the metal rack next to the CCP system. Take only the required holders from their cases, and be sure to return them to the original cases after use. **Do not touch holders or tools with bare hands. Always wear gloves when handling them.**



**When using the cryo function, contact the staff in advance. Confirm the liquid nitrogen refill schedule before making a reservation.**



**Shield plates must be prepared by each user. Please arrange them within your laboratory or purchase them as consumables from our facility. If you wish to purchase, contact the staff in advance.**

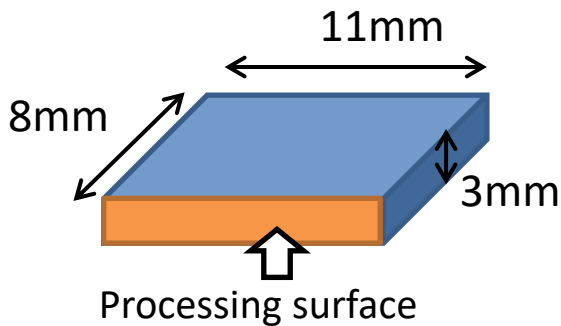
Shield Plates for CCP (JEOL Catalog)

Part No.: 804448248 Specification: Shield Plate (L) Price: Approx. JPY 15,000 per piece

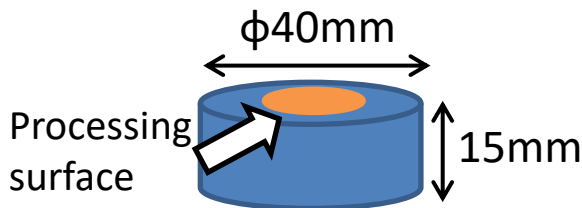
Part No.: 804437742 Specification: Magnetic Type Price: Approx. JPY 8,000 per piece

# Sample Preparation (if required)

For the Normal Holder



For Large Sample Rotation Holder



Samples mounted on the CCP system should basically be in a plate shape. The maximum size depends on the type of holder. If the sample is too large, cut it to the appropriate size. For samples with irregular shapes, please consult the staff in advance.

## Cross-section Processing

- Normal Holder: 11 mm × 8 mm × thickness 3 mm
- Large Holder: 20 mm × 7 mm × thickness 3 mm
- CP base Holder: 11 mm × 8 mm × thickness 2 mm
- Positioning Holder: 11 mm × 8 mm × thickness 3 mm
- Transfer vessel Holder: 11 mm × 8 mm × thickness 3 mm

When mounting samples on holders such as the normal holder, a minimum depth of approximately 4 mm is required to secure the sample with a clip.

## Surface Processing

- Large sample rotation Holder: Diameter 40 mm × thickness 15 mm
- Resin-embedded samples: Diameter 25.4 mm × thickness 8–16 mm



Precision Cutting Machine

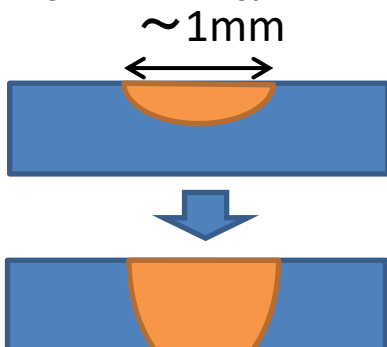


Diamond Wire Saw

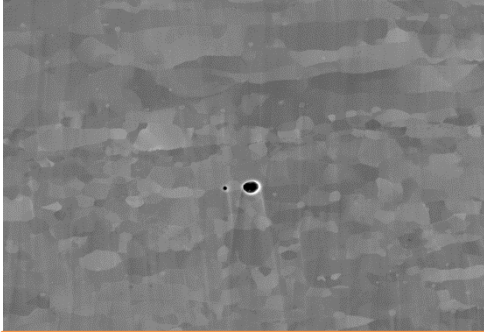
A cutting machine is available in the laboratory. Please use it when cutting samples. However, you must receive training from the staff in advance before using the machine.

With the CP system, it is possible to produce a clean cross-section with a width of approximately 1 mm, free from mechanical polishing damage and deformation layers, suitable for SEM, EBSD, and AES analysis.

As shown on the left, the sample is gradually milled in a bowl-shaped profile from the top. With sufficient processing time, the milling can penetrate through the

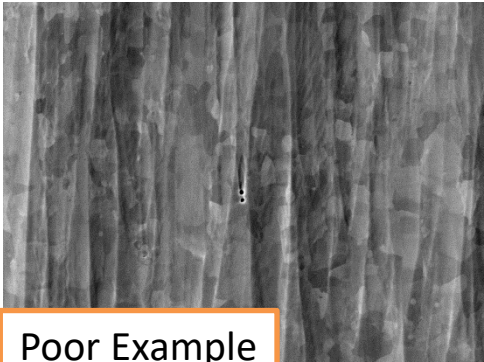


# Sample Preparation (if required)



## Good Example

(A generally good result, although slight beam streaks are present, originating from a hole on the cross-section that acts as a starting point for beam-induced artifacts.)

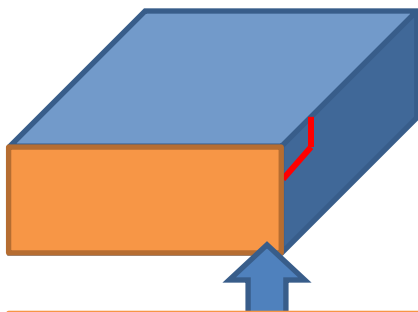


## Poor Example

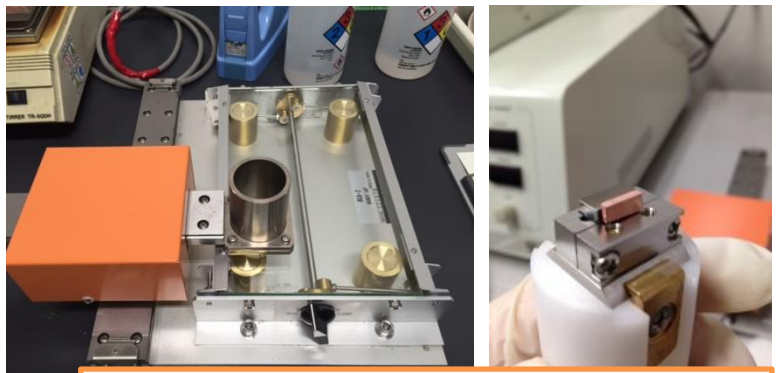
Cross-sections prepared by the CP system may exhibit vertical streaks caused by the  $\text{Ar}^+$  ion beam. To reduce these beam-induced artifacts, apply the following procedures:

- Make the top surface and cross-section of the sample as smooth and flat as possible.
- Ensure that the top surface is well aligned and in close contact with the shielding plate.
- Reduce the acceleration voltage and mill the sample more slowly.

For surface preparation, finish the sample using polishing paper of at least #800 grit or finer. Using a precision lapping machine (Handy Lap) allows the top surface and cross-section to be polished in parallel. When using the Handy Lap, refer to its operation manual.

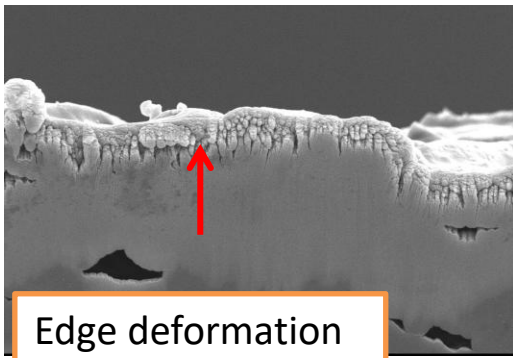
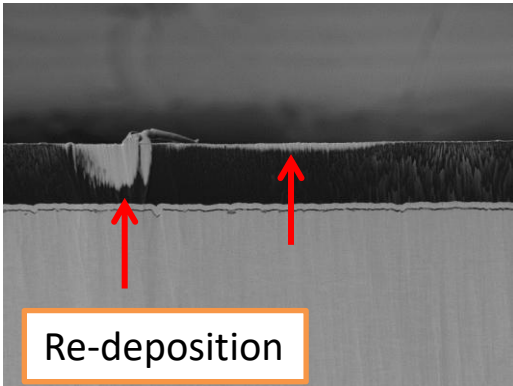


The cross-section surface and the top surface are perpendicular. It is desirable that both the top and bottom surfaces are smooth and flat.



Handy Lap and Holder for CP sample

# Sample Preparation (if required)

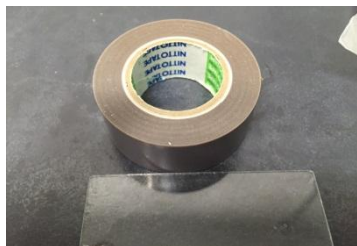


The CP system can produce clean cross-sections; however, near the sample edge (approximately within 100  $\mu\text{m}$  from the top surface), shape deformation and re-deposition of sputtered material are likely to occur.

For samples where surface polishing to remove roughness on the top side is not feasible (e.g., powders, wire-like samples, or when the top surface is the observation target), it is necessary to protect the top surface (e.g., by resin embedding) in order to obtain a clean cross-section.

In some cases, a clean cross-section near the target edge can be obtained by covering the surface near the edge and milling from the backside (the side opposite to the observation surface).

