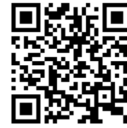
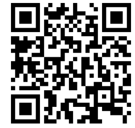
2026/4/28 update


Rules video


施設のルール
動画

Auger Electron Spectroscope(AES) Basic manual Acquisition section

Laboratory of XPS analysis

Contact K. Suzuki Ext. 6882
S. Yoshida Ext. 6882

Caution

Please read carefully and follow these rules.

- No outdoor shoes, food, or drinks in the lab.
- Report equipment malfunctions to facility staff immediately. Emergency contact info is by the lab door.
- Handle equipment with care.
- Do not remove lab items without permission.
- Manage your valuables. Lock the lab after use on holidays or at night.
- When moving the stage, check the analysis chamber carefully.
- Restore software and hardware settings to default after use.
- Do not connect personal USB drives or any external storage devices directly to the instrument control PC. Use the laboratory-designated USB drive and transfer data via the analysis PC, or access the data through the shared folder on the analysis PC. Do not touch items in the lab with bare hands. Clean any tools you soil.
- Reserve in advance and use the equipment during your time slot. Cancel reservations by the previous day. Same-day cancellations are invalid. For extensions, add reservations on the same day.
- Your lab is responsible for equipment issues during use. Inform your supervisor about usage.
- Contact staff and attend training before using equipment for the first time.
- For reactive, large, fragile, or gas-emitting samples, consult staff before use.
- Contact staff for permission before using a transfer vessel.

Before using

Please fill in the usage logbook with the date, start time, your name, laboratory name, analysis chamber vacuum level (before use), sample information, and ARIM theme number (if applicable).
Please do not misplace the pen.

Analysis chamber vacuum gauge

Please check the vacuum levels of the analysis chamber and the ionization chamber. If the values are significantly degraded, please contact the staff.

Normal values:
Analysis chamber: 10^{-7} to 10^{-8} Pa
Ionization chamber: 0.1 $\times$ 10^{-2} Pa

The AES PC and software are kept running at all times. Please turn on only the display. If the SEM software or Auger Master is not running, please restart them.

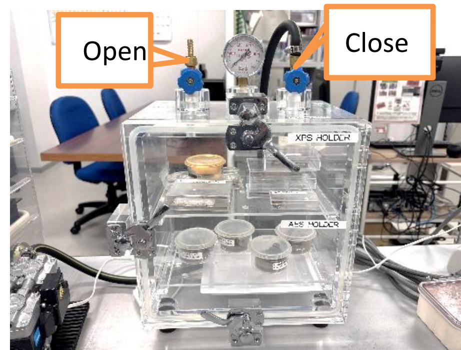
PC for AES analysis

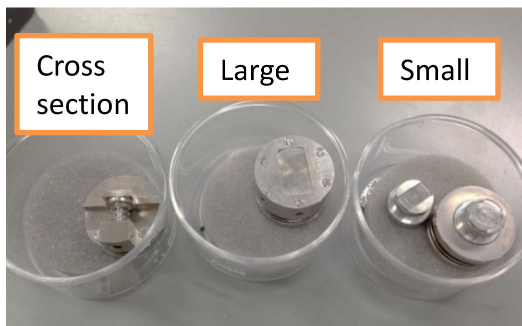
The AES analysis PC and the instrument control PC are connected via a network. Open the “AES-PC Shortcut” icon on the analysis PC’s desktop to access and retrieve data. To edit data on the instrument PC, open “VMware Workstation” on the analysis PC’s desktop, then launch “Auger Master Processing” on the Linux OS. The data directory on the instrument PC is [/home/aes/AESData/Desktop]

Sample preparation

In Auger analysis, contamination (surface pollution) strongly affects the results. Please prepare the cleanest possible sample surface.

- Store samples in an air-free (non-exposed) environment whenever possible.
- Perform ultrasonic cleaning (e.g., pure water, ethanol, acetone), dry immediately with an air gun, and, if possible, heat the sample using a hot plate.
- For cross-sectional observation, use a cross-section polisher (CP) to obtain a smooth and clean surface.
- Remove dust and debris thoroughly using a blower.
- Cleaning and heating the sample holder before use is also effective for reducing contamination.
- Mechanical methods such as fracturing the sample with a knife or pliers, or scraping the surface, can also be effective.





AES sample holders are stored in a vacuum desiccator. The pump is always running, so open and close the air-side and pump-side valves to retrieve them.

The following three types of holders are available:

- **Small holder** (sample size: thickness ≤ 4 mm, diameter ≤ 12 mm)
- **Large holder** (sample size: thickness ≤ 5 mm, diameter ≤ 20 mm)
- **Cross-section holder** (sample size: thickness ≤ 7 mm, 11 mm × 10 mm)

The large and cross-section holders have a tilt angle limitation and can only be tilted up to 55° .
Use the small holder for EBSD measurements.

Sample preparation

Remove the side screw of the small holder to detach the lid. There are two types of lids available.

Fix the sample by clamping it between the holder base and the lid. If the sample is thick, disassemble the lid before inserting the sample.

- After fixing, gently shake the holder to confirm that the sample is securely fixed.
- Tighten all screws firmly and take care not to lose them.

The large holder can also be disassembled. There are three types of lids available.

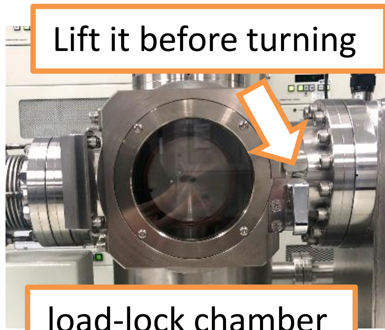
For the cross-section holder, clamp the sample as shown in the figure on the left. Positioning the sample so that it is in contact with the bottom of the holder improves stability.

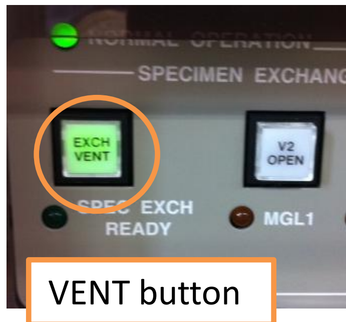
- Fix the sample so that the cross-section slightly protrudes from the holder.

If the fixation is insufficient, or for powder samples, use carbon tape or similar materials to secure the sample. Clean the holder after use.

- If sample drift is a concern, use a paste (e.g., conductive paste). After application, evacuate the sample for several hours.
- Available fixing materials include carbon tape, silver paste, carbon paste, copper tape, double-sided tape, and instant adhesive.

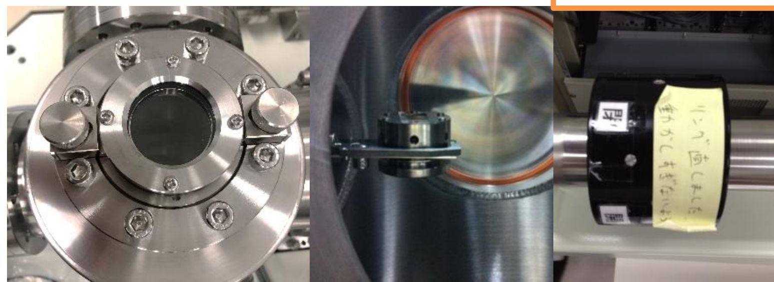
Sample loading





Release the lock of the load-lock chamber door. Press the VENT button to bring the load-lock chamber to atmospheric pressure.(The VENT button will light up green when pressed.)

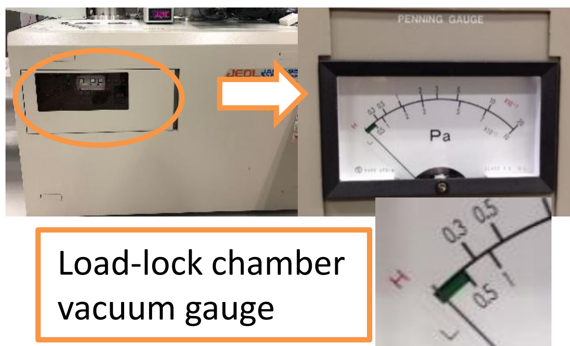
Insert the fork into the groove on the bottom of the holder.



You can also view the inside from the top of the load-lock chamber.

Do not over-rotate

Insert the groove on the top of the holder into the fork inside the load-lock chamber, then rotate the magnet ring so that "CLOSE" is facing upward. Remove the fork, close the door, and lock it. Make sure the magnet ring is positioned at the rear end, then press the VENT button again to evacuate the load-lock chamber.



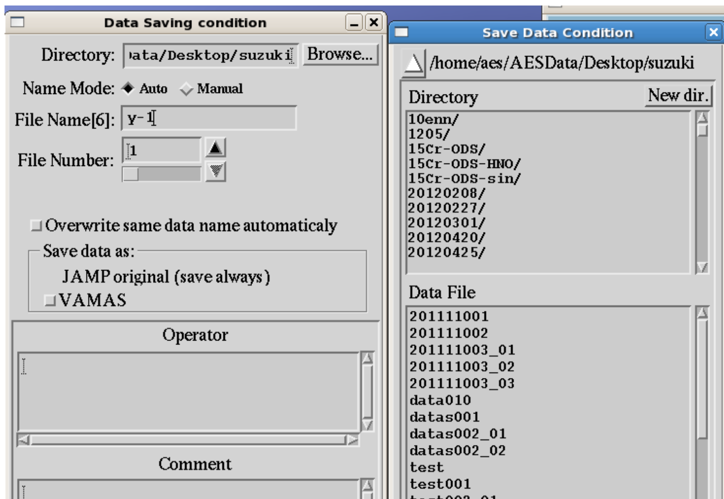
Further evacuation is still possible.↑

Continue evacuation in the load-lock chamber for a sufficient time. Guidelines:

Metal plate: approximately 30 minutes
Powder sample: approximately 2 hours

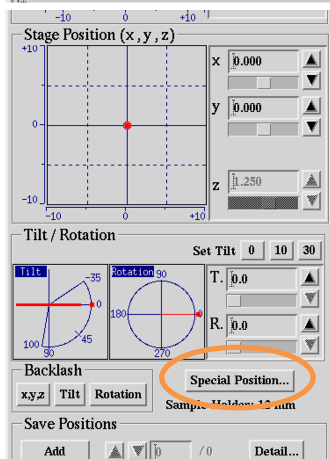
Open the operator panel door of the instrument and check the vacuum level of the load-lock chamber. Continue evacuation until the gauge needle reaches the end.

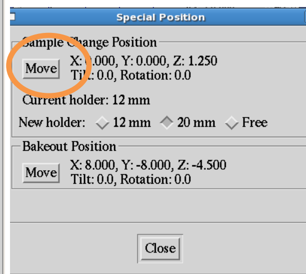
Sample loading



While evacuation is in progress, set the data saving conditions in Auger Master. Go to **File → Saving Condition**, and specify the save location and file name.

- Image and spectral data are automatically saved with the file name plus a sequential number.
- The file name must be within 6 characters.
- Use only alphanumeric characters (letters and numbers) for directory and file names.

