2025/7/18 update

Rules video

X-ray Photoelectron Spectroscope (XPS) Basic manual Acquisition section

Laboratory of XPS analysis

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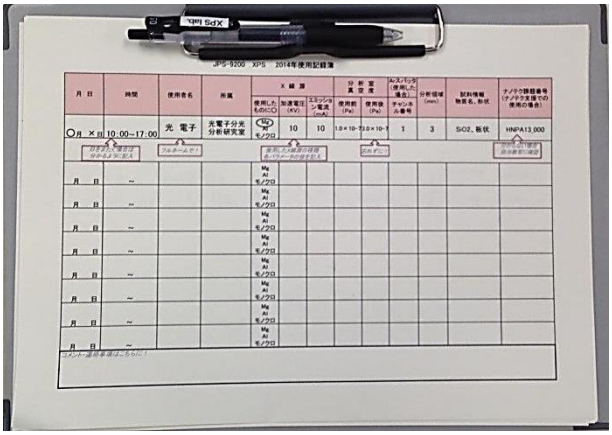
Caution

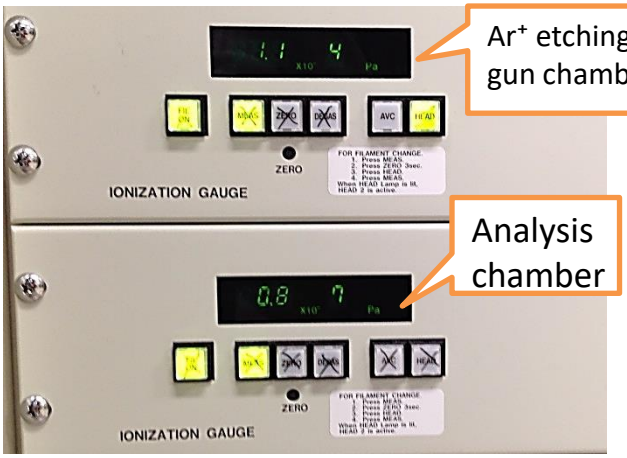
Please read carefully and follow these rules.

1.No outdoor shoes, food, or drinks in the lab.
2.Report equipment malfunctions to facility staff immediately. Emergency contact info is by the lab door.
3.Handle equipment with care.
4.Do not remove lab items without permission.
5.Manage your valuables. Lock the lab after use on holidays or at night.
6.When moving the stage, check the chamber carefully. Excessive movement may cause collisions with the X-ray source or other parts, leading to damage.
7.Restore software and hardware settings to default after use.
8.Do not connect personal USBs to the analysis PC. Use the lab's USB via the dedicated PC.
9.Do not touch items in the lab with bare hands. Clean any tools you soil.
10.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.
11.Your lab is responsible for equipment issues during use. Inform your supervisor about usage.
12.Contact staff and attend training before using equipment for the first time.
13.For reactive, large, fragile, or gas-emitting samples, consult staff before use.
14.Contact staff for permission before using a transfer vessel.

Before using



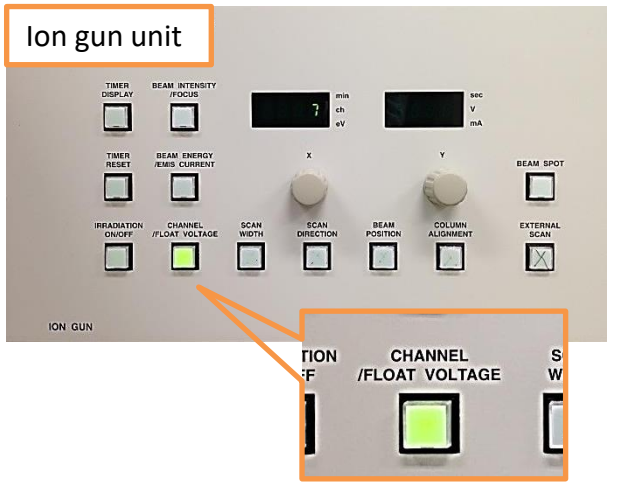
Write your name and start time in the “logbook” and write finish time and parameters after using. Check the analytical chamber vacuum before use and write the value. Use as scheduled. Change of reservation on the day is invalid.