The laboratory provides G-2 epoxy resin for CP use and a vacuum heating/deaeration system. Please consult the staff if you wish to use these facilities.



PTFE tape / Glass slide

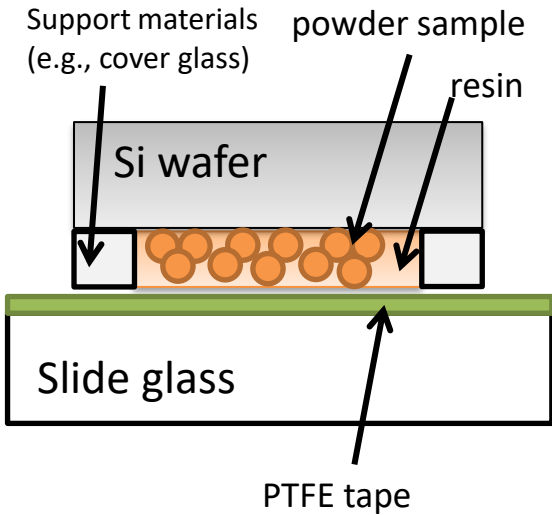


G-2 epoxy resin  
Base resin(white) Hardener(yellow)



Vacuum heating/deaeration system

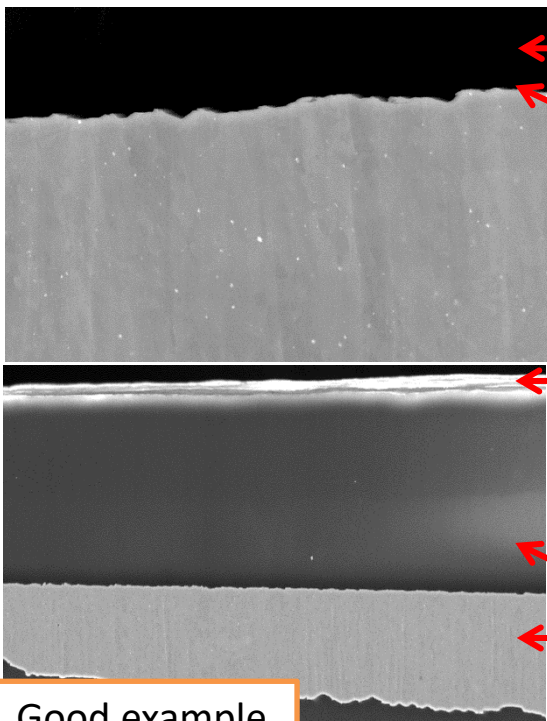
# Sample Preparation (if required)



## How to Use G-2 Resin

1. Mix the base resin and hardener at a ratio of **10:1**, then perform vacuum deaeration.
2. Apply the resin to the sample surface, then carry out vacuum heating and deaeration at **80 °C** for several minutes.
3. To flatten the resin surface, invert the sample and attach it to PTFE tape (see left figure). For small samples, fix the sample onto a support substrate (e.g., Si wafer) before inversion.
4. Thermally cure at either **90 °C for 90 minutes** or **120 °C for 20 minutes**.
5. After polishing the cross-section, set the sample in the CP system.

Example of sample fixation before thermal curing



resin layer

By applying a protective layer, a clean cross-section can be obtained even near the surface edge.

The resin layer acts as a protective buffer, mitigating edge deformation and re-deposition.

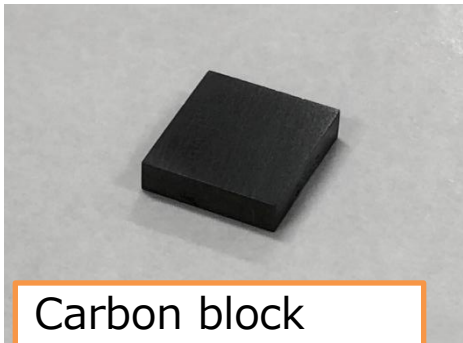
resin layer  
sample

Good example

The key requirement is to prepare a smooth, flat protective layer (approximately 100 μm thick) that can act as a substitute surface to suppress re-deposition. Alternatively, if the sample is fixed on a flat substrate, re-deposition can be reduced by milling from the substrate side.

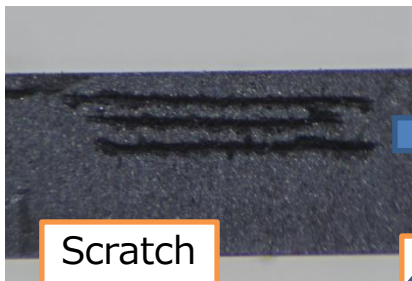
For heat-sensitive samples, applying a gold coating helps dissipate heat during processing. It is also effective to mill at a lower acceleration voltage over a longer time. Using the cryo function or intermittent irradiation mode can further improve results.

# Sample Preparation (if required)

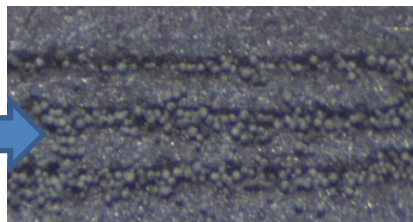


Carbon block  
(sample stage for SEM)

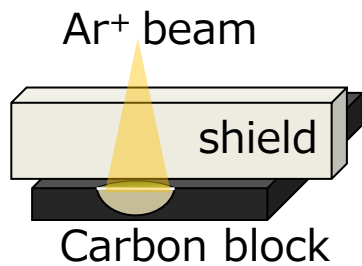
When preparing cross-sections of powder samples, as an alternative to resin embedding, a method of making a scratch on the side of a carbon block and directly embedding the sample into it can be used.



Scratch



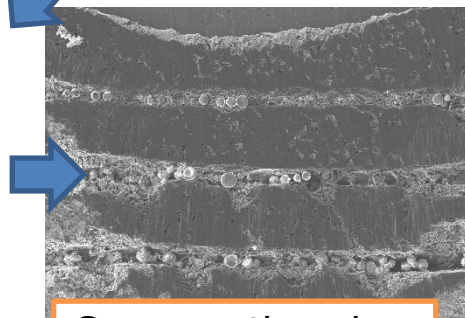
Embedded powder sample



Ar<sup>+</sup> beam

shield

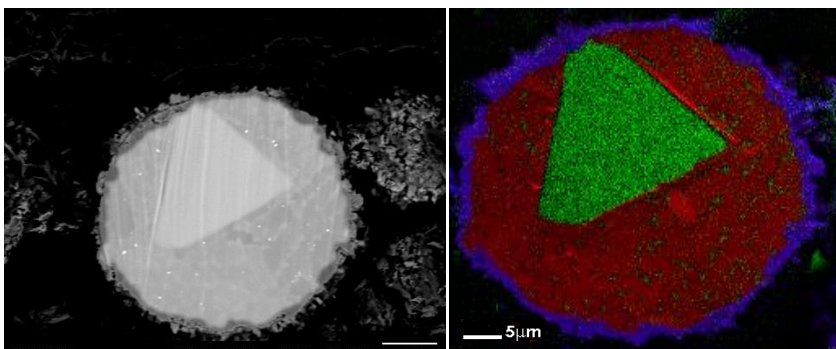
Carbon block



Cross-section view

For relatively large particles or irregularly shaped samples, it is effective to attach the sample onto a thin Si wafer using resin, adhesive, or wax. The cross-section side should then be polished to obtain a flat, and milling should be performed from the Si wafer side.

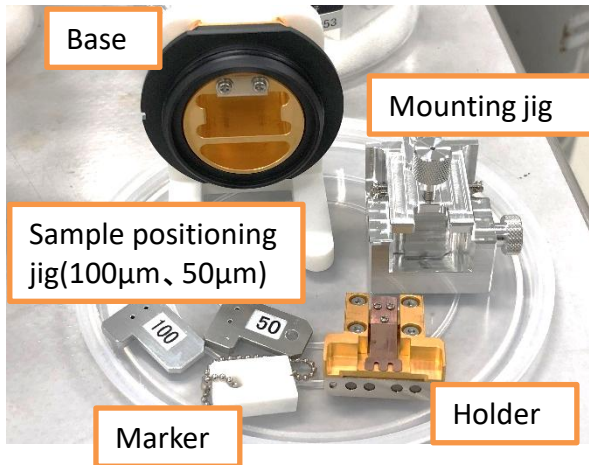
In general, it is preferable to perform milling from the side opposite to the target layer (or region of interest).



Cross-section SEM image & AES mapping for a powder sample

# Sample setup (Normal / large holder)

Normal holder(magnetic) set



Prepare the holder shown in the left-hand figure from the normal and large holder case. The maximum allowable sample sizes are as follows:

Normal holder (magnetic / non-magnetic):

11 mm × 8 mm × thickness 3 mm

Large holder:

20 mm × 7 mm × thickness 3 mm

If the sample is too small, it cannot be fixed with the clip. In such cases, please use resin embedding or fix the sample onto another substrate.

There are two types of shielding plates: magnetic and non-magnetic. They do not significantly affect the quality of the cross-section. In general, the magnetic type is easier to handle.