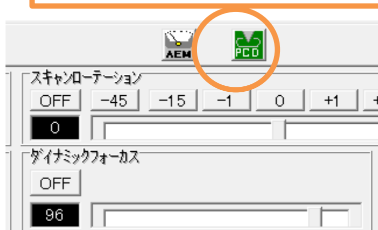




In Auger Master, select **Observation → Sample Manipulation**. Click **Special Position**. In the pop-up window, click **Move** under Sample Change Position. Select the holder type from New Holder, then click **Close**.

- Small holder → 12 mm
- Large / cross-section holder → 20 mm

PCD icon
Green: In Gray: Out

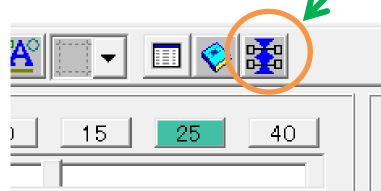


Make sure that:

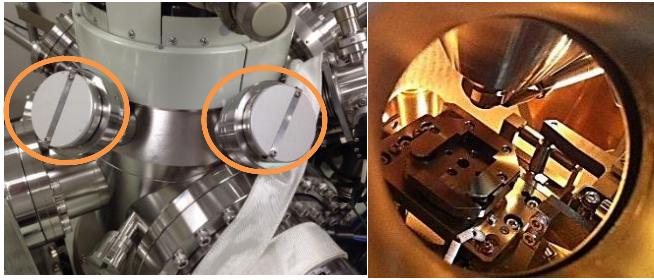
- PCD is inserted (icon shows green: In / gray: Out)
- The secondary electron detector is OFF

If the SEM control panel is not displayed, click the corresponding icon to open it.

Secondary electron detector



Sample loading



Remove the viewport cover and check the inside of the analysis chamber.

Confirm that the magnet ring is fully at the rear position, then press the V2 button to open the V2 valve.(The valve will not open unless the magnet ring is fully retracted.)

While observing the chamber, push the magnet ring forward to transfer the sample onto the stage. Rotate the magnet ring to “OPEN,” confirm that the hook is released, and then retract the magnet ring. Press the V2 button again to close the V2 valve, and reinstall the viewport cover.

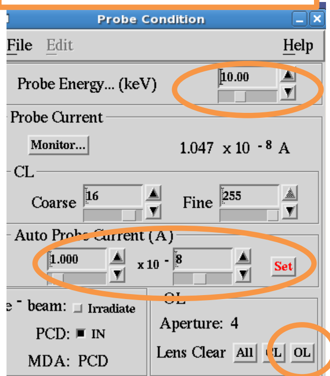
Check the vacuum level of the analysis chamber.

If the pressure is worse than 5.0×10^{-6} Pa, retrieve the sample and re-evacuate it in the load-lock chamber.

If the pressure is better than 5.0×10^{-6} Pa, continue evacuation until it reaches the 10^{-7} Pa range.

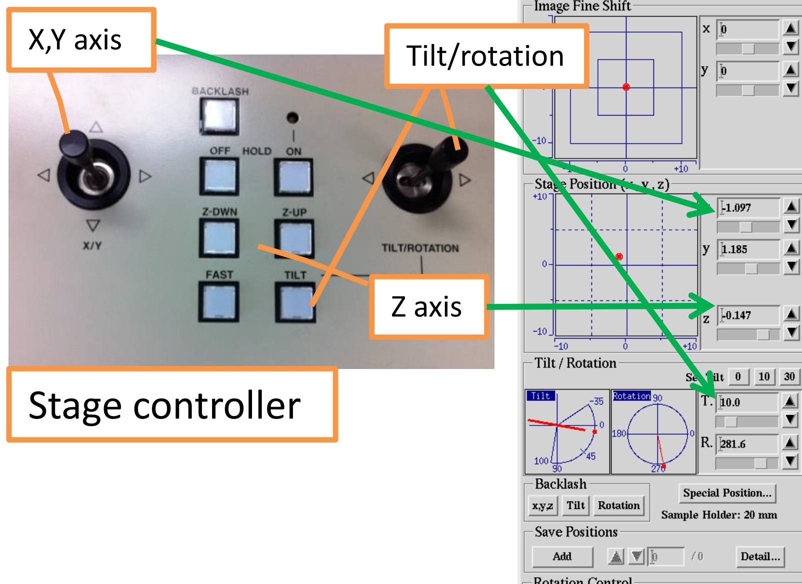
Sample observation

probe condition



Select **Observation → Probe Condition**. Enter the electron beam acceleration voltage in **Probe Energy** and the current in **Auto Probe Current**, then click **Set**.

- For general use, **10 kV / 10 nA** is recommended.
- Click **OL** at the lower right to demagnetize the lens. When changing beam conditions, first adjust the focus, then repeat **demagnetization → refocusing** several times until the focus no longer shifts.
- A higher probe current improves the S/N ratio in Auger analysis.
- For EBSD, **15–20 kV** is recommended.
  The probe current should be around **30 nA** at low magnification, and slightly lower at high magnification.
- If the current exceeds **20 nA (at 10 kV)**, the objective aperture number must be changed.
- For observation of very small areas, reduce the probe current and use **SEM mode No. 3**.

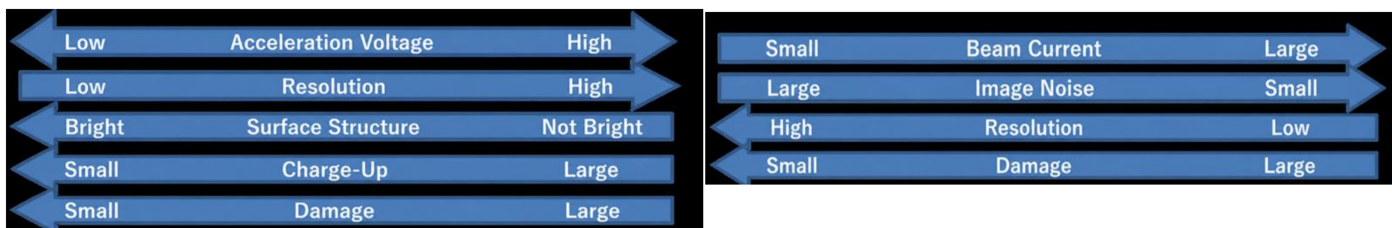


After setting the voltage and current, set **PCD** to **Out** and turn the **secondary electron detector ON**. At the same time, the analysis chamber vacuum gauge will turn OFF. The SEM image can then be observed.

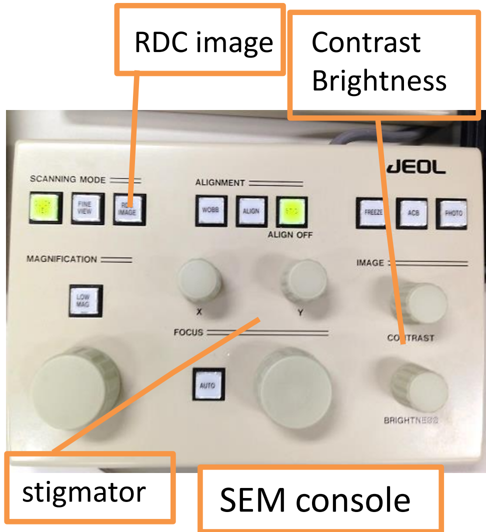
Move the stage to the desired observation area.

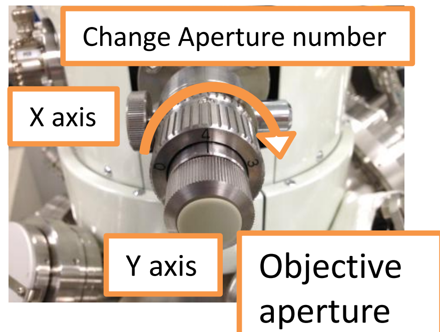
When making large movements, take extreme care to avoid the sample colliding with any internal components.

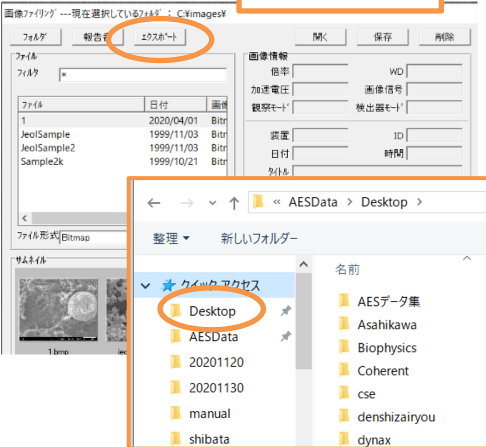
Relationship between electron beam conditions and parameters



Beam alignment and imaging







Perform alignment of the electron beam.
At the observation area, switch the scan mode to **RDC Image**, and adjust magnification, focus, contrast, and brightness. For observations above **5,000×**, additionally adjust **stigmator X/Y**.
If the stigmator is misaligned, the defocused image will appear stretched in one direction.
Defocus the image and check that the blur is circular (concentric).
For observations at tens of thousands magnification, also adjust the **objective aperture X/Y** (especially after changing voltage or current).
Turn **HT Wobbler ON**, and adjust the objective aperture X/Y so that the image position does not shift during periodic defocusing. After adjustment, turn the wobbler **OFF**, and readjust focus and stigmator.
The objective aperture number is normally set to **No. 4** for high-magnification observation.
Using a smaller aperture number allows a higher probe current.
When changing the aperture number, adjustment of the objective aperture X/Y is also required.

SEM Console Functions

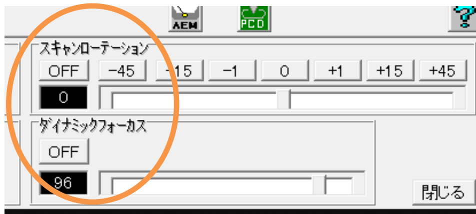
FINE VIEW: Reduces scan speed to obtain a higher-quality image. Two speed levels are available; press the button to toggle.
FREEZE: Freezes the image after one scan cycle is completed.
PHOTO: Starts FINE VIEW, then automatically freezes after scanning and opens the SEM image save window.

Click **Export**, select **Desktop** from Quick Access, and save the data in your folder on the AES-PC.
Image data on the AES-PC can be accessed via:
AESdata → Desktop → your folder on the AES-PC desktop.

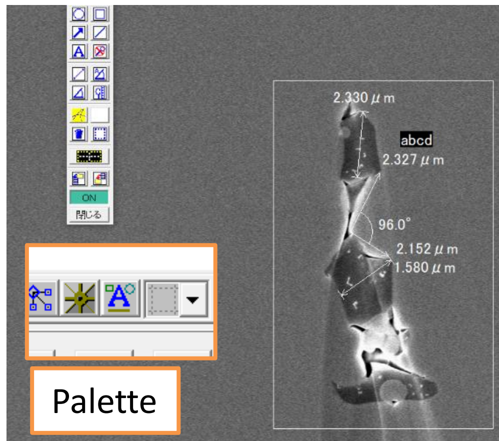
Observation Functions

SEM mode number

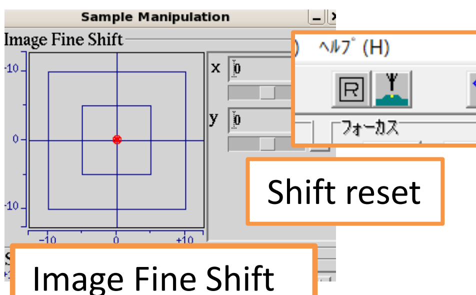
If the probe current is high (around 10 nA), set the **SEM mode number** to 0. If the probe current is low (around 0.1 nA), set it to No. 3 to obtain a clearer SEM image.