Check the Ar^+ etching gun chamber vacuum and the analysis chamber vacuum. Normally,

- ✓ Ar^+ etching gun $\sim 10^{-4} Pa$
- ✓ Analysis chamber $\sim 10^{-7} Pa$

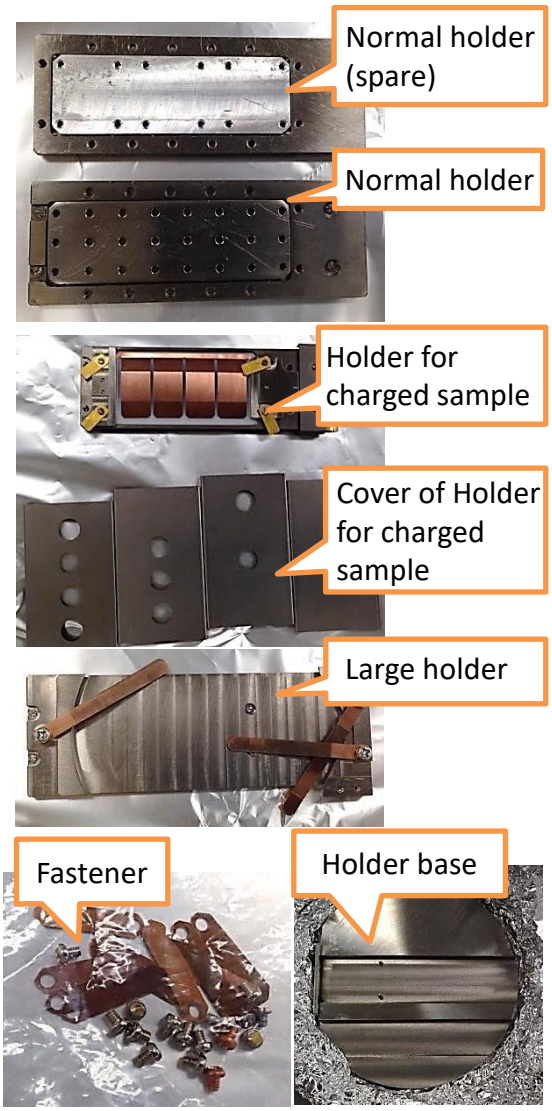
If the degree of vacuum is significantly deteriorated, inform facility staff.



Press the “Channel / float voltage” button on the ion gun unit to light it up and confirm that the display number is "7“.

The current and voltage conditions of the ion gun are input to each channel, and the ion gun is set by selecting the channel number. Channel "7" is a setting that does not use etching gun.

Sample preparation

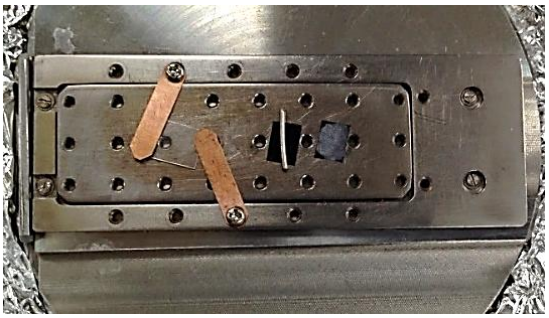


Before starting up the equipment, set your sample onto the holder. Holders are stored in the vacuum desiccator. For standard samples, use the normal holder. A dedicated holder is available for larger samples (up to 100 mm in diameter). If you are concerned about samples prone to emitting gas, fragile, or unstable, please consult with the staff. Use Fasteners or carbon tape to secure the sample onto the holder. Ensure that the sample is completely fixed.

For Powder Samples

Attach the powder to the holder using carbon tape or press it onto a metal film (such as Al, In, or Si wafer). Use an air blower to blow off loose particles at least 30 times to remove any powder that is not properly fixed. Additionally, apply light impacts or vibrations to the holder to shake off excess powder. Even a small amount of scattered powder inside the equipment can cause malfunctions.