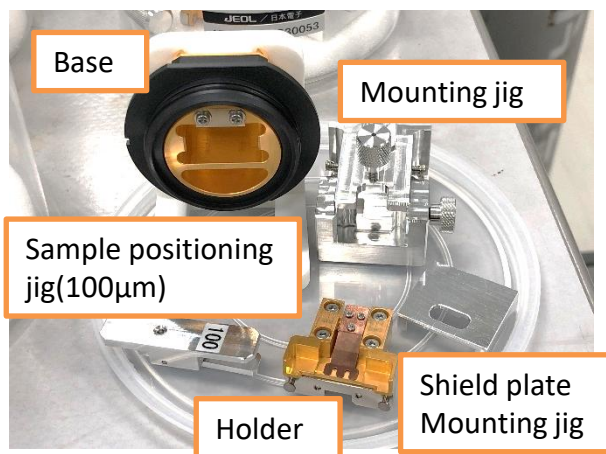
For other special holders, the following shielding plates can be used:

For air-sensitive samples: magnetic / non-magnetic

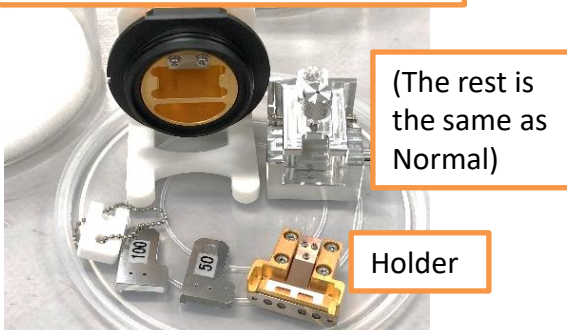
Sample stage holder: magnetic

Positioning holder: magnetic

Normal holder(non-magnetic) set

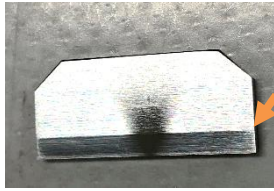


Large holder(magnetic) set



Please bring the required shielding plates or purchase them from the facility. For purchases, please contact the staff.

# Sample setup (Normal / large holder)

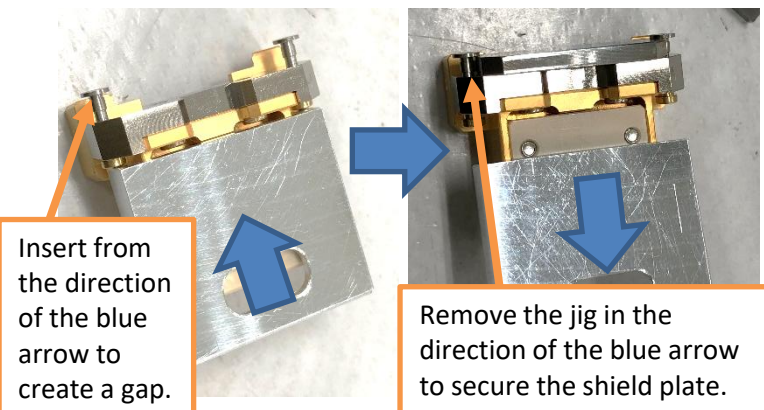
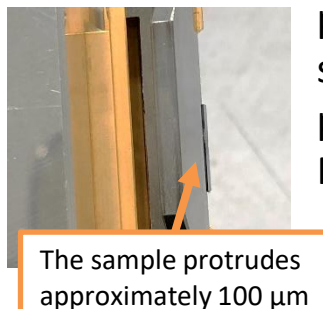
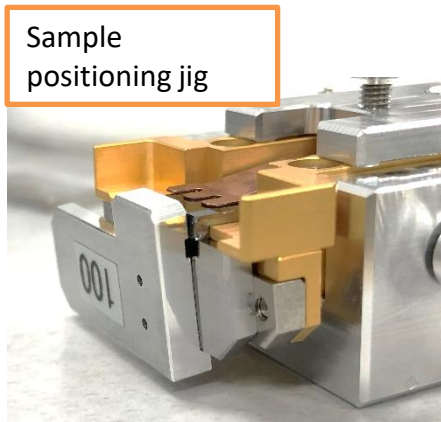
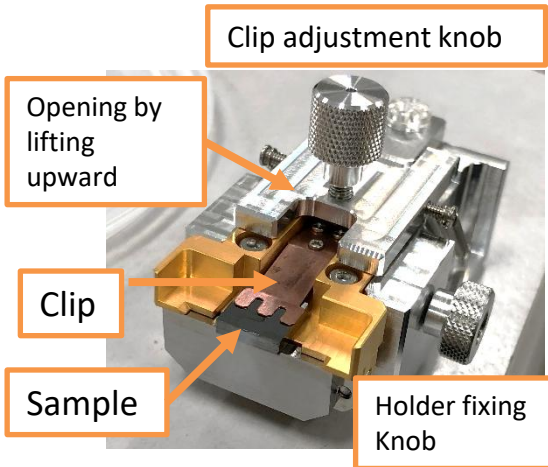


The side with the visible horizontal line is the front side.

Attach the magnetic shielding plate to the magnet section of the sample holder. The side with the visible horizontal line is the front side. Open the clip adjustment knob upward, mount the holder onto the mounting jig, and then close the clip adjustment knob. Turn the clip adjustment knob clockwise to open the clip. Insert the sample so that it slightly protrudes from the shield plate, then turn the knob in the opposite direction to clamp it.

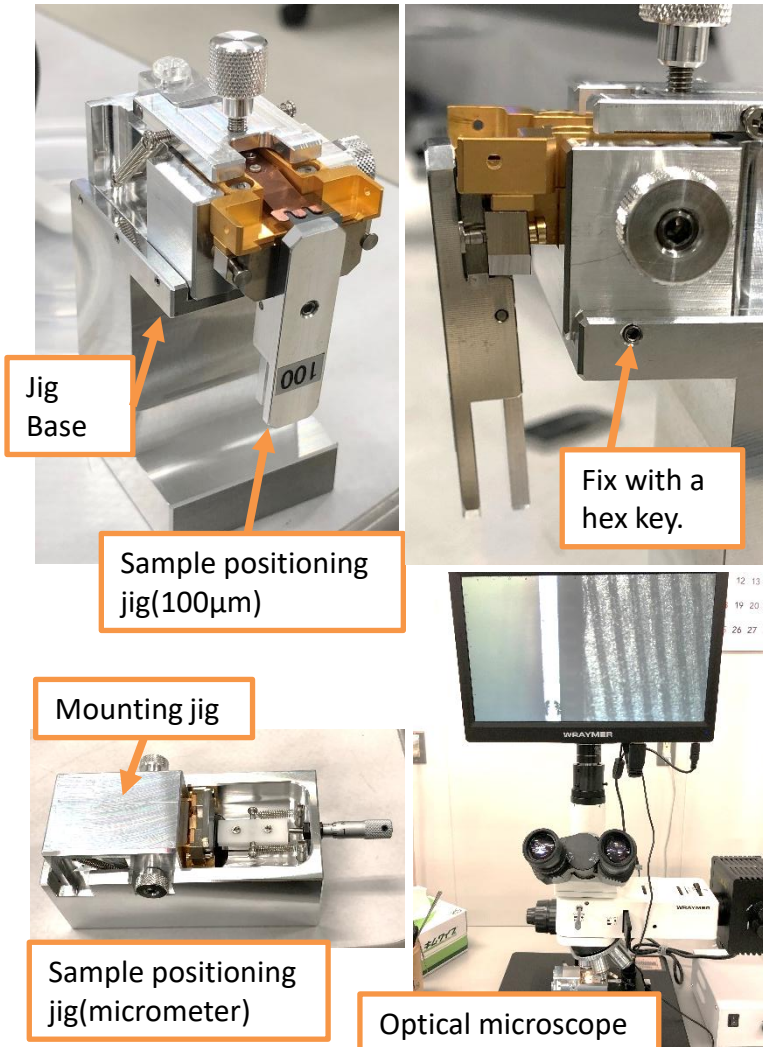
Attach the sample positioning jig to the shielding plate as shown in the left figure. The sample will be pushed inward and adjusted to protrude approximately 100  $\mu\text{m}$  from the shielding plate.

If the adjustment fails, repeat the process. If the cross-sectional shape is distorted, proper 100  $\mu\text{m}$  positioning cannot be achieved. Pre-polishing is required.



For non-magnetic samples, set the shield plate mounting jig on the holder as shown in the left figure, place the shield plate, and then remove the jig to secure it in position.

# Sample setup (Normal / large holder)



For non-magnetic normal holder, set the clip-type sample positioning jig on the holder as shown in the left figure. It is easier to operate if the mounting jig is placed in advance on the jig base (stored in the CCP Box).

For fine adjustment other than 50 µm or 100 µm protrusion, use the sample positioning jig (micrometer , stored in the CCP Box) and an optical microscope. While observing the sample protrusion, rotate the micrometer to push the sample inward and adjust the position.

Once the sample is set, insert the holder into the base as shown in the left figure.



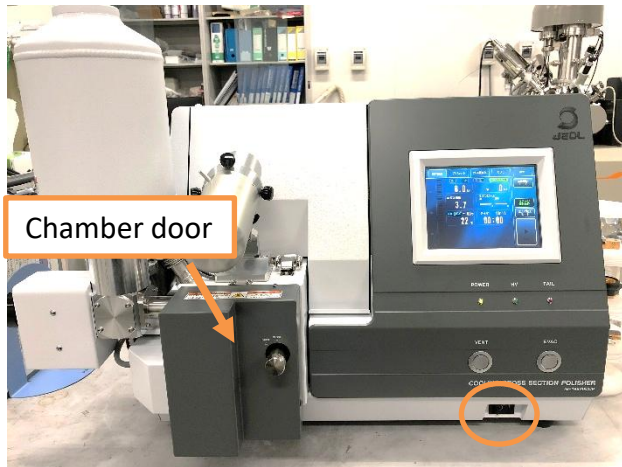
Pay attention to the insertion direction. The side with the base screw is the upper side.