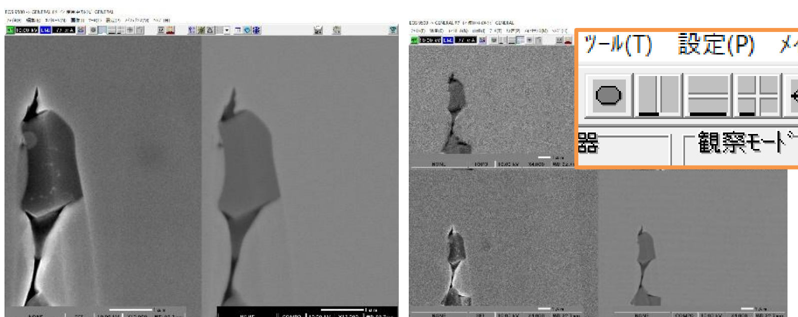
Turning **Scan Rotation ON** and adjusting the scroll control will rotate the image.
Turning **Dynamic Focus ON** and adjusting the scroll control helps maintain focus across samples with height variations.



Click the **Palette** button to add text annotations to the SEM image or measure object sizes using a ruler tool, and save the results.

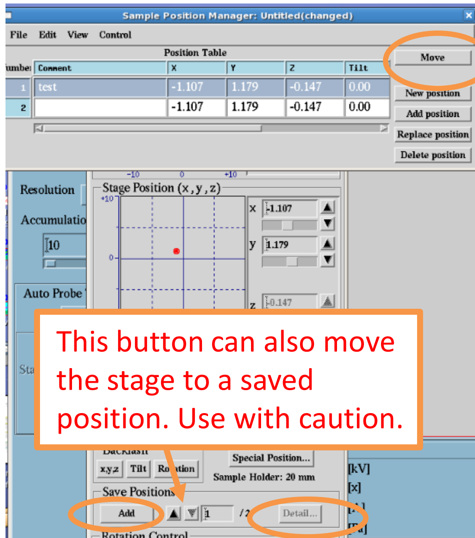


You can move the image by dragging with the mouse without moving the stage.
The movable range is limited to the **Image Fine Shift** range in Sample Manipulation (approximately ±7 µm in X/Y). To return to the initial position (origin), Click the icon shown in the figure on the left.



Two-screen and four-screen display modes are available.
You can view **secondary electron (SEM) images** and **backscattered electron images** simultaneously.(See p. 13)

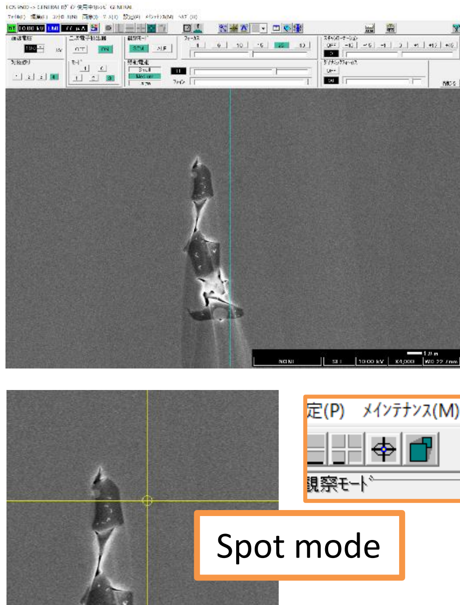
Observation Functions



To save the current stage position, click **Add** in Save Positions. Multiple positions can be stored. To recall a position, click **Detail**, select a number from the table, and click **Move**.

After use, delete all position data.

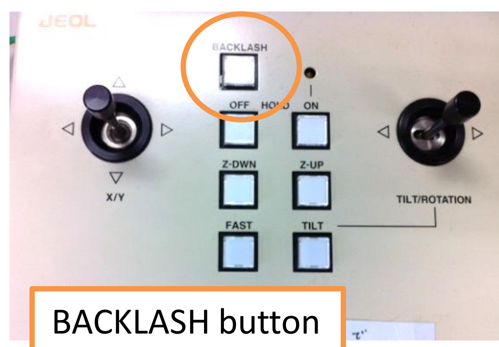
Use this function with care.



Press the **Spot** button to display a blue cross on the image. Press it again to turn the cross yellow and switch the beam from scan mode to **spot mode**. In spot mode, the beam is irradiated only at the center of the cross, and the image is frozen during this time.

At low magnification, the spot position may slightly deviate from the designated position.

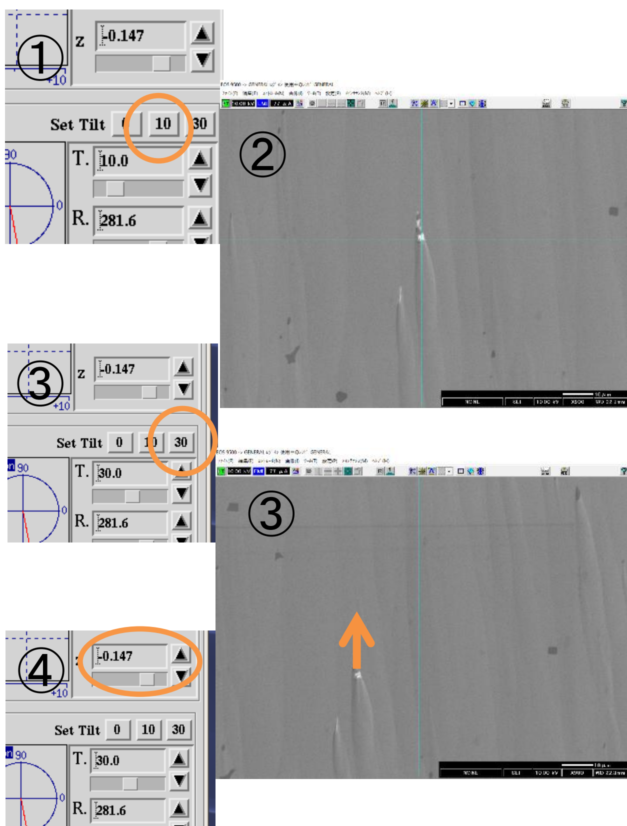
In spot mode, you can perform simple Auger point analysis. Change the analysis point setting to **Scan**, and move the cross to the desired location.



If the image drifts during observation:

- Press the **BACKLASH** button to eliminate backlash.
- Wait for a while after introducing the sample into the analysis chamber.
- If charging occurs, adjust the voltage, current, or stage position.
- Re-mount the sample if necessary.

Eucentric adjustment and BSE imaging



When performing Auger analysis or using the Ar⁺ ion gun

Eucentric alignment

①Tilt the stage to **10°** .
②At the observation point, move a visible target to the center of the screen using **X/Y**.
③Tilt the stage to **30°** , and check whether the target moves up or down on the SEM image (ignore lateral movement).
④If the target moves upward, lower the **Z** position.
If it moves downward, raise the **Z** position to return the target to its original position.
Repeat steps ①–④ while gradually increasing magnification from around **50× to ~1500×**.
(Final adjustment should be done at **30° tilt**.)

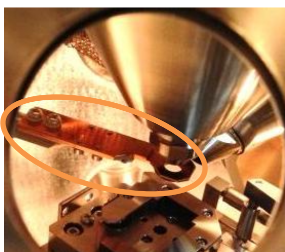
- If the observation area is moved significantly, readjust the eucentric position.
- If the eucentric position is incorrect, milling with the Ar⁺ ion gun will be ineffective, and the Auger signal intensity will decrease.

Backscattered Electron Detector (COMPO / TOPO Imaging)

Loosen the fixing screw

BSE detector insertion valve

With the tilt axis at 0° , turn OFF the secondary electron detector. While visually checking the stage, loosen the fixing screw and rotate the BSE detector insertion valve (located at the rear of the instrument) toward the IN side until it stops. Confirm visually that the detector is inserted. Do not tilt the stage after insertion, as it may cause a collision.

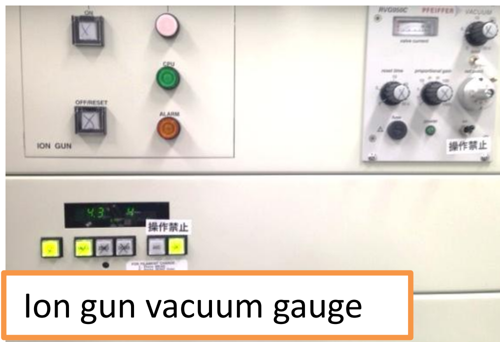


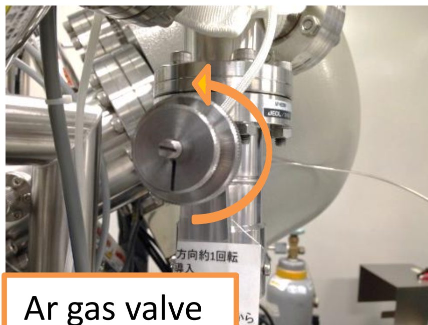
Backscattered electron detector

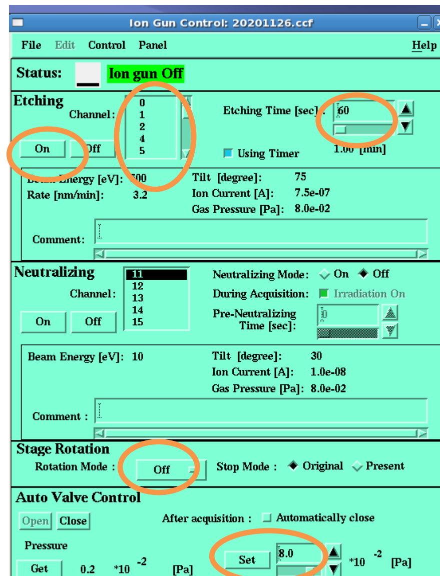
Turn the secondary electron detector ON, then click SEI at the bottom right of the SEM software and select COMPO or TOPO.

- After use, be sure to retract the detector. Do not forget that it is inserted.
- Observation depth: on the order of several hundred nanometers
- TOPO image: shows shading as if illuminated from above
- COMPO image: heavier elements appear brighter, lighter elements appear darker

Ar^+ ion etching







The Ar^+ ion beam can be used to remove surface contamination and oxide layers. It is effective not only for analysis but also for improving SEM observation.

- However, for chemical state analysis, ion beam irradiation may cause reduction or atomic mixing. Therefore, it is not recommended in such cases.
- Do not use the ion gun when an EBSD camera or a BSE detector is inserted.
- If the stage is not at the eucentric position, the irradiation area will be shifted from the observation point.

Introduction of Ar Gas

- Check the vacuum level of the analysis chamber before introducing Ar gas.
  Do not introduce gas if the vacuum is poor.
- If the vacuum degrades significantly or an error occurs, immediately close the Ar gas valve.(Be careful not to turn it in the wrong direction.)