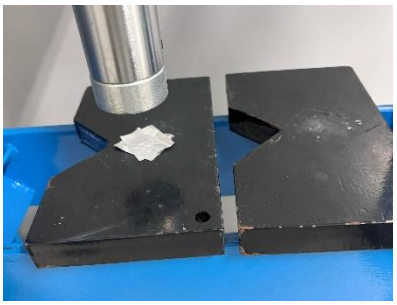
Remove the bottom plate of the holder when the sample is thick.

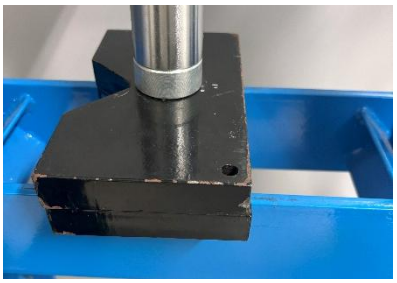


Make sure to clamp both sides of a sample. Set a sample does not move even if the holder is upside down. Pay attention to the positional relation between the X-ray source and fastener & sample.

Sample preparation (Appendix)

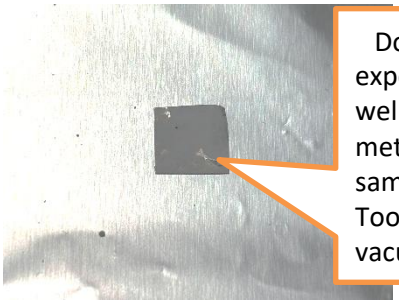
Release valve
Once the cylinder rises slightly, tighten it immediately.





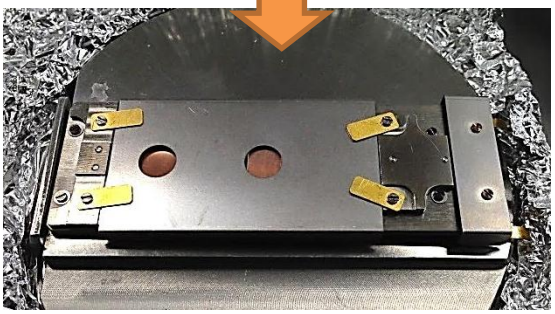
・Embedding Powder Using Indium (In) Foil

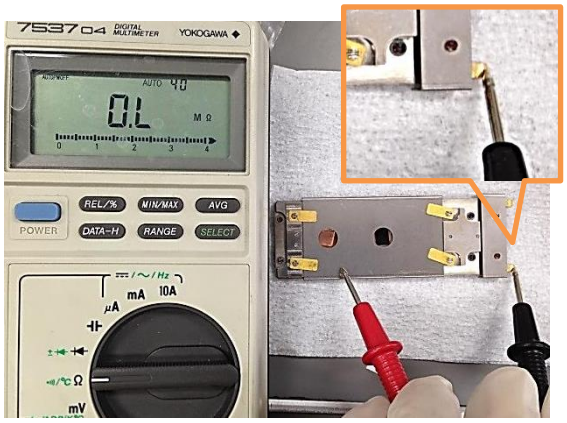
1. Cut two 3 cm square pieces of aluminum foil and one 1 cm square piece of In foil.
2. Place the In foil on top of one piece of aluminum foil and spread the powder sample flatly with a spatula, approximately the size of several spoonfuls, on top of the In foil.
3. Place the second aluminum foil sheet on top of the In foil and lightly press it with your fingers.
4. Hold the sandwich of aluminum foils carefully to prevent shifting, and place it on the black base of the press machine.
5. Put the other base plate on top of the sample and apply pressure with the hydraulic press (sufficient pressure within reasonable limits).
6. Release the pressure from the press machine and retrieve the sample.
7. Carefully remove the top aluminum foil without tearing it.Cut the sample, along with the aluminum foil and In foil, to the required size (around 5–10 mm square).
8. Using tweezers, gently remove any loose powder by applying vibration or impact.
9. Attach the sample to the holder and blow off any loose powder using a blower.