Once this recess is fully penetrated, that position can no longer be used.

Note that the shield plate is a consumable part. Once the lower step is penetrated by the beam, that area can no longer be used. Multiple positions can be utilized by changing the sample location. Typical beam penetration occurs after approximately 8 hours at 8 kV, or 24 hours at 6 kV.

# Equipment setup (Normal / large holder)



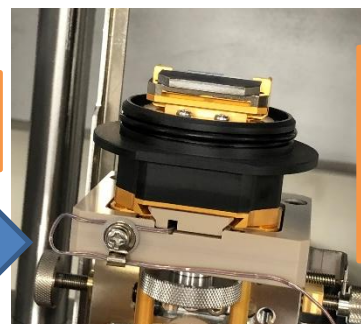
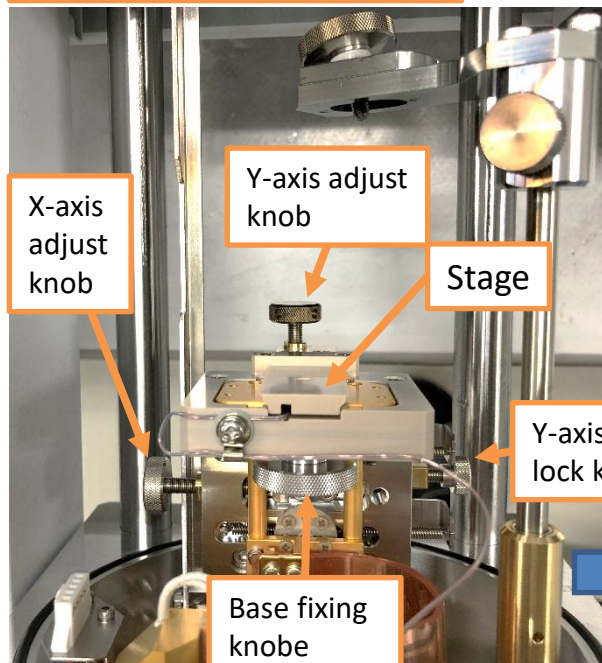
The CCP power supply is normally kept ON. If it is OFF, switch it ON. Next, press and hold the VENT button for about 2 seconds to return the chamber to atmospheric pressure. Once the chamber reaches atmospheric pressure, release the chamber lock and pull the door forward to open it.



When completed, the indicator will turn ON.



Select the tab on the right side of the monitor screen, and press the [English] button to switch the display to English. After opening the chamber, place the base on the stage and tighten the base fixing knob to secure it.

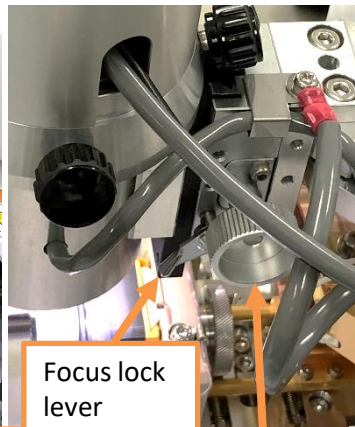


Pay attention to the orientation. The shield plate side must be on the upper side.

# Equipment setup (Normal / large holder)



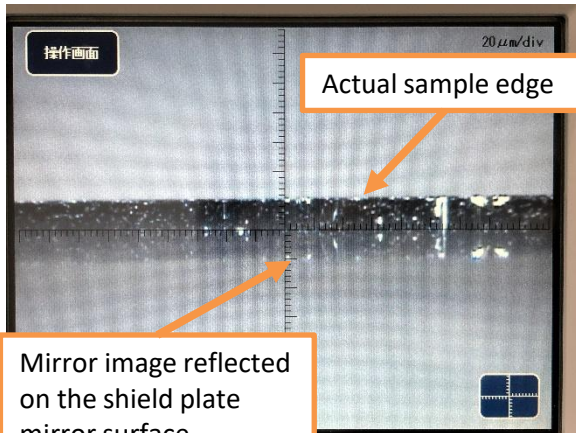
Do not handle the system roughly. Do not touch unrelated knobs; this may cause misalignment.



Focus lock lever

Focus knob

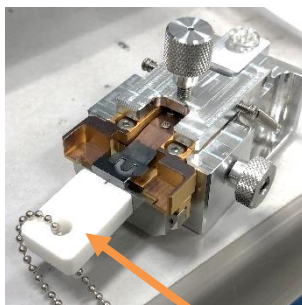
After setting the base, gently raise the microscope upright and select the camera tab on the display. Adjust the focus using the focus knob on the left side of the microscope and observe the sample.



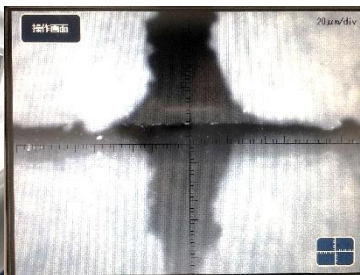
Actual sample edge

Mirror image reflected on the shield plate mirror surface.

Use the X-axis adjustment knob to align the target processing position to the center of the screen. If the processing position is difficult to identify, attaching a marker (magnetic type only) will help. Adjust the Y-axis so that the symmetry axis of the mirror image coincides with the X-axis displayed on the screen. The scale division is 20  $\mu\text{m}$  per unit.



Marker (align the notch to the target position and attach to the shield plate).

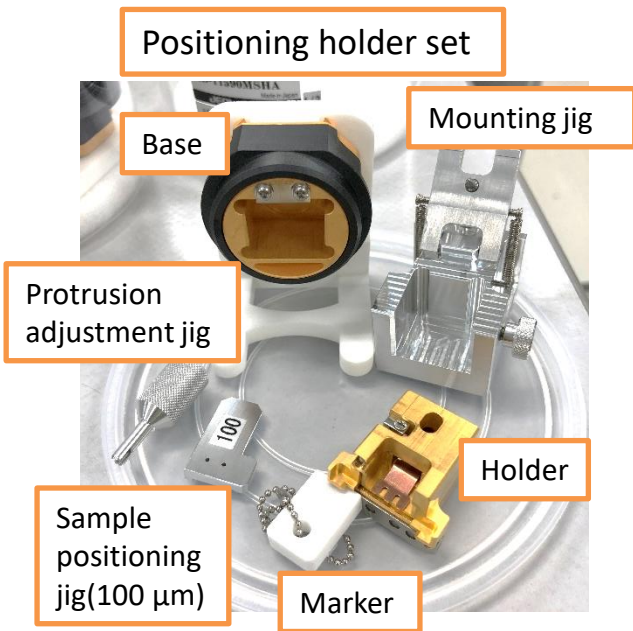


Align the notch of the marker to the center position. Remove the marker after adjustment.

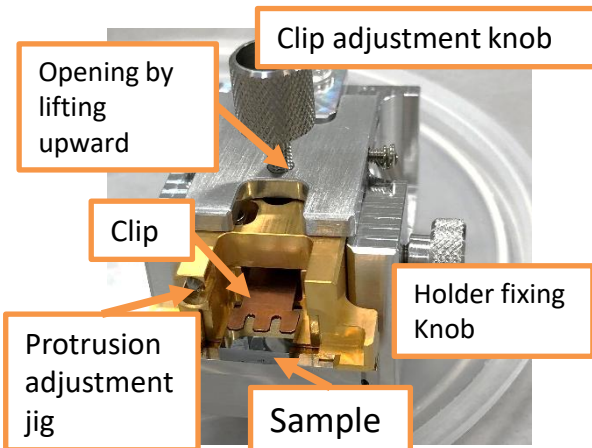
Once positioning is completed, gently lay the microscope down, close the chamber, lock the chamber, and press the EVAC button to start evacuation.

For subsequent steps, refer to "Beam irradiation (P.23–27)".

# Equipment setup (Positioning holder)



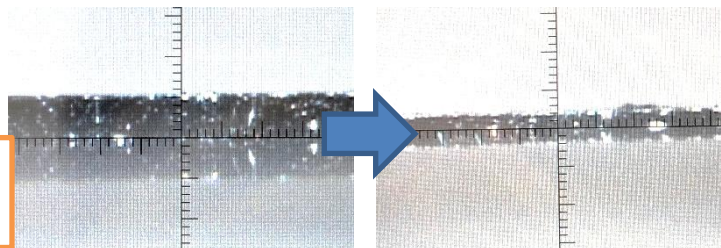
Using the positioning holder, the sample protrusion can be finely adjusted while mounted on the stage. The maximum sample size is **11 mm × 8 mm × 3 mm** (thickness). The protrusion can be adjusted within  $\pm 0.5$  mm from the reference value on the stage. **This holder does not support cryo operation.** A magnetic-type shielding plate is used.