1. Rotate the Ar gas valve slowly counterclockwise and increase the pressure to approximately **10.0 × 10^{-2} Pa** on the ion gun gauge. There is usually a marker at the 6 o'clock position, and about one full turn reaches this value. Do not over-supply gas.
2. Turn the **Auto Valve Controller** switch ON.
3. In Auger Master, go to **AES → Ion Gun Condition**. Select the channel number for etching under **Etching**. Under **Stage Rotation**, select **Off** or **One-way** (normally Off). One-way rotates the stage during irradiation.
4. Enter 7.5 in **Auto Valve Control**, and click **Set**. Wait until the gas pressure stabilizes at the set value.

Ar^+ Ion Irradiation

1. Set the etching time under **Etching**.
2. Click **On** in the Etching section to start irradiation. The etched area is approximately **1 × 1 mm** centered on the observation point.

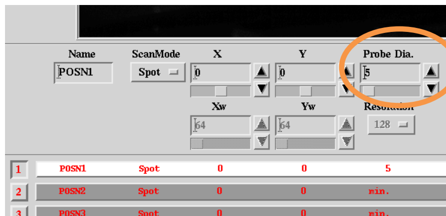
Handling of charging-prone samples

For samples with poor electrical conductivity, electron beam irradiation generally causes the surface to charge negatively. This makes it difficult to obtain SEM images and spectra. In Auger analysis, the analysis depth is extremely shallow, so coating with carbon or gold is often not suitable. Therefore, other methods must be used to reduce charging.

Mask with Al foil

Sample and Stage Preparation

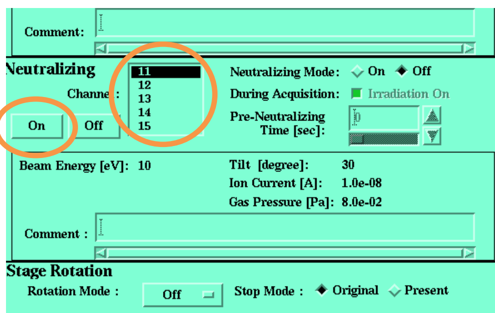
- Ensure good electrical contact using carbon tape or conductive paste.
- Mask areas other than the analysis region with aluminum foil.
- Remove insulating debris and excessive surface roughness.
- Tilt the stage to a high angle (around **80°**) to increase secondary electron emission and reduce negative charging.



Probe size setting(µm)

Beam Conditions and Spectrum Acquisition

- Use the lowest possible accelerating voltage and probe current.
- For spectral acquisition, increase the probe size or defocus the electron beam.
- Perform preliminary measurements to check whether charge shift occurs and whether the spectrum is stable.



Channels from Ch11 to Ch15 are available for various beam settings.Ch15 may cause slight sputtering.

Use of Neutralizer (Electron Flood Gun)

Low-energy Ar^+ ion irradiation can help reduce negative charging around the analysis area.
Refer to "Ar^+ ion Etching(p. 14)" to introduce Ar gas and adjust the pressure. Then, in the **Neutralizing** menu, select the channel for the neutralizer and click **On**.
Acquire spectra while the neutralizer is operating.

This method is effective for reducing charging around the analysis area, but for charging directly at the analysis point, stage tilting is more effective.

Samples prone to oxidation and contamination

If a sample is prone to oxidation or contamination, peak intensities may change even during analysis. As a countermeasure, cleaning and heating of the holder (which may carry contaminants or moisture) is effective. If possible, apply the same treatment to the sample.

Heater

Heat gun

Cleaning and Preparation Procedure

- Ultrasonically clean the holder with ethanol. (Optionally, ultrasonically clean with ultrapure water.)
- After drying with a blower or similar, heat the holder to **above 100 ° C** using a heater or heat gun. (If possible, mount the sample during heating as well.)
- Introduce the holder into the preparation chamber while still warm. (If it cools down, moisture may reattach.)
- Perform preliminary measurements to monitor increases in contamination (carbon) or oxygen signals.

Electron beam-sensitive samples

For heat-sensitive samples, electron beam irradiation may cause surface deformation or changes in peak intensity. These effects can be mitigated by adjusting sample preparation and analysis conditions. Alkali metals, alkaline earth metals, and halogens often show peak attenuation under electron beam irradiation. Check the extent of these effects by performing preliminary or repeated measurements.

Carbon coater

- Adjustment of Beam Conditions. Reduce the accelerating voltage and probe current as much as possible. As a guideline, set the accelerating voltage to approximately 10 × the target peak energy to maintain a good S/N ratio.
- Increase the probe diameter (Probe dia.) to spread the beam, or perform area analysis instead of point analysis.
- Apply a thin carbon coating, or coat areas other than the region of interest with a conductive layer to improve heat dissipation.

Auger measurement

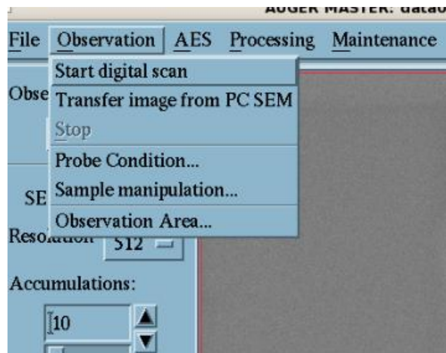
The following analysis methods are mainly used:

- **Wide scan** - Acquire spectra over a wide energy range; mainly used for elemental analysis
- **Split scan** - Measure only the energy region of specific elements; mainly used for chemical state analysis
- **Line profile** - Line analysis. Acquire peak and background intensities of specific elements to generate a line profile
- **Spectrum line profile** - Line analysis. Measure the energy region of specific elements, calculate intensity, and generate a line profile
- **Depth profile** - Depth analysis. Repeat Ar^+ etching and spectral acquisition
- **Auger image** - Acquire peak and background intensities of specific elements to generate elemental mapping
- **Spectrum image** - Create a data cube in which each pixel contains spectral data, and reconstruct mapping images

The main items that must be set or prepared for analysis are as follows:

1. **Acquisition of analysis area** - Capture the analysis area in Auger Master
2. **Setting of analysis conditions** - Specify analysis mode, energy range, step size, acquisition time, number of accumulations, and auto probe tracking interval
3. **Selection of analysis position** - Specify the analysis location as a point or area; the beam spot size during analysis can also be set
4. **ROI settings** - Specify the elements to be measured and define peak and background regions
5. **Ion gun settings** - Set ion beam output and irradiation time, and configure the neutralizer used during charging
6. **Auto probe tracking settings** - During measurement, images of the analysis area are acquired and compared with the initial image; if a shift is detected, the beam position is corrected to the original position. Used for samples prone to movement, high magnification, or long measurements
7. **Pre-acquisition** - Acquire spectra before the main measurement to check whether the spectra are stable. Data is not saved
8. **Main acquisition** - Acquire spectra with accumulation. The number of accumulations and other parameters can be changed during measurement

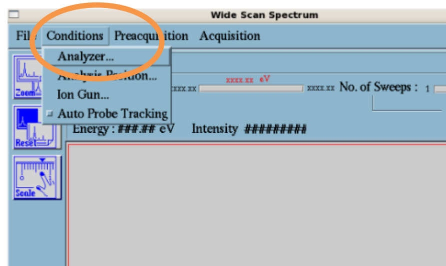
Wide scan



1. Acquisition of analysis area

SEM images are acquired into Auger Master via **Observation → Start Digital Scan**.

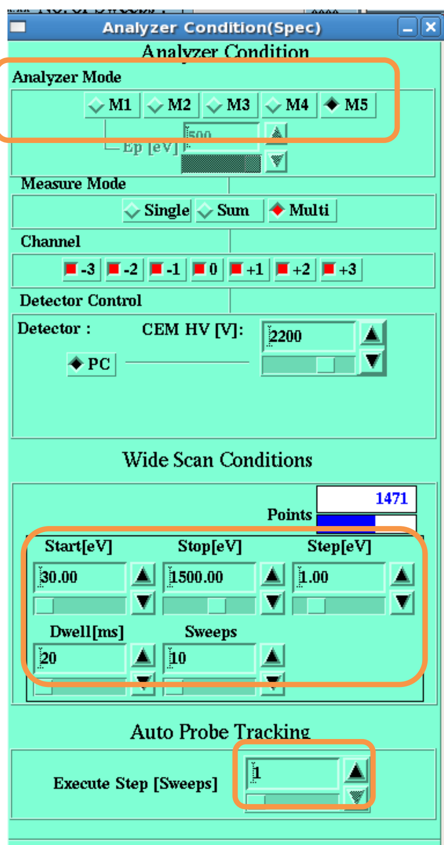
The resolution, number of accumulations, and acquisition area can be adjusted in the left menu of Auger Master.



2. Setting of analysis conditions

Go to **AES → Spectrum → Wide Scan Spectrum** to open the wide scan window.

In **Wide Scan Spectrum → Conditions**, select **Analyzer**.



Analyzer mode selection

M1: Energy resolution is constant regardless of the measured energy. The pass energy is specified. A smaller value provides higher resolution. Generally not used.
M2–M5: Energy resolution decreases proportionally with increasing measurement energy.
M2: 0.05%, M3: 0.1%, M4: 0.35%, M5: 0.5%.
Resolution worsens in this order, but intensity increases.
M2 and M3 are mainly used for chemical state analysis, while M4 and M5 are used for compositional analysis.

Wide Scan Conditions settings

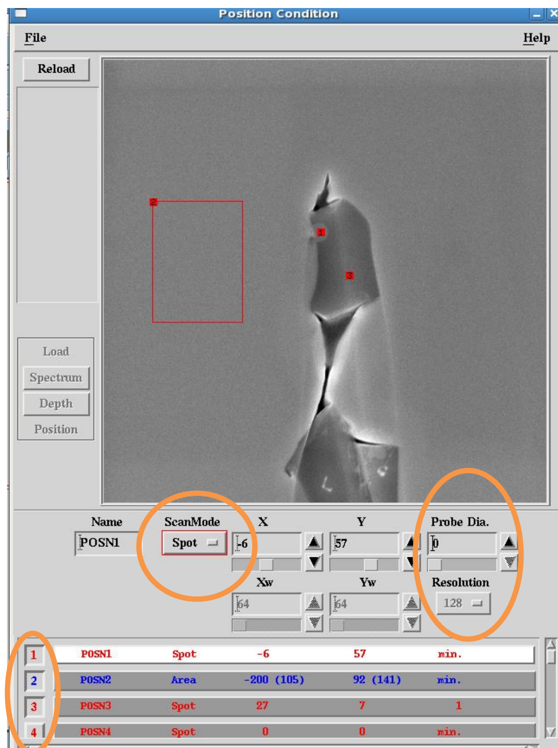
Specify the energy range to be measured. Set the step size, acquisition time, and number of accumulations. The number of accumulations can be changed during acquisition. Recommended step size:
Compositional analysis: **1.0 eV**
Chemical state analysis: **0.2 eV**

Auto Probe Tracking settings (if used)

Set how frequently probe tracking is performed.

After completing the settings, the measurement time is displayed at the lower left of the Wide Scan Spectrum window.