Do not analyze areas where In foil is exposed. If the sample doesn't adhere well, remake or change the fixation method. After preparation, secure the sample with a fastener or carbon tape. Too much sample will delay the vacuum process.

Sample preparation (Appendix)





・For charge-neutralizing sample holder

The neutralization effect is enhanced by applying a bias voltage to the sample. By applying +100V to the sample's bottom and creating a potential difference with the GND lid, neutralized electrons are efficiently directed to the sample surface. This also helps suppress surface potential shifts during Ar^+ ion gun etching.

Place the sample, close the lid, and secure it. The sample thickness should not exceed 1.3mm, and ensure the inside metal parts of the holder do not contact the lid. Make sure there is no electrical continuity between the lid and terminal parts after fixing, as continuity will prevent voltage application and nullify the effect.

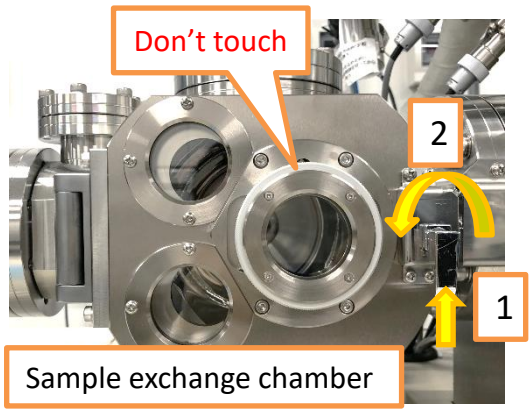
・Surface Contamination of Samples

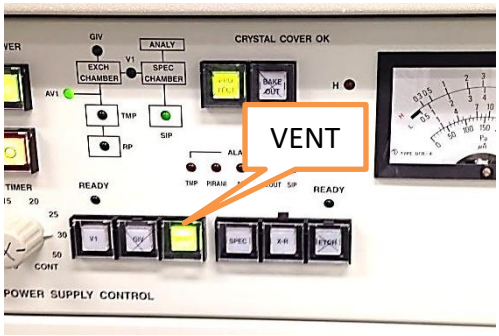
To prevent surface contamination from air, samples created by dry processes should be immediately wrapped in aluminum foil or stored in a vacuum chamber to avoid exposure to the atmosphere. For samples made by wet processes, clean them with acetone, followed by ultra-pure water rinsing, thoroughly blow dry with air duster, and then wrap them in aluminum foil or similar material.

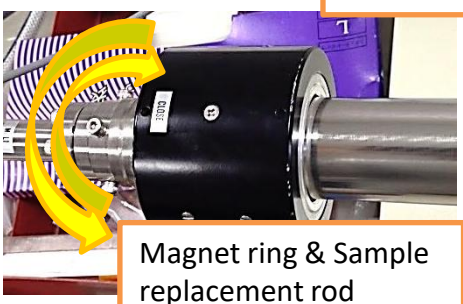
・Reference Peak for Samples

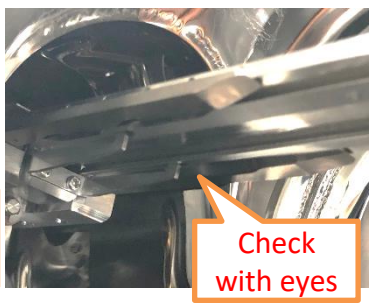
To correct charge shift, it's helpful to include a reference peak on the sample, especially if one is absent. For carbon-containing samples, the C1s peak may overlap with the sample's carbon, making it unsuitable. Use an Au thin film or mesh, or coat the sample with Au for measurement. For powders, mix in another powder with known chemical states, but be cautious as charge shifts may differ.