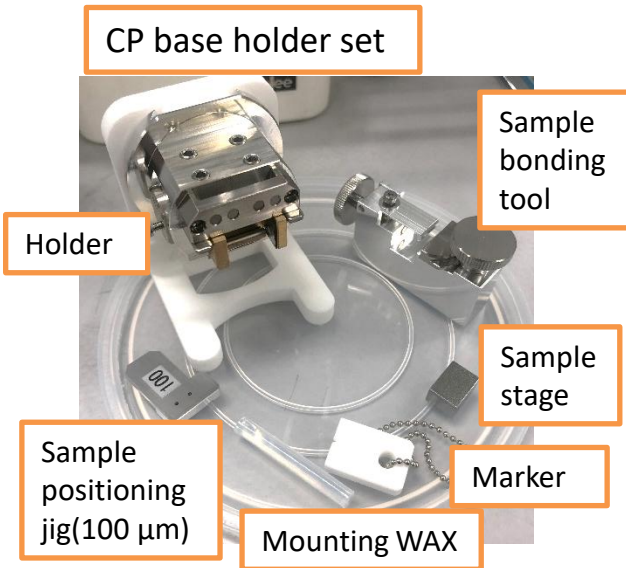
Sample mounting and stage installation are the same as for the normal type. Refer to “Equipment setup (normal/large holder) P.9–13”. First, adjust the sample protrusion to 100 μm using the sample positioning jig (100 μm). After fixing the base on the stage, check the sample edge with the camera. Then adjust the protrusion by turning the knob on the protrusion adjustment jig.



Protrusion adjustment process



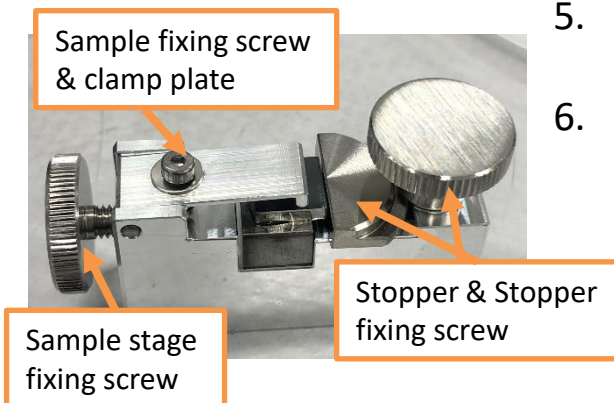
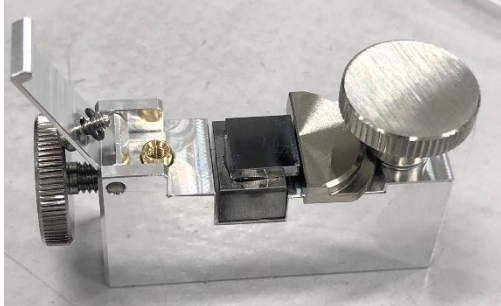
# Sample setup (CP base holder)



A sample mounted on a sample stage for the older CP model can be installed on the holder and processed with the CCP. The protrusion can be adjusted within 2 mm from the edge of the sample stage. The maximum sample size is **11 mm × 10 mm × 2 mm (thickness)**. This holder does not support cryo operation. A magnetic-type shielding plate is used.

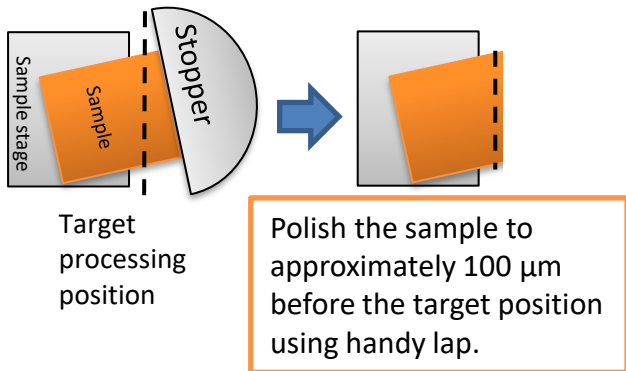
## How to use the sample bonding tool

1. Heat the sample stage to 120 ° C using a vacuum heating/deaeration unit, and apply mounting wax.
2. Place the sample stage on the sample bonding tool and secure it with the fixing screw.
3. Open the sample clamp plate and place the sample so that the cross-section to be processed contacts the stopper.
4. Close the sample clamp plate and secure it with the screw.
5. Heat the entire bonding tool at 120 ° C for approximately 3 minutes.
6. Allow the tool to cool, then remove the sample.

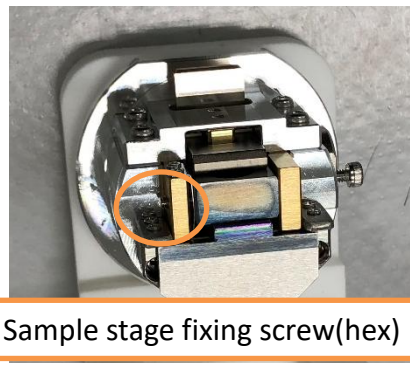
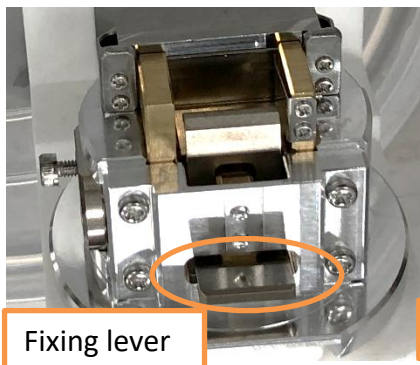


After use, reheat the sample stage to detach the sample, clean it with acetone, and return it.

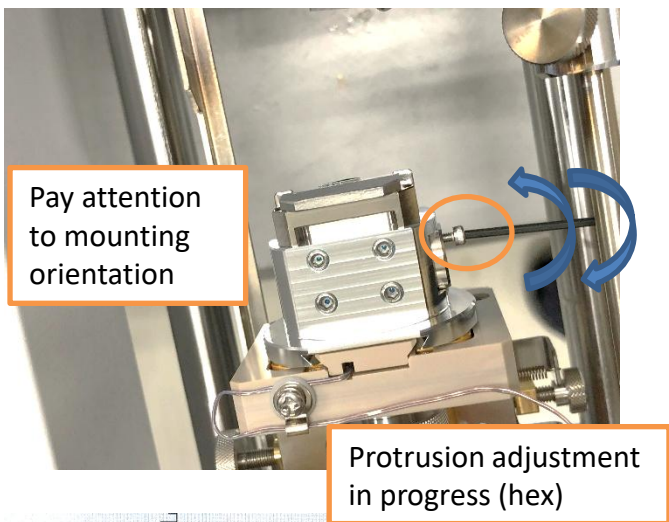
# Equipment setup (CP base holder)



The sample mounting angle can be adjusted by changing the orientation of the stopper. This is useful for positioning the target cross-section.



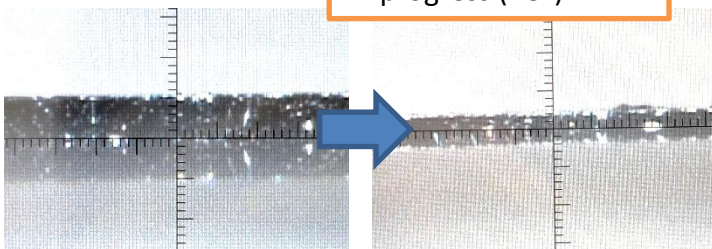
Press the holder fixing lever and set the sample on the holder. Secure it with the sample stage fixing screw. Then attach the shield plate to the holder.



Set the holder on the stage as is. For stage installation, refer to "Equipment setup (Normal, Large) P.9–13".

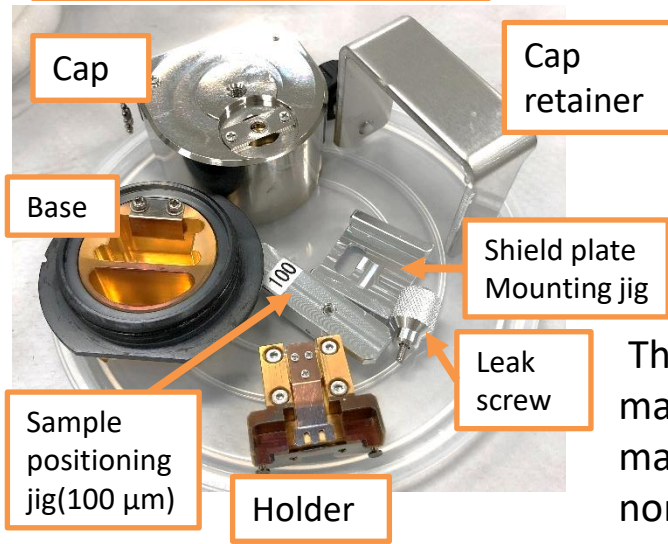
While observing the camera image, adjust the sample protrusion by turning the screw shown in the left figure.

For subsequent steps, refer to "Beam irradiation (P.23–27)" and "After processing (P.28)".