Wide scan



3. Selection of analysis position

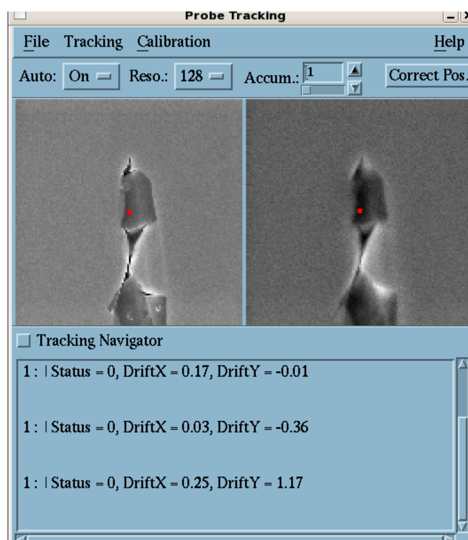
In **Wide Scan Spectrum → Condition → Analysis Position**, select the analysis location from the acquired image.

Selection of analysis points

1. Press the checkboxes **No. 1–20** at the bottom of the screen.
2. Select **Scan mode**.

   By selecting **Scan** and using SEM spot mode, the analysis position can be specified by moving the spot on the SEM image.
3. Specify the analysis position.
4. Specification of Probe Dia. and Resolution

   If Probe Dia. is set to 0, the smallest beam diameter is used (approximately 10 nm). If another value is entered, the beam expands to the specified diameter (µm), allowing analysis over a wider area and resulting in a more averaged and stable spectrum. Be sure to check the setting, as values from a previous user may remain. Note that the value may not be applied unless you press Enter after input.



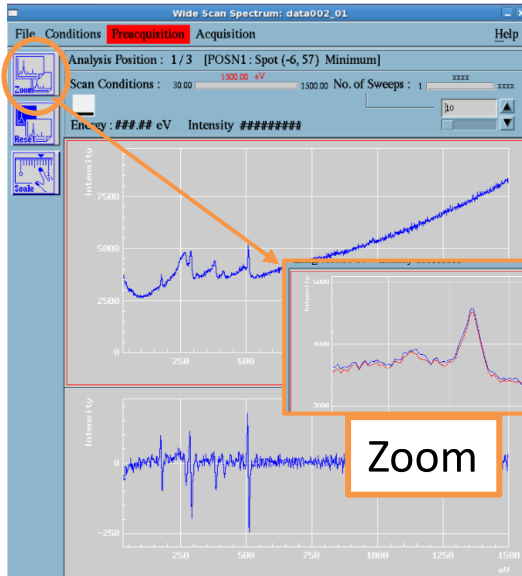
Left: original image
Right: acquired image

6. Auto Probe Tracking settings (if used)

Image drift during measurement is corrected using **Image Fine Shift**. Use this function for high-magnification analysis (around **10,000 ×**) where precise positioning is required. Before use, return **Image Fine Shift** to the origin and acquire the SEM image in Auger Master. If the initial position is already near the limit of the Image Fine Shift range, this function may not work properly.

1. Select **AES → Probe Tracking Control**.
2. Select **File → Reload** to load the SEM image.
3. Switch **Auto** to ON.
4. Set appropriate values for resolution and number of accumulations.
5. Press **Correct Pos.** to confirm proper operation.

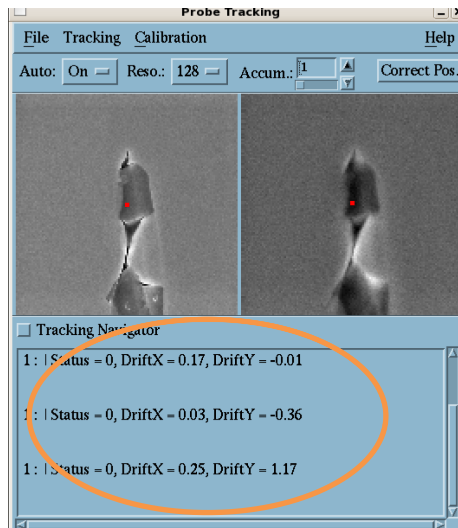
Wide scan



7. Pre-acquisition

In Wide Scan Spectrum → Preacquisition → Start, preliminary acquisition begins.

The measured spectrum is displayed in the upper panel, and its differential form is shown in the lower panel. The blue trace represents the previous acquisition, and the red trace represents the real-time acquisition. If the spectral shape is stable, proceed to the main acquisition. This data is not saved. The acquisition continues indefinitely unless Stop is pressed.



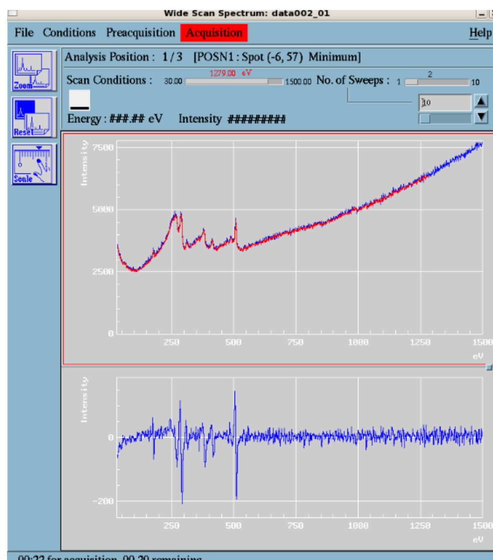
If the spectrum does not stabilize

- The sample is moving
- Charging is occurring
- The sample is being damaged or reduced by the beam
- The analysis point is located at a phase boundary
- Contamination is present
- Check and eliminate these possible causes.

When using Auto Probe Tracking

Probe tracking also operates during pre-acquisition, so check how much positional drift occurs.

If the movement during measurement is large relative to the target analysis area, shorten the tracking interval. (The shift values shown on the left are in µm.)



8. Main acquisition

In **Wide Scan Spectrum → Acquisition → Start**, the main acquisition begins.

To stop during acquisition, select **Stop**.

If **Pause** is used, parameters such as current and stage position can be adjusted before resuming.

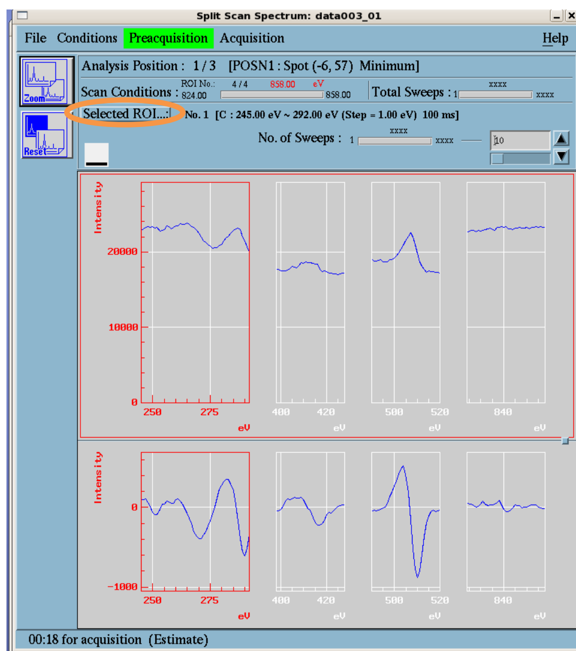
Leaning on the instrument or making loud noises may cause the analysis position to shift. Please keep the environment stable and quiet.

Split scan

Refer to Wide Scan for items not listed below.

2. Setting of analysis conditions

Go to **AES → Spectrum → Split Scan Spectrum** to open the Split Scan window. For **Analyzer Condition** settings, refer to Wide Scan.

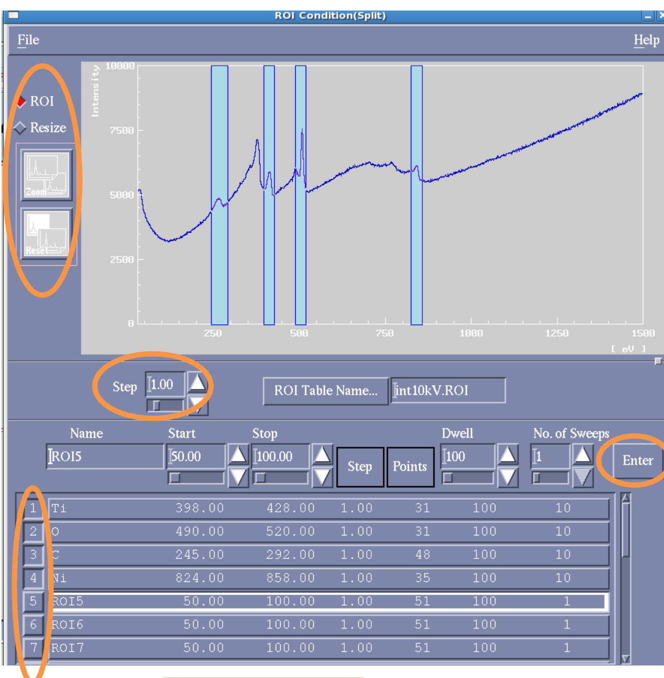


Click **Selected ROI** to change the number of measurements for each element.

4. ROI settings

Go to **Split Scan Spectrum → Conditions → ROI Condition (split)** to open the ROI window. The previously acquired Wide spectrum is displayed. Select or enter the element to be measured from the ROI table below, and click the corresponding number button. The measurement range is displayed in light blue on the spectrum.

Click the **Enter** button to update and register the ROI table contents.

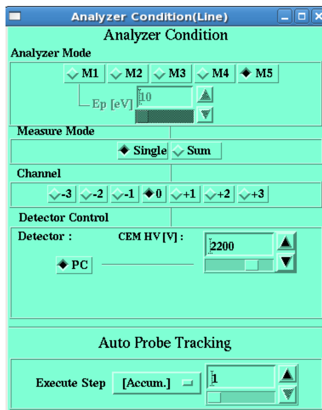


ROI table

While checking the displayed spectrum, adjust the measurement range for each element. You can also set acquisition time, number of sweeps, and step size.

To enlarge the displayed spectrum, click **Resize**, then use the mouse to zoom in. To return to ROI settings, click **ROI**. To refer to another spectrum, select and open it from **File**.

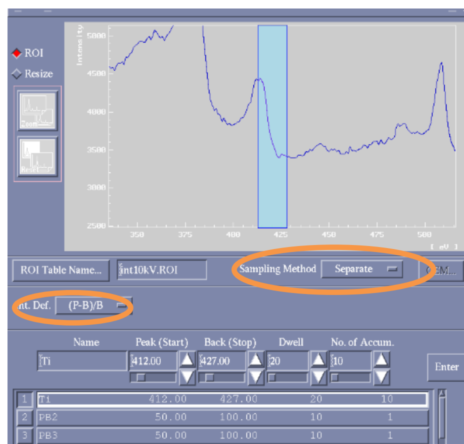
Line profile



Refer to Wide Scan for items not listed below.

2. Setting of analysis conditions

Go to **AES → Line Profile → Line Profile** to open the Line Profile window. Set the **Analyzer mode** and **Auto Probe Tracking**.

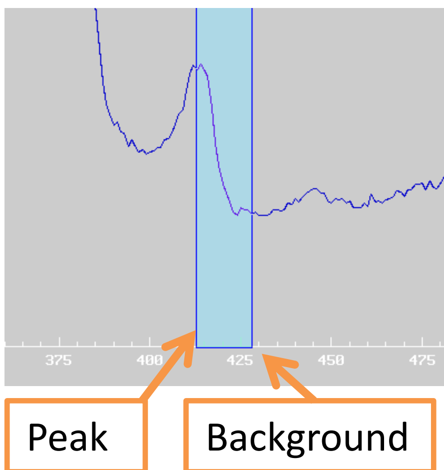


4. ROI settings

Refer to Split Scan for basic operations.