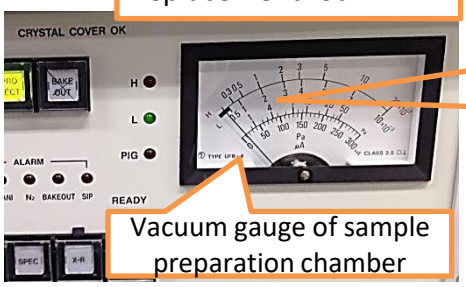
Set into the sample exchange chamber











Introducing the Holder into the Sample Preparation Chamber.

1. Unlock the door and press the VENT button to return the chamber to atmospheric pressure.
2. Slightly move the magnet ring forward to make the rail visible.
3. Place the holder horizontally on the rail, ensuring it is securely attached at the back.
4. Rotate the magnet ring on the sample insertion rod from "OPEN" to "CLOSE.“
5. Check that the hook is properly engaged by looking through the bottom.
6. Move the magnet ring back, close and lock the door. Press the VENT button again to restore the vacuum.

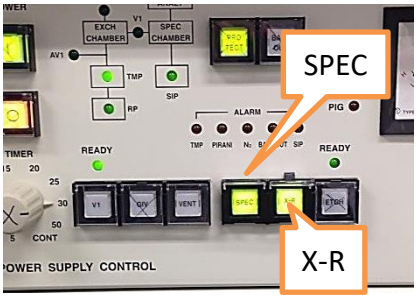
Make sure the holder is properly set before continuing, as failure to do so will prevent the sample from being introduced into the analysis chamber.

It takes about 30 minutes to achieve vacuum for small metal plate samples, while for powder samples, it takes over 2 hours. During the vacuum process, you can proceed with the equipment startup outlined on the next page.

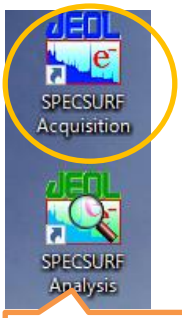
Startup equipment

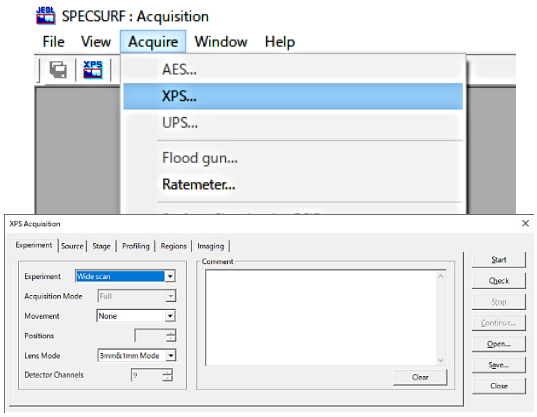
Turn on the chiller first. Next, press “SPEC” button and “X-R” button to light up.

The chiller power supply is always first. Pressing X-R button activate another chiller(you will hear motor sound). If the chiller is not running, press X-R again.



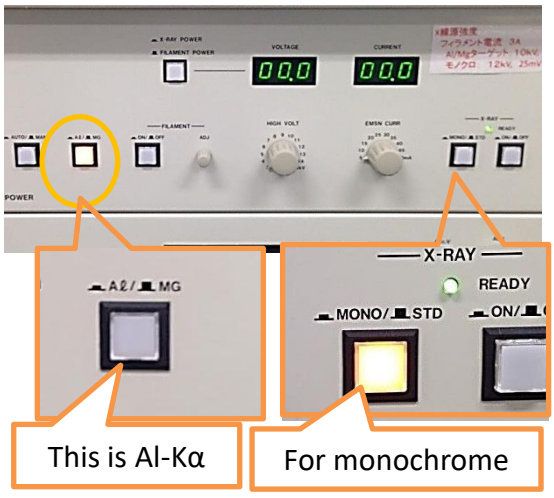
Turn on the stage lamp, the camera power, the camera monitor, XPS-PC, and PC monitors. After starting the PC, click "SPECSURF Acquisition" on the desktop to launch the XPS software.