# Sample setup (Transfer vessel holder)

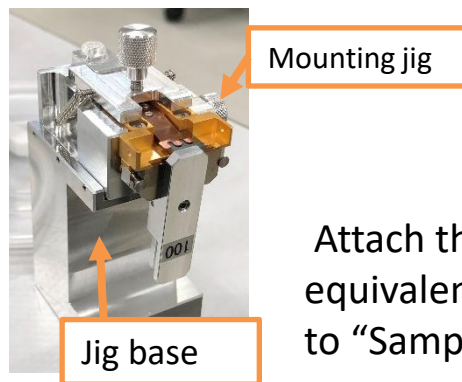
Transfer vessel holder set



Using the transfer vessel holder, samples can be transported without atmospheric exposure from loading to retrieval.

**If the holder is taken out for sample setup, be sure to obtain permission from staff.**

The holder body is the same as the non-magnetic normal holder. When using the magnetic type, remove it from the normal holder case.



The maximum sample size is **11 mm × 8 mm × 3 mm (thickness)**. Prepare the required mounting jig from the normal holder case and the jig base from the CCP Box as needed.

Attach the sample and shield plate in a glove box or equivalent environment. For mounting procedures, refer to “Sample setup (normal/large holder) P.9–13”.



After setting the holder on the base, align the groove position and attach the cap.

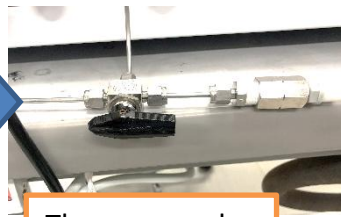
To prevent the cap from coming off during transport, attach the cap retainer as shown in the left figure.



# Equipment setup (Transfer vessel holder)



Three-way valve  
(normal / air leak)



Three-way valve  
(Ar gas leak)

Switch the three-way valve located at the rear of the equipment as shown in the left figure. During leak operation, Ar gas is introduced.

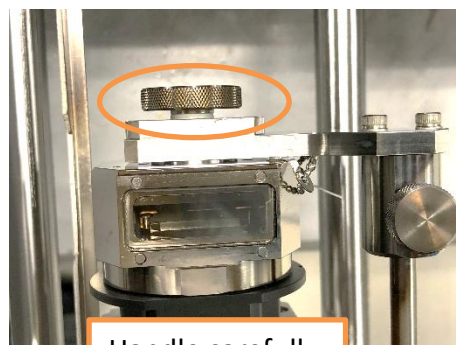
Cap fixing knob



Remove the cap retainer and install the base on the stage. For stage installation, refer to "Equipment setup (normal/large holder) P.12–13". Remove the observation window cover of the cap. Gently raise the microscope and adjust the X- and Y-axes while observing the sample. Then fix the Y-axis using the Y-axis lock knob.



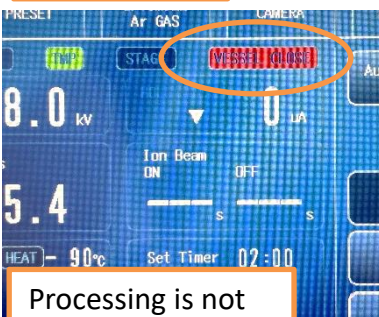
Door control knob



Handle carefully

Align the chamber door control knob to "CLOSE" and pull it forward. Then attach the cap fixing knob to the threaded hole of the cap. When the control knob reaches "CLOSE," the "VESSEL CLOSE" indicator on the operation screen turns red. Beam irradiation is not possible while it is in the CLOSE state.

After installation, gently lay the microscope and close the chamber door. Be sure to attach the observation window cover to the cap before closing the chamber. Lock the chamber and press the EVAC button to start evacuation.



Processing is not possible when the indicator is red.



Ensure the chain does not get tangled when attaching the observation window cover.

# Equipment setup (non-atm. exposure Holder)



The cap detaches inside the chamber as shown

Perform sufficient evacuation, and once a vacuum level on the order of  $10^{-4}$  Pa is reached, carefully push in the door control knob and switch it to “OPEN”. The cap will detach. For subsequent steps, refer to “Beam irradiation (P.23–27)”.

## Sample removal after processing

Refer to “After processing (P.28)”. After processing is completed and the equipment has returned to room temperature, press the VENT button for 2 seconds. The VENT button will blink at 1-second intervals for approximately 1 minute. It will then blink rapidly **for about 8 seconds**. During this period, operate the door control knob (switch to CLOSE and pull it forward) to seal the sample in a slightly reduced-pressure Ar atmosphere. This sealing method is recommended.

After that, the VENT button will remain steadily ON. If sealing is performed at this timing, the sample will be sealed in Ar at atmospheric pressure. Release the chamber lock and open the chamber. Carefully remove the cap fixing knob, then push in the door control knob again and switch it to “OPEN”.

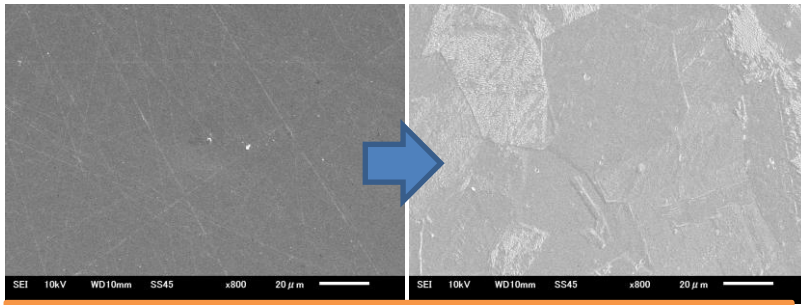


Bring the leak valve into the glove box.

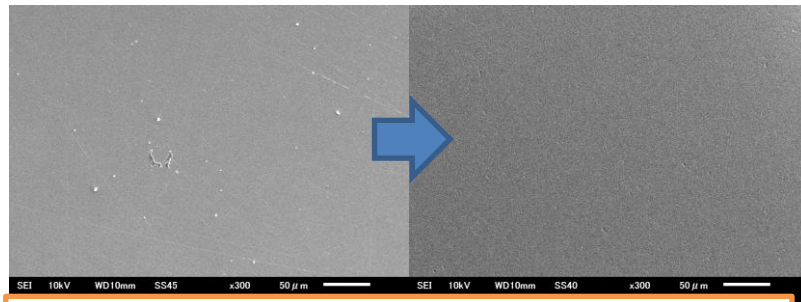
Continue the procedure from step 3 of “Shutdown procedure”. Attach the cap retainer and transport the sample to the glove box.

When opening the cap in the glove box, attach the leak valve as shown in the left figure to release the pressure inside the cap, allowing it to be removed. **After use, promptly return all holders and related components.**

# Sample setup (Large sample rotation holder)



SUS mirror-polished after processing at 70° tilt, 5 kV / 10 min



SUS mirror-polished after processing at 80° tilt, 4 kV / 15 min

The large-area rotating holder enables surface processing of samples. The effect varies depending on the tilt angle and irradiation conditions. It is effective for final finishing after mechanical polishing (removal of polishing scratches, abrasive particles, and damaged layers), as well as for enhancing microstructures by selective etching. It is also useful as a pre-treatment for SEM observation and EBSD analysis.

## Typical processing conditions (for reference)

Removal of fine surface scratches

Voltage: 3–5 kV Tilt angle: 60–80° Time: ~5 min

Removal of damaged layer after mirror polishing

Voltage: 3 kV Tilt angle: 80–90° Time: 3–5 min

Topography formation by selective etching

Voltage: 3–5 kV Tilt angle: 0–60° Time: varies

Prepare the components shown in the left figure. The maximum allowable sample size is  $\phi 40$  mm  $\times$  thickness 15 mm. Two types of sample height adjustment plates are available: For 10 mm type: adjustable for thicknesses of 8–12 mm. For 15 mm type: adjustable for thicknesses of 12–16 mm.

## Large sample rotation holder set

Holder

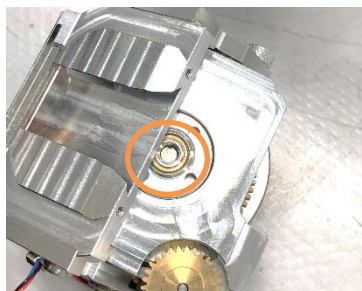
Centering tool

Sample height adjustment plate

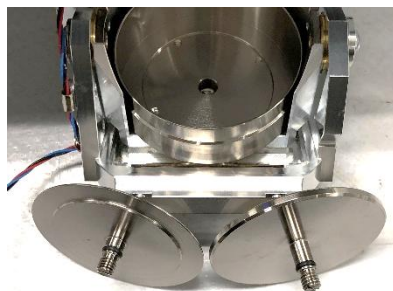
Clamp

For 1-inch ( $\phi 25.4$  mm) resin-mounted samples, the clamp shown in the left figure can be used for easy fixation (thickness range as above). For flat samples or thin specimens, use an SEM sample stage and fix the sample with tape or similar.