Here, the peak and background used to generate the Line Profile intensity are set using two methods. The definition of intensity is selected from **Int.Def.**. Basically, **(P – B) / B** is sufficient.

Dividing by B helps reduce the effect of surface roughness at the analysis point.



• Separate (PB separate) method

The intensities at the specified peak and background energy values are measured individually.

Select **Separate** in **Sampling Method**, then set the elements to be measured. For each element, specify the energy values for the peak and background. In the spectrum display (light blue bars), the left bar indicates the peak, and the right bar indicates the background.

Compared to the simultaneous PB method, this method is easier to set. As long as the peak is visible, measurement can be performed reliably. However, it requires more measurement time than the simultaneous PB method. It cannot be used when the peak intensity (P) is small and lower than a rising background (B). Also, the P and B settings cannot be changed after measurement.

Line profile

CEM

• Simultaneous (PB simultaneous) method

Instead of measuring P and B separately, the seven detectors are assigned to measure P and B, and data are acquired simultaneously.

Select **Simultaneous** in **Sampling Method**, specify the element, and click the **CEM** button to open the **P/B Simultaneous Condition** window.

CEM settings

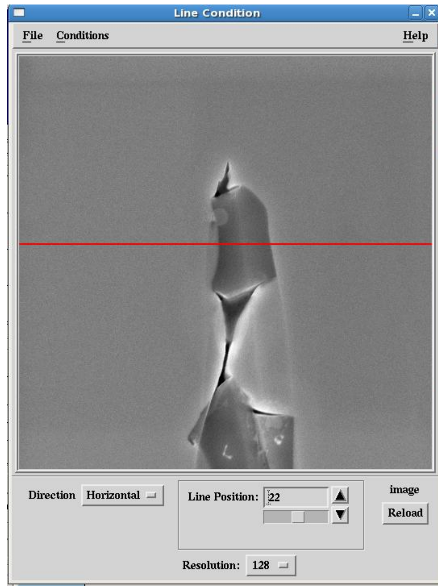
Adjust the display range so that the peak of the element to be measured is clearly visible. Change the **Analyzer mode** to **M1**, and adjust the **Ep** value so that both the peak and background fall within the range of **−3ch to +3ch**.

The seven channels from −3ch to +3ch correspond to individual detectors. The numerical values below indicate the energy measured by each detector. Changing **Ep (pass energy)** alters the overall energy range covered by the seven detectors, as well as the energy values assigned to each detector.

Adjust the energy values of each channel and assign them to either Peak or Background. Set the energy value of the background channel (e.g., **3ch**) as the **Gain value calculation energy**. Repeat the CEM setup for each element.

In the example above, −3ch and +3ch are assigned as Background, and the others are assigned as Peak. In general, place the peak center at **0ch** (this can be set by moving the bar on the spectrum), then adjust **Ep** so that −3ch and +3ch correspond to the background. If the peak has a tail extending toward the low-energy side, it is better to assign only the high-energy side as Background and use the remaining channels as Peak. In addition to Peak and Background, channels can also be set to **None** (not measured).

Line profile

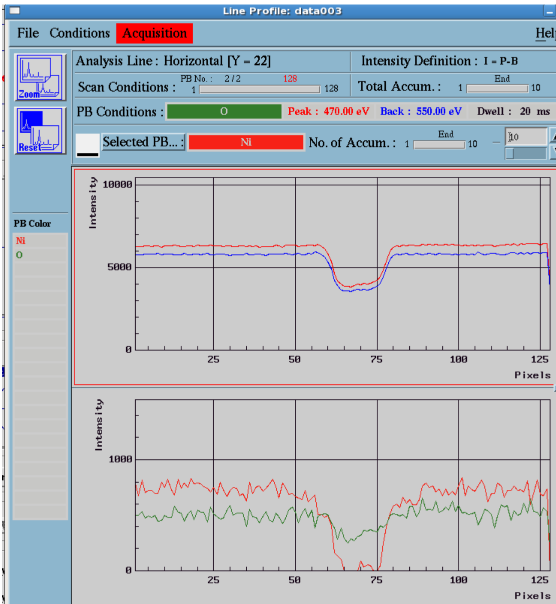


3. Selection of analysis position

In **Line Profile → Condition → Analysis Position**, specify the analysis line from the acquired image. Select the orientation (vertical or horizontal) using **Direction**, and set the resolution using **Resolution**.

For example, if the SEM image has a width of about **5 µm** and the resolution is set to **256**, one step corresponds to approximately **20 nm**. Since the Auger analysis spot size is approximately **10–20 nm**, reducing the step size further does not significantly improve accuracy.

If analyzing from one edge of the image to the other, set the acquisition magnification to **300× or higher**. At lower magnifications, the signal sensitivity differs significantly between the center and edges of the image, which may affect the results.

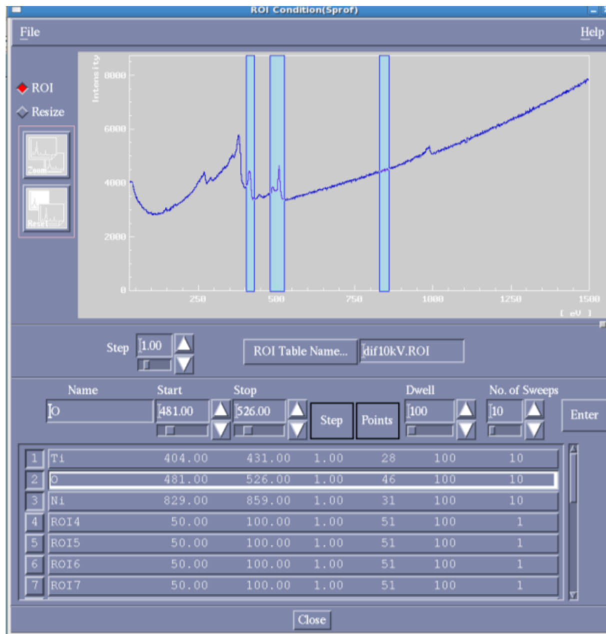


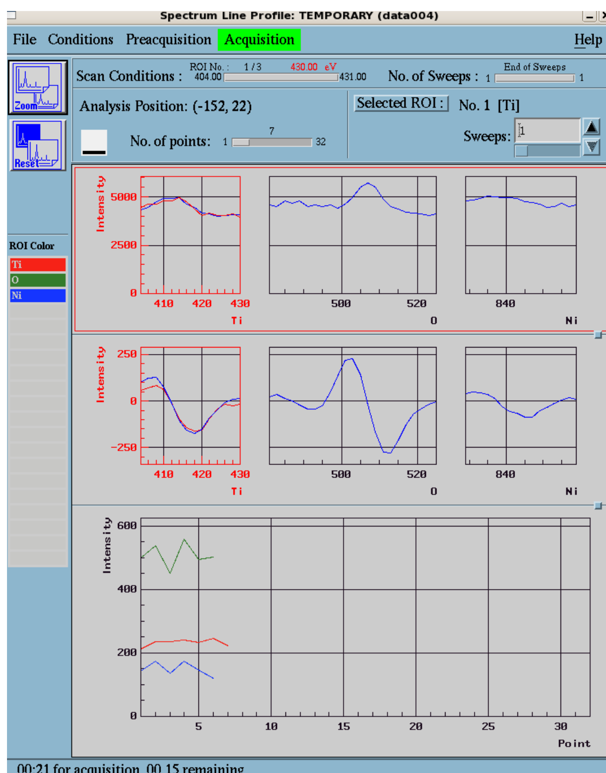
Top: P/B intensity
Bottom: Line profile
The horizontal axis is **pixels** in both cases.

Other settings are the same as for Wide Scan. There is no preliminary acquisition. Click **Select PB** to change the number of accumulations for each element.

For example, in (P − B) / B using the simultaneous PB method, when B is measured at two or more points, a first-order linear function is created from B. After subtracting the intensity of each P, the result is divided by the average value of B. This value is then accumulated over the scan. The appearance of mapping images may differ when data are loaded in Processing versus Investigator. This is because Processing calculates intensity differently, using the total accumulated values of P and B along with the ratio of channel numbers. In Investigator, intensity is calculated by generating a first-order linear function for B at each measurement point, which more accurately compensates for surface roughness effects. In addition, pixels judged to be at the noise level are assigned an intensity of zero in Investigator.

Spectrum line profile





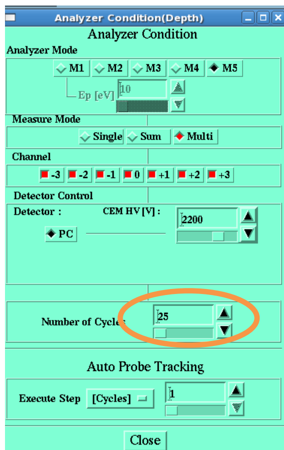
Spectrum Line Profile differs from Line Profile in that it does not acquire only P and B. Instead, spectra are collected for each element, the peak intensity is read from the differential form, and the distribution at each pixel is displayed. Because spectra are acquired, this method requires more time. Use this method when it is difficult to define P and B (e.g., unstable spectra or low peak intensity), or when performing line analysis after spectral deconvolution.

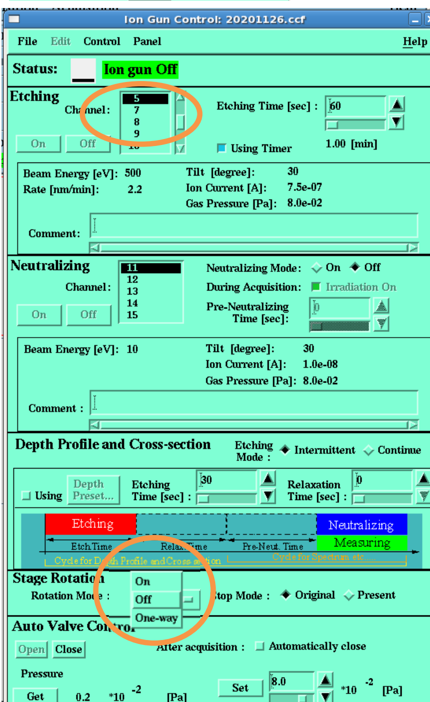
1–3: Refer to Wide Scan and Line Profile
4: Refer to Split Scan for ROI settings
6–8: Refer to Wide Scan

Top: acquired spectra
Middle: differential form of the spectra
Bottom: line profile graph of intensity vs. pixel based on the differential data, where line analysis progresses gradually from the left side.

The intensity is defined as the difference between the maximum and minimum values in the differential spectrum.

Depth profile





1. Acquisition of analysis area

Refer to Wide Scan. Be sure to set the sample to the eucentric position.

2. Setting of analysis conditions

Go to **Auger Master → AES → Depth Profile → Depth Profile** to open the window. In **Analyzer Condition**, set the **Analyzer mode**, **Number of Cycles**, and **Auto Probe Tracking** (if used).