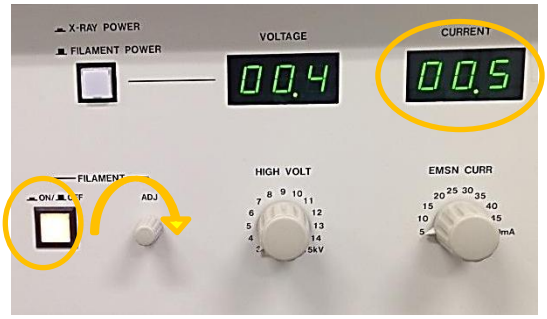


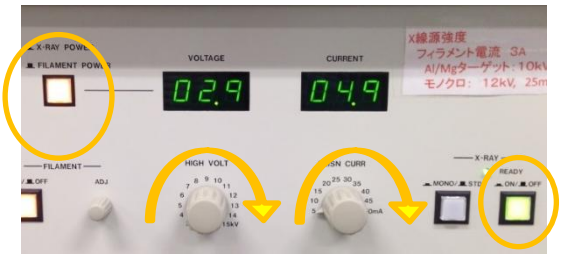


Select “XPS” from the Acquire menu to open XPS acquisition window (if not, PC reboot)

Startup equipment



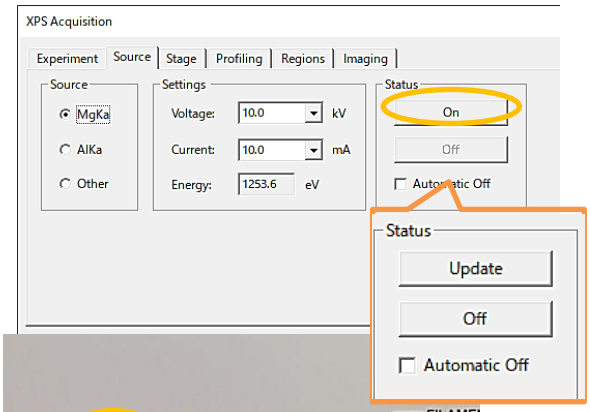


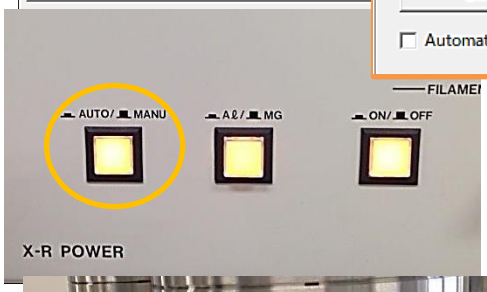


X-ray Setup

1. Choose the type of X-ray source to use
   - Mg-Kα
     About 40% stronger than Al-Kα. The Auger peak appears approximately 230 eV lower than the Al-Kα.
   - Al-Kα
     Can measure higher energy (B.E.) than Mg-Kα. The Auger peak appears approximately 230 eV higher than the Mg-Kα.
   - Monochromatic X-ray (Al)
     Higher energy resolution than Mg-Kα/Al-Kα. No satellite peaks, lower background. Intensity is about 1/10 of Mg-Kα.The sample must be flat.
2. If using Mg Kα, light up the Al/Mg button; if using Al Kα, turn it off. For monochromatic X-ray, select Al and press the MONO/STD button to light it up.
3. Turn ON the FILAMENT power.
4. Slowly turn the ADJ knob to increase the current to 3.5A. And wait for 5 minutes after increasing.
5. Switch the display to "X-RAY POWER" and turn ON the X-ray power. Apply 3kV and 5mA.
6. Increase the voltage by 1kV every 3 minutes until reaching 10kV (for monochromatic X-ray, set to 12kV and wait for 30 minutes).
7. Increase the current by 5mA every 3 minutes until reaching 10mA (for monochromatic X-ray, set to 25mA).
8. Finally, turn ON the Analyzer power.

Startup equipment





Measure size	left	right
3.0mmϕ	36	36
1.0mmϕ	24	12
0.7mmϕ	12	10
0.2mmϕ	12	8
0.03mmϕ	4	1

Select the **Source** tab from the **SPECSURF XPS Acquisition**. After entering the type of X-ray source, voltage, and current values, click **"On"** in the **Status** section.