# Sample setup (Large sample rotation holder)

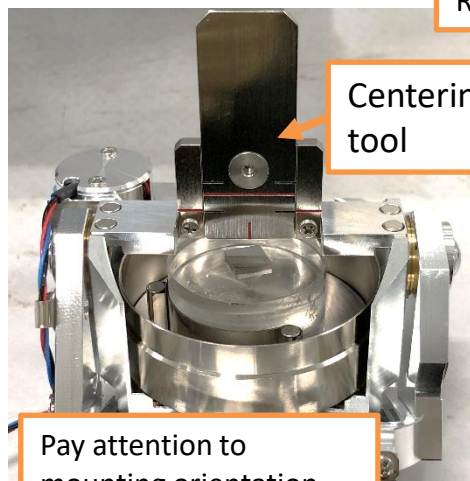


Height adjustment screw



Left: for 15 mm  
Right: for 10 mm

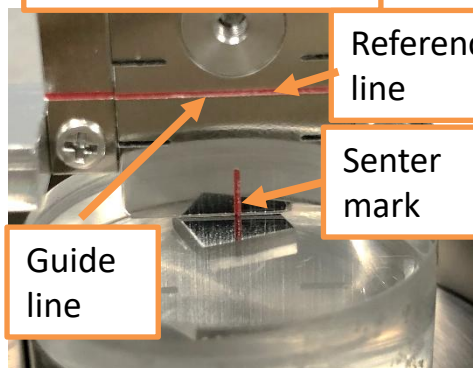
To adjust the height of the sample height adjustment plate or to remove it, turn the screw on the back side of the holder as shown in the left figure.



Centering tool

Pay attention to mounting orientation

Place the sample (with the clamp attached) on the adjustment plate and secure it magnetically. Mount the centering tool on the holder slightly lifted. Lower the indicator plate until it touches the sample. Adjust the sample position so that the processing point aligns with the red center mark. At the same time, adjust the height of the sample so that the reference line and the guide line coincide. (Perform adjustment while keeping the indicator plate slightly lifted. Contact may scratch the sample.)

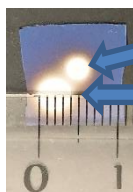


Reference line

Center mark

Guide line

Processing area (depends on tilt angle):



30° : approx.  $\phi$ 2.5 mm

60° : approx.  $\phi$ 4 mm

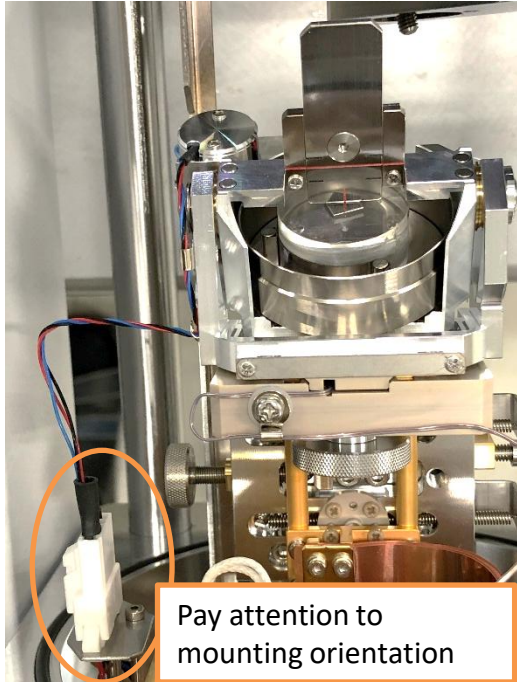
80° : approx.  $\phi$ 10 mm



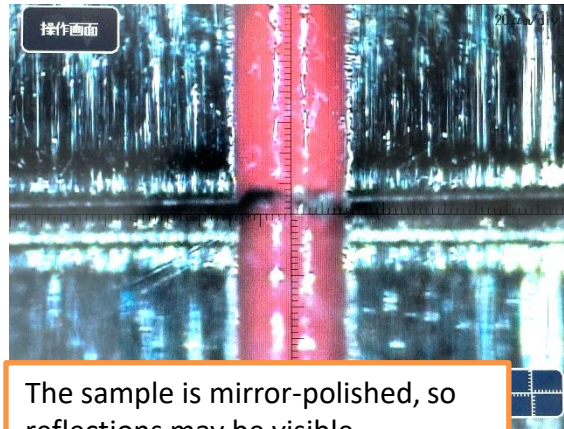
Tilt angle fixing knob (hex)

Loosen the tilt angle fixing knob, adjust the tilt angle according to the processing purpose, and then secure it.

# Equipment setup (Large sample rotation holder)



Open the chamber by referring to “Equipment setup (Normal/large holder) P.12–13”. With the centering tool attached, install the holder on the stage and secure it. Connect the holder connector to the connector port shown in the left figure. Handle the connector with care. On the camera screen, adjust the X- and Y-axes so that the center mark is positioned at the center.



Raise the indicator plate of the centering tool and gently remove the tool. Lay the camera down, close the chamber, lock it, and start evacuation (EVAC).



For subsequent steps, refer to “Beam irradiation (P.23–27)”. For sample removal after processing, refer to “After processing (P.28)”. Do not forget to disconnect the connector.

When the connector is connected, the display switches to the rotating holder mode. Press the button to turn the display green.

# Beam irradiation (Preset & Auto Start)



If fine adjustments are not required, select a beam irradiation condition from the presets and use the automatic start mode. For manual operation, refer to “Beam Irradiation (Manual / Intermittent Mode), pp. 25–26.”



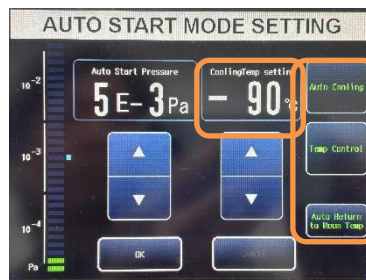
During evacuation, select the Preset tab and choose one of the conditions on the left (1: 8 kV, 2: 6 kV, 3: 4 kV), then apply it to the operation screen.

Return to the operation screen and set the processing time using the timer.

Next, select the automatic start mode on the operation screen. In the automatic start settings window, click the OK button. If the Auto Cooling button is green, cooling will be performed. If cooling is not required, click the Auto Cooling button to turn it white. If cooling is required, set the cooling temperature.



Without cryo:  
Standard operation.



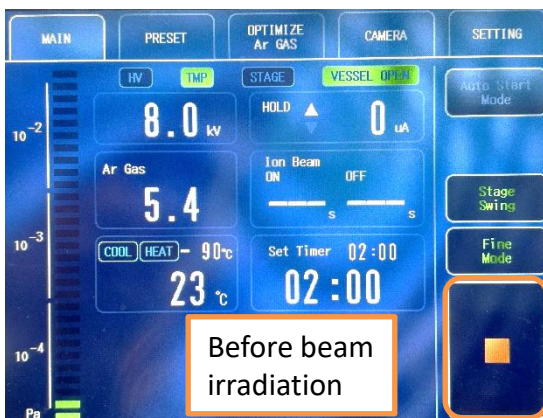
With cryo: Set all indicators on the right to green and set the temperature.

When using the cryo function, please contact the staff in advance. The cryo function is available only for the standard holder, large holder, and air-sensitive holder.

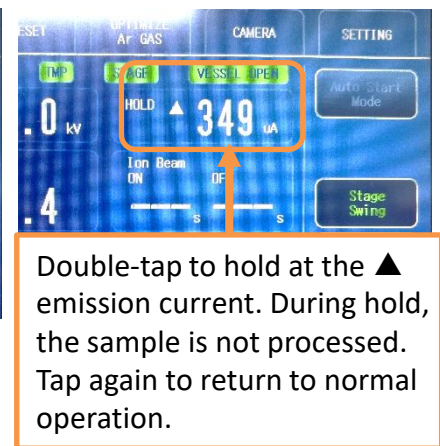
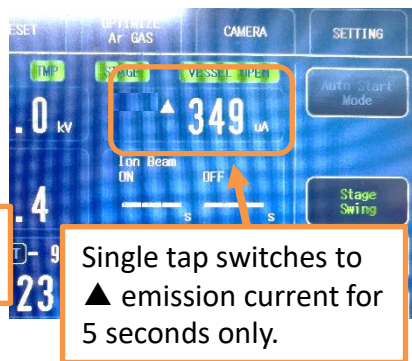
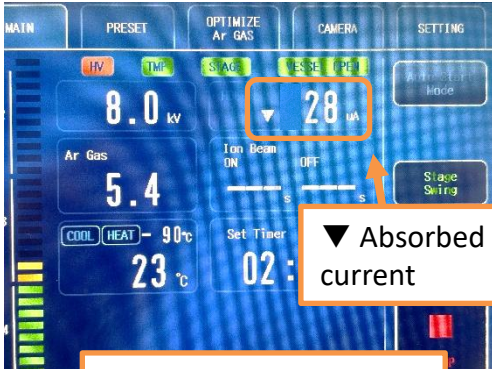
# Beam irradiation (Preset & Auto Start)



When you return to the operation screen, the automatic start mode will be highlighted in green. Press Start to begin the automatic start mode. Once the vacuum reaches  $5 \times 10^{-3}$  Pa, beam irradiation will start automatically. To stop the process, press the Stop button. When the cryo function is enabled, cooling will also start.



Beam irradiation will begin once the set temperature is reached (this takes approximately 60 minutes). After beam irradiation starts, check the emission current and absorbed current, and record them in the logbook. (The current values may be unstable during the first few minutes. Please confirm the values after stabilization. If the current is unusually low or shows abnormal behavior, try the gas optimization described on the next page.)



If the finishing mode is selected (green), an additional 30 minutes of low-voltage intermittent beam irradiation will be performed after processing. This may improve the quality of the cross-section.

# Beam irradiation (Manual & intermittent mode)

First, after evacuation, determine the optimal Ar gas pressure for setting the beam irradiation conditions manually.