- **Number of Cycles** specifies how many times the sequence of Ar^+ etching and analysis is repeated. Note that the first cycle is always analysis without Ar^+ etching.
- For **Auto Probe Tracking**, the step type can be selected from **Cycles**, **ROI**, or **Sweeps**. Set an interval that avoids excessive drift.
- Regarding the depth interval for analysis, in Auger analysis, about **90%** of the detected signal originates from the top ~**2 nm** of the surface. Therefore, if depth profiling can be performed at intervals of ~**2 nm**, data loss is minimal.
- Set **Cycles** and **etching time** appropriately, taking into account the etching rate of each channel and the material. When analyzing chemical state changes in depth using Ar^+ etching, it is recommended to verify whether the etching process itself alters the chemical state.

3. Selection of analysis position

Refer to Wide Scan

4. ROI settings

Refer to Split Scan

5. Ion gun settings

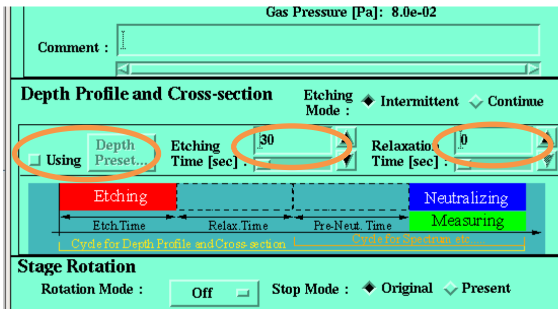
Refer to Ar^+ ion etching for Ar gas introduction and pressure adjustment.

Go to **Depth Profile → Conditions → Ion Gun Control** to open the etching settings window. Select the channel number for etching under the **Etching** menu at the top.

Select **Off** or **One-way** for **Stage Rotation**.

Since continuous irradiation from a single direction can roughen the surface, rotating the stage during irradiation helps reduce roughness. However, rotation may cause positional shift after rotation, so **Off** is recommended when precise analysis positions are required.

Depth profile



In **Depth Profile and Cross-section** (middle section), enter **Etching Time** and **Relaxation Time**.

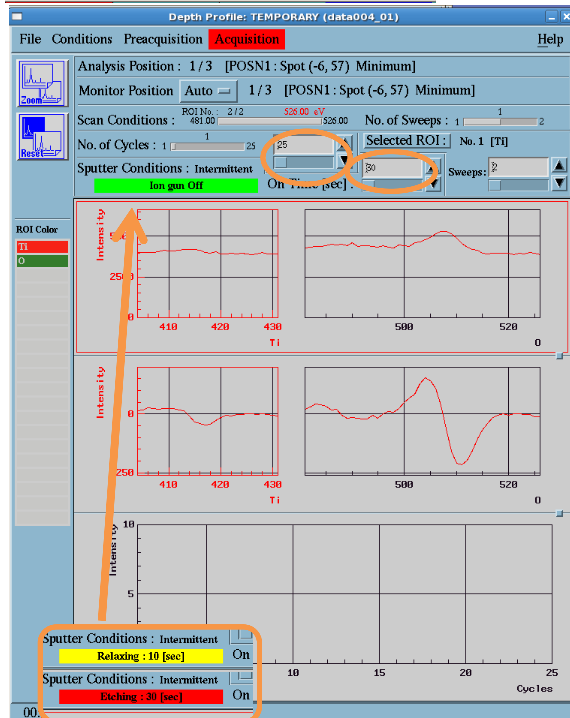
Relaxation Time is the waiting time after Ar^+ etching to allow relaxation of the surface electric field. Set it to approximately **10 seconds**.

Etching Time Table

Etching Time[sec]	Repeat	Cycle
10	10	2 - 11
60	10	12 - 21
300	1	22 - 22

New | Add | Delete | Ok | Cancel

Turn **Using** ON, then click **Depth Preset** to open the **Etching Time Table**. You can set individual etching times for each cycle.



6. Auto probe tracking settings

Refer to Wide Scan. When the stage is rotating, tracking may not work properly.

7. Pre-acquisition

Refer to Wide Scan. No etching is performed during this step.

8. Main acquisition

Start acquisition from **Acquisition → Start**. The upper panel shows spectra for each ROI, the middle panel shows the differential spectra, and the lower panel displays **Intensity vs. Cycle** graphs with color-coded ROIs.

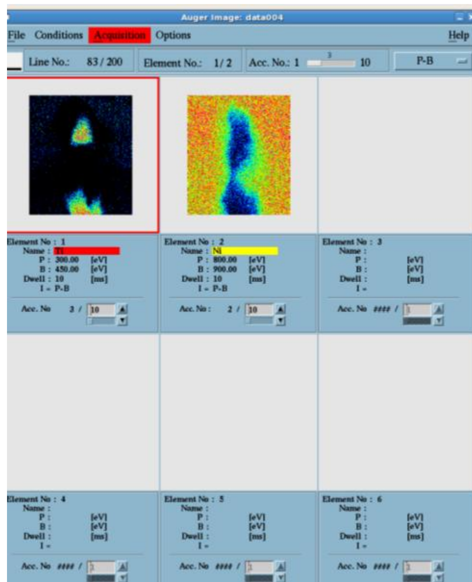
Sputter Conditions displays the status of the Ar^+ ion gun.

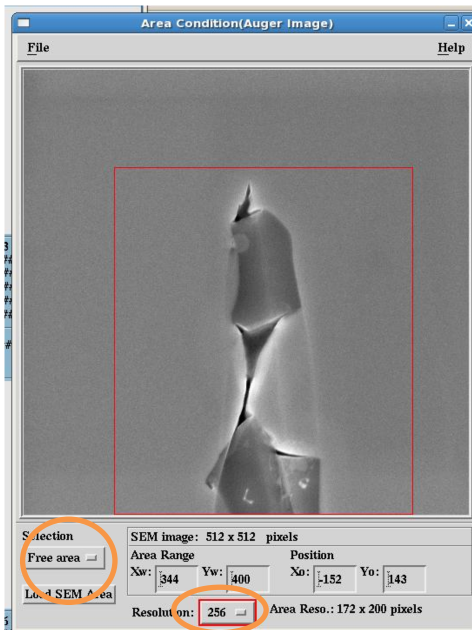
During acquisition, you can change **Cycle**, **Sweep for each ROI**, and **etching time** from the top menu.

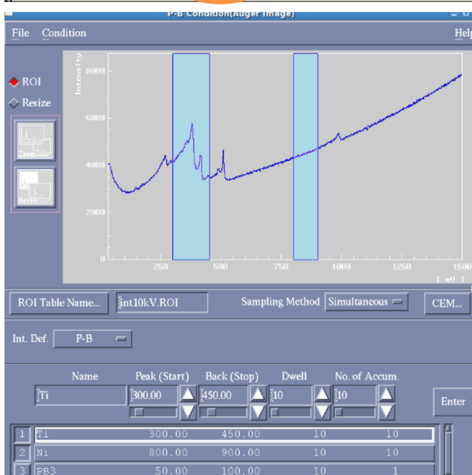
Select ROI

No.	ROI name	Start[eV]	Stop[eV]	Step[eV]	Dwell[ms]	Sweeps
1	Ti	404.00	431.00	1.00	20	2
2	O	481.00	526.00	1.00	20	2

Auger image







1. Acquisition of analysis area

Since this is a long measurement, acquire the SEM image after returning **Image Fine Shift** to the initial position so that **Auto Probe Tracking** can function properly.

2. Setting of analysis conditions

Go to **AES → Auger Image → Auger Image** to open the window. Set the **Analyzer mode** and **Auto Probe Tracking** (refer to Wide Scan).

3. Selection of analysis position

In **Auger Image → Condition → Analysis Area**, specify the analysis area from the acquired image. Select either full area or region selection in **Selection**. For region selection, drag the mouse to define the area. Set the resolution using **Resolution**.

Doubling the resolution increases the measurement time by a factor of four.

4. ROI settings

Refer to Split Scan and Line Profile.

Images are generated by acquiring P and B and applying the defined intensity calculation. The method for acquiring P and B can be selected from **Separate (PB separate)** or **Simultaneous (PB simultaneous)**.

The **Simultaneous method** is recommended over the Separate method. The Separate method requires approximately twice the measurement time, resulting in longer acquisition. In mapping with the Simultaneous method, P and B settings for each channel can be modified afterward in **Image Investigator**, allowing partial correction of errors in P/B assignment caused by shifts in peak and background positions due to differences in analyzer mode.

6. Auto probe tracking settings

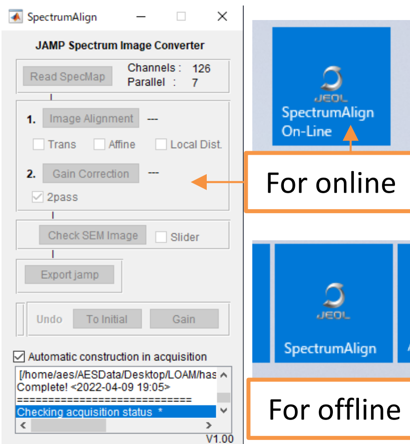
Refer to Wide Scan

8. Main acquisition

After the first scan for each element is completed, data can be accessed. Check in **Image Investigator** whether the P/B settings are appropriate (for the Simultaneous method).

Spectrum image

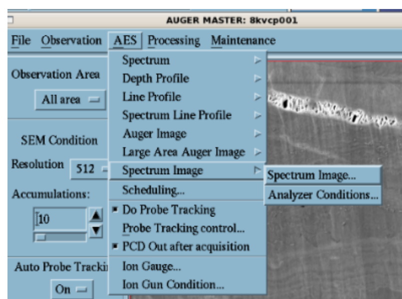
Spectrum Image is a newly added mapping method in Auger Master. Unlike the conventional Auger Image described on the previous page, spectra are sequentially acquired at all pixels within the field of view. From the cube data containing spectral information, mapping images can be generated and spectra can be extracted from selected regions. Drift correction and noise reduction functions are highly effective. Since scanning is performed continuously, beam damage can be reduced compared to point analysis. Although the measurement takes time, it allows comprehensive acquisition of information over the entire field of view in a single measurement. The acquired cube data is reconstructed using a Windows-based software called **EFSEMviewer**.



Before starting, make sure that **SpectrumAlign (Online)** is running on Windows. If not, launch it from the Windows Start menu. This software automatically generates the cube data. Be careful not to select **SpectrumAlign (Offline)**.

1. Acquisition of analysis area

Since this is a long measurement, acquire the SEM image after returning **Image Fine Shift** to the initial position so that **Auto Probe Tracking** can function properly.

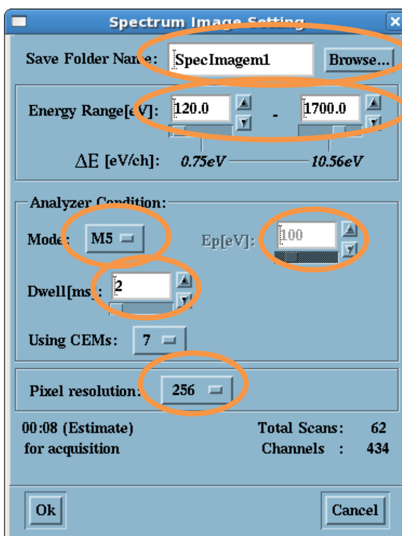


2. Setting of analysis conditions

Go to **AES → Spectrum Image → Spectrum Image** to open the window. From **Conditions → Analyzer**, set only **Auto Probe Tracking** (refer to Wide Scan). Basically, set up the measurement in units of **Frames**.