Check the **Automatic Off** option to set the X-ray to automatically turn off at the end of the measurement. This is useful if you tend to leave the system idle after measurements.

Once the **Status** changes to **Update**, press the **AUTO/MANU** button on the X-ray source unit to switch to **AUTO** mode.

If you wish to change the X-ray source settings later, return to **MANU** mode from **AUTO**.

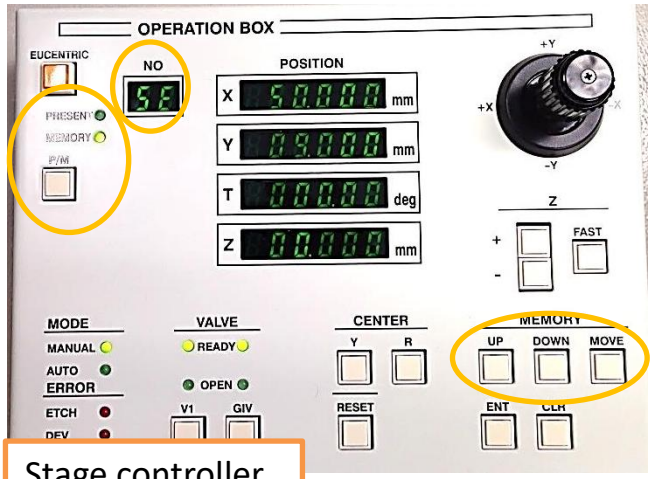
Adjust the measurement range using the two apertures in the left image. Loosen the locking screws, then turn the aperture to the CLOSE direction to narrow or OPEN direction to widen the range, aligning it with the values in the table. For a setting of 36, fully open the aperture in the OPEN direction and adjust to 0.

Avoid over-rotating and turning in the wrong direction.

For comparing pre- and post-etching depth profiles, set the aperture to 1mm ϕ or 0.7mm ϕ. Use the 3mm & 1mm mode for 0.7mm ϕ.

0.2mm ϕ and 0.03mm ϕ have poor sensitivity and cannot measure trace components.

Transfer into analysis chamber

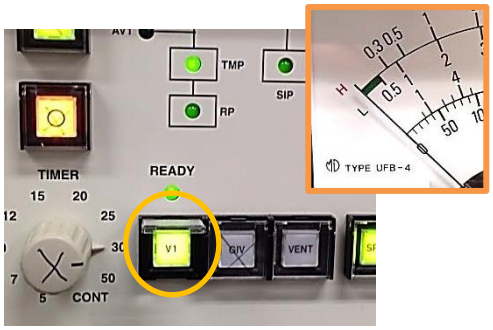


Stage controller

Move the stage to the sample exchange position

1. Press the P/M button on the stage controller to switch to MEMORY mode.
2. Use the UP/DOWN buttons to select memory No. SE.
3. Press the MOVE button to move the stage.

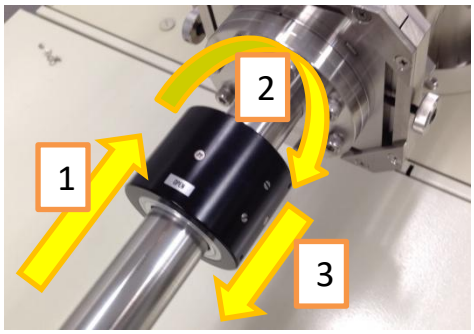
The stage should already be at the sample exchange position, but always verify to ensure correct positioning.



Introducing the Sample to the Stage

1. Ensure the preparation chamber is sufficiently evacuated, then press the V1 button to open the partition.
2. Push the black ring on the introduction rod forward while monitoring the stage through the observation window, guiding the holder into the analysis chamber.
3. Once fully inserted, turn the ring to "OPEN," pull the black ring completely back, and press V1 again to close the partition.

If anything feels off, stop immediately and contact staff for assistance.



The stage will fit in the groove on the back of the holder.

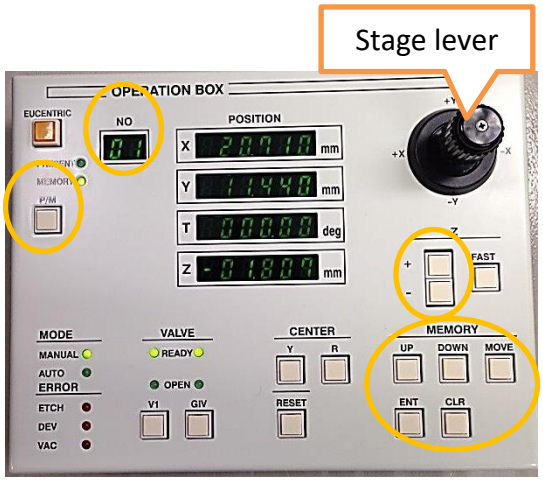
Move it to a position where it cannot move.

Turn the magnetic ring and pull it out.

Transfer into analysis chamber

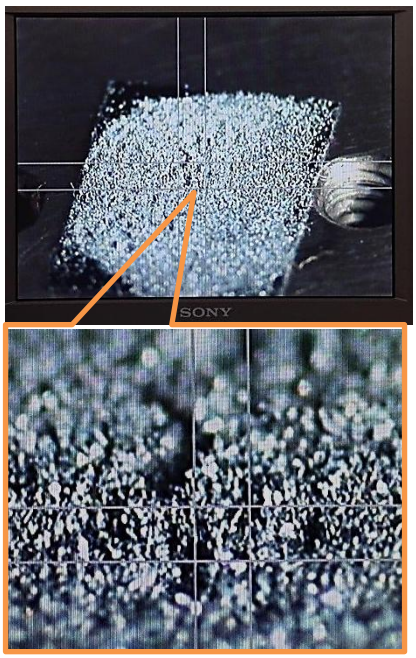
After closing the V1 valve, check the vacuum level in the analysis chamber

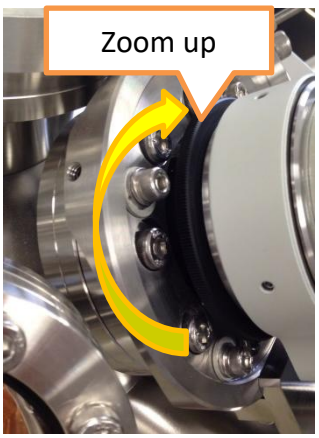
- **Above 5.0×10^{-6} Pa**: Immediately retrieve the sample to the preparation chamber and continue pumping there.
- **Below 5.0×10^{-6} Pa**: Monitor the vacuum level in the analysis chamber until it stabilizes. If the vacuum deteriorates above 5.0×10^{-6} Pa, return the sample to the preparation chamber. Aim for a vacuum level below 1.0×10^{-7} Pa for optimal conditions.



Register Measurement Positions in Memory*(For Mg-Kα/Al-Kα use only)*

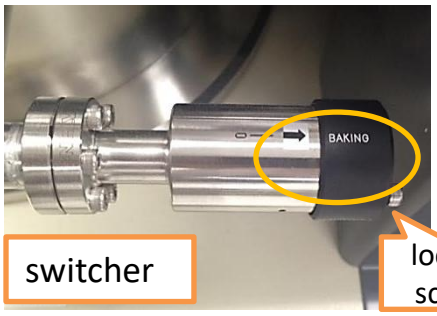
1. Move the stage to the desired measurement position using the stage lever. The measurement center aligns with the crosshair on the camera monitor.
2. Adjust the Z-axis value to focus the camera on the sample at the crosshair using the camera zoom(Lower the stage to account for the sample thickness).
3. Return the zoom to its original setting and verify the XY position is correct.
4. Switch to MEMORY mode by pressing the P/M button. Use the UP/DOWN buttons to select the desired memory number, then press ENT to register the position.
5. Repeat