Optimal region



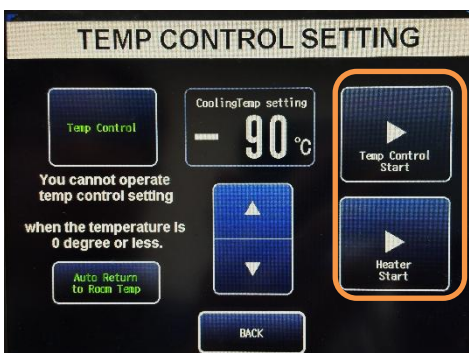
## Gas Optimization (Auto)

After evacuating to below  $5.0 \times 10^{-3}$  Pa, select the Gas Optimization tab on the operation screen. Set the beam acceleration voltage, then adjust the Ar gas pressure range according to the selected voltage. Press Start to begin optimization.

After a 10-minute gas purge, a graph of emission current versus Ar gas pressure will be displayed. The optimal pressure is slightly beyond the peak emission current, at approximately 90% of the maximum value. Touch the graph near the optimal point to set the corresponding pressure value on the operation screen.

Once the optimal gas pressure is set, return to the operation screen, set the acceleration voltage and timer, and press Start to begin processing.

To use the cryo function in manual mode, press the Temperature button before starting, set the cooling temperature, and then press Start Temperature Control. When using the cryo function, please contact the staff in advance. Returning to room temperature can also be started manually from the left-side panel. Once the set temperature is reached, start processing manually.



# Beam irradiation (Manual & intermittent mode)



## Gas Optimization (Manual)

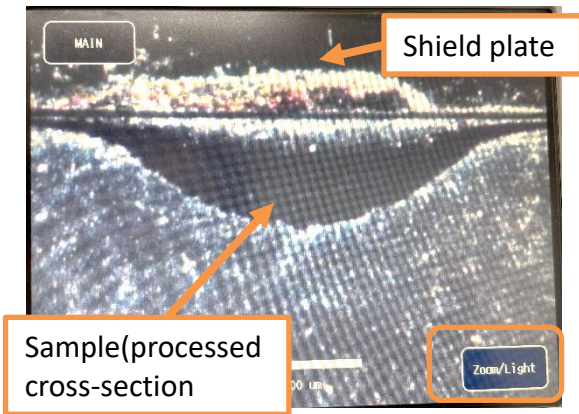
After evacuating to below  $5.0 \times 10^{-3}$  Pa, set the acceleration voltage and timer, then press the Start button. Once beam irradiation begins, tap the ammeter twice to hold it at ▲ (emission current). Tap the Ar gas flow setting and gradually adjust the value. Find the pressure slightly beyond the maximum output, where the emission current is approximately 90% of the peak value, and set it accordingly. Tap the ammeter again to release the hold, and continue processing.



## Intermittent mode

To reduce damage to the cross-section caused by beam irradiation, intermittent ion beam irradiation can be used. Press the Ion Beam ON/OFF button, and set the ON time and OFF time. Press the Start button to begin processing.

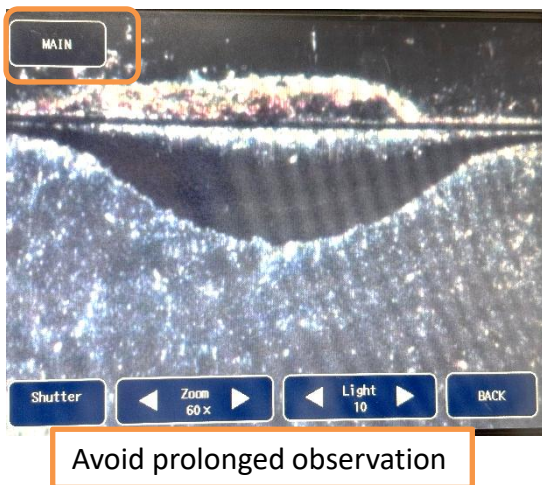
# Beam irradiation (Sample observation during processing)



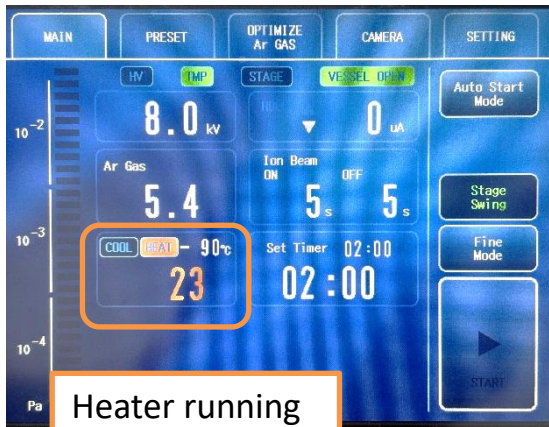
During processing, the stage continuously oscillates in a cradle-like motion. By selecting the Camera tab, you can observe the inside of the chamber (processed cross-section) and monitor the progress. The observation position can be adjusted using the micrometer located on the left side of the instrument. The camera focus can be adjusted by turning the FOCUS knob. The magnification can be changed from  $\times 20$  to  $\times 100$ .

When operating the micrometer, if it begins to rotate freely (i.e., no resistance and no movement of position or focus), do not continue turning it. Instead, rotate it in the opposite direction. After observation, be sure to return to the operation screen. Leaving the camera view on during processing may damage the camera.

After processing is complete, refer to "After processing, p. 28" to remove the sample.



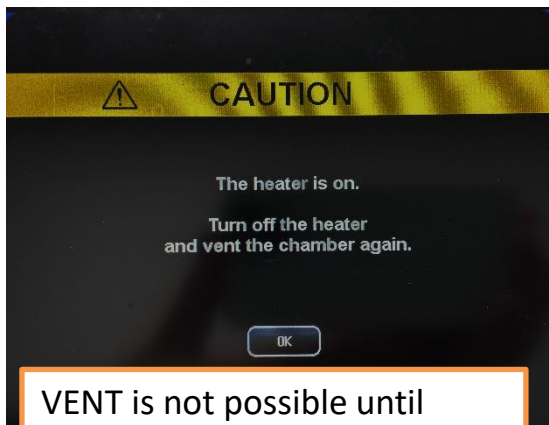
# After processing



When processing is complete, an alarm will sound. If the cryo function is in use, the heater will start automatically and return the stage temperature to room temperature (this takes approximately 30 minutes). **VENT cannot be performed until the temperature has returned to room temperature.**

## Shutdown Procedure

1. Press and hold the VENT button for 2 seconds to return the chamber to atmospheric pressure.
2. Release the chamber lock and open the chamber door.
3. Loosen the base fixing knob and remove the base.
4. Close the chamber door again, engage the chamber lock, and press the EVAC button.
5. Remove the holder from the base, and collect the shield plate and sample.
6. Return the base, holder, and other components to the original case.
7. For transfer vessel holder, return the three-way valve to the air leak side.
8. Fill in all required fields in the logbook, including the end time and processed depth.
9. Leave the CCP power turned on.



VENT is not possible until room temperature is reached

CP base holder: Loosen the sample stage fixing screw (hex).  
Large sample rotation holder: Disconnect the connector.

# Q&A

Please contact the staff if you have any questions or encounter any of the following issues:

- **Beam current does not appear, is lower than usual, or the milling rate is slower than expected after starting processing**

→ Try performing gas optimization (see pp. 25–26) to find an appropriate Ar gas pressure. Gas optimization can be performed with the sample in place. You may also try increasing the acceleration voltage. After starting, it is recommended to check the current after some time has passed. In addition to beam-related issues, improper sample setup may prevent the beam from hitting the sample.

- **An alarm sounds immediately after pressing Start, and processing does not begin**

→ The timer has not been set. After setting the timer, press the Reset button to apply the settings.

- **The cryo function was used, but the set temperature could not be reached, and processing could not start.**

→ To restart, first activate the heater and return the system to room temperature. Refill the liquid nitrogen tank, then set the cooling conditions again. It is recommended to restart with a slightly higher (less aggressive) target temperature. When cooling below  $-120^{\circ}\text{C}$ , do not use automatic start. Instead, disable temperature control and start cooling only. After reaching the target temperature, start processing manually to avoid errors.

- **How long does cooling or returning to room temperature take?**

→ This depends on the system condition, but typically the temperature decreases as follows (in ~10-minute intervals):

$25^{\circ}\text{C} \rightarrow 0^{\circ}\text{C} \rightarrow -30^{\circ}\text{C} \rightarrow -60^{\circ}\text{C} \rightarrow -77^{\circ}\text{C} \rightarrow -92^{\circ}\text{C} \rightarrow -102^{\circ}\text{C}$  (~60 min) →  $-110^{\circ}\text{C} \rightarrow -116^{\circ}\text{C} \rightarrow -120^{\circ}\text{C}$  (~90 min)

Returning to room temperature from  $-120^{\circ}\text{C}$  takes approximately 45 minutes.

- **An error occurs indicating abnormal discharge and voltage cannot be applied**

→ The ion gun requires internal cleaning. Please contact the staff.

Recovery may take approximately 2 hours.

- **Can a previously milled area be processed again?**

→ Yes, it is possible. However, slight misalignment from the first milling may cause visible beam streaks near the edges. If the protrusion is slightly reduced compared to the first milling, re-deposition may occur more easily. If reprocessing is required, it is recommended to re-polish the cross-section before milling again.