3. Selection of analysis position

There is no separate setting for the analysis position. The acquired image itself becomes the analysis area.



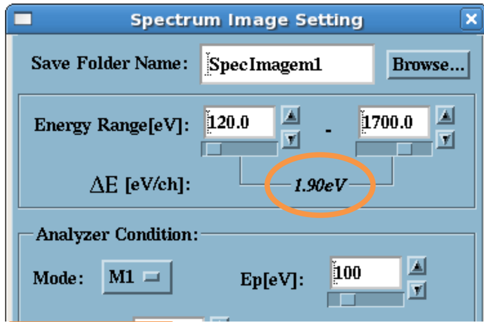
4. ROI (Spectrum condition) settings

Specify the save location and file name using **Browse**. In the specified directory, Auger Image files for each energy range (extension: **.A**) will be saved. A combined cube data file (extension: **.jamp**) will then be generated at the same directory level with the specified name.

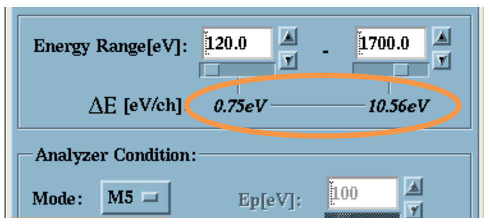
Set the spectral acquisition range using **Energy Range**. From **Analyzer Condition**, set **Mode**, **Ep** (when using M1), and **Dwell**. Set the **Pixel resolution**.

After setting up the measurement, the estimated measurement time will be updated.

Spectrum image



For M1



For M5

4. Spectrum condition settings (continued)

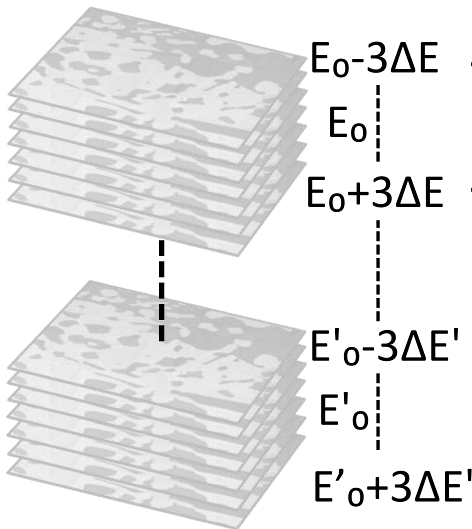
In this measurement, the specified energy range is scanned sequentially using the defined mode, energy steps, and seven detectors.

For M1

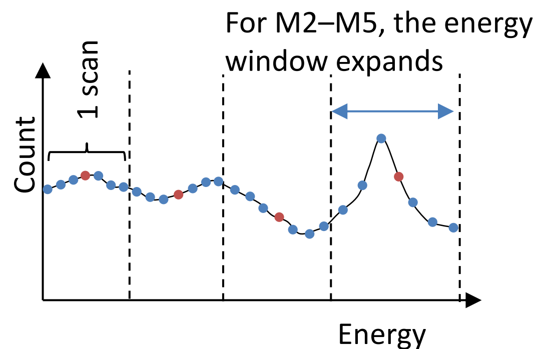
The energy step is determined by the **Ep** setting and is independent of the measurement energy, resulting in equally spaced energy intervals. This is suitable when fine measurement is required in the high-energy region.

For M2–M5

The energy step depends on the measurement energy, resulting in non-uniform energy intervals. M5 provides the widest energy range and highest sensitivity. M2 provides the narrowest energy range and lower sensitivity. In general, use **M5**. If higher energy resolution is required (e.g., when peaks are close together in the high-energy region), narrow the energy range and use a lower mode number.



1 scan



Concept of data acquisition

5. Ion gun settings

If measurements are to be performed while using the neutralizer (electron flood gun), refer to pp. 14–15.

6. Auto probe tracking settings

Refer to Wide Scan. Use this function at high magnification.

8. Main acquisition

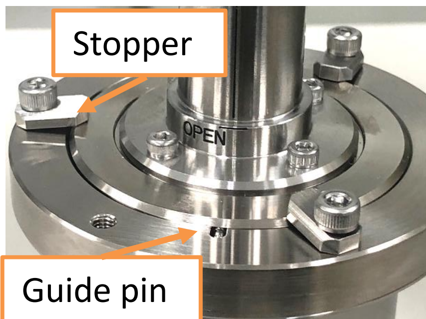
Start acquisition from **Acquisition → Start**. There is no pre-acquisition. If the measurement is interrupted, cube data will be generated from the data acquired up to that point.

Transfer vessel (obtain staff approval before use)

The transfer vessel allows sample introduction without exposure to air. Be sure to contact the staff before use.

Transfer vessel
Transport case attachment

Holder for
Transfer vessel



Operation inside the glove box

1. Fix the sample to the holder.
2. Release the stopper of the transport case and remove the transfer vessel.
3. Turn the transfer vessel knob to **OPEN**.
4. Place the sample holder into the transfer vessel.
5. Turn the knob to **CLOSE**.
6. Align the guide pin, attach the transfer vessel to the transport case, and tighten the stopper.

Do not contaminate the parts that will be placed under vacuum.

Closed state

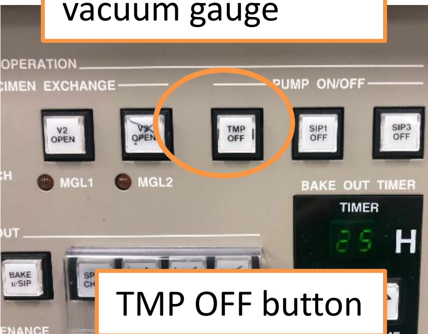
Operation at AES

1. VENT the load-lock chamber.
2. Loosen the fixing knob of the blank flange, slide the stopper outward, and remove the blank flange.
3. Wrap the removed flange with aluminum foil.

Transfer vessel(obtain staff approval before use)

Mounting the transfer vessel

Load-lock chamber vacuum gauge



TMP OFF button

Operation at AES

4. Remove the transfer vessel from the transport case, align the guide pin, and mount it on the top of the load-lock chamber. Slide the stopper inward and secure it again.
5. Close the load-lock chamber door and press the **VENT** button to start evacuation.
6. Remove the operator panel of the instrument and check the vacuum gauge of the load-lock chamber. Wait until it reaches **1×10^{-4} Pa**.
7. Press the **TMP OFF** button. Confirm that the magnet ring is fully retracted.
8. Carefully turn the vessel knob clockwise to slightly open the vessel, just enough to allow evacuation.(**Always stop the TMP before opening.**)
9. Press the **TMP OFF** button again to restart evacuation. Pump down until the vacuum gauge reaches **0**.
10. Open the vessel fully to **OPEN**, and while observing through the window, operate the magnet ring to retrieve the sample holder. Retract the magnet ring and close the vessel to **CLOSE**.
11. Follow the standard sample introduction procedure to transfer the sample holder to the analysis chamber.
12. Open the vessel again to **OPEN**.

Transfer vessel (obtain staff approval before use)

When retrieving the sample into the vessel after analysis (with the vessel still mounted)

1. Close the vessel from **OPEN** to **CLOSE**.
2. Following the standard shutdown procedure, retrieve the sample holder to the load-lock chamber.
3. Open the vessel to **OPEN** using the knob, operate the **MGL**, and place the sample holder into the vessel.
4. Close the vessel to **CLOSE**.
5. VENT the load-lock chamber.
6. Loosen the fixing knob at the top of the load-lock chamber, slide the stopper outward, and remove the vessel.
7. Attach the blank flange, set the stopper, and secure it.
8. Close and lock the load-lock chamber door, then press the **VENT** button to evacuate.
   For the remaining steps, refer to the End procedure.

End procedure

First of all

Return all hardware and software parameters to their initial settings (e.g., acceleration voltage **10 kV**, objective aperture **No. 4**, SEM mode **No. 0**, Analyzer mode **M5**, Auto Probe Tracking **OFF**, Dynamic Focus **OFF**, etc.)

If using the Ar^+ ion gun

Stop the Ar gas supply before removing the sample.

- Turn the **Auto Valve Controller** switch **OFF**
- Rotate the Ar gas valve **clockwise by 1/4 turn** (to the 9 o'clock position)
- Wait **3 minutes** for cooling
- Rotate the Ar gas valve **clockwise by 3/4 turn** (to the 6 o'clock position)

If using the BSE detector or performing EBSD measurement

Before moving the stage to the sample exchange position, retract the backscattered electron detector and the EBSD camera. Especially for the EBSD camera, always retract it first. If the stage is moved first, it may collide with the camera.

Move the stage to the sample exchange position

As during loading, select **Sample Manipulation → Special Position**, click **Move**, and select the holder.

Before removing the sample

Turn the secondary electron detector **OFF** and set the **PCD to IN**. Remove the viewport cover to allow visual inspection of the chamber.

Removing the sample

Press the **V2** button to open the V2 valve. Push the magnet ring forward to catch the holder. Rotate the magnet ring from **OPEN to CLOSE**, then pull it fully backward. Press the **V2** button again. Always visually check the inside of the analysis chamber. Unlock the load-lock chamber and press the **VENT** button to return to atmospheric pressure. Reattach the viewport cover.

After removing the sample

Press the **VENT** button again to evacuate the load-lock chamber. After cleaning, store the holder in the desiccator. Retrieve the data via the network from the AES analysis PC. Leave the SEM software and Auger Master running on the instrument PC, and turn off only the display power. Check the vacuum level of the analysis chamber and complete the remaining entries in the logbook.

Q&A

- **The V2 button does not respond**
  → Move the magnet ring fully to the left end.
- **The VENT button was pressed, but evacuation did not start**
  → The load-lock chamber door may not have been fully closed when pressed, causing interruption. Contact the staff.
- **The sample holder does not fit onto the stage**
  → The fork may not be properly inserted into the upper groove of the holder in the load-lock chamber / The stage position has not been returned to the initial position.
- **No SEM image appears even when the secondary electron detector is ON**
  → Set the **PCD to OUT** / Try pressing the **ACB** button / Increase the probe current.
- **"P-H" is displayed on the vacuum gauge after introducing Ar gas**
  → Immediately close the Ar gas valve (clockwise) and contact the staff.
- **COMPO image cannot be observed properly, or becomes completely white even after pressing ACB**
  → Adjust focus using the SEM image. Adjust contrast and brightness manually. If it saturates too easily, try reducing the probe current.
- **Low intensity and noisy spectra**
  → Increase the probe current / Check if Auto Probe Tracking is unintentionally ON and causing positional shift / Perform eucentric alignment / The analysis point may be in a shadowed position relative to the analyzer (adjust R or X/Y) / Check the Analyzer mode setting.
- **Distorted spectra**
  → Apply charging countermeasures / If beam damage (e.g., surface melting) is observed, adjust electron beam conditions, especially the spot size.
- **Target Auger peaks do not appear in the spectrum**
  → Modify wide scan conditions / If the carbon peak is large, perform sample cleaning or Ar^+ etching.
- **Some buttons in Auger Master are missing or unusable**
  → Exit Auger Master via **File → Exit…**, then restart it from the Auger Master icon
  → If the issue persists, exit from the **VMware Workstation** file menu and select **Power Off**, then restart from the **VMware Workstation Pro** icon in Windows
  → Contact the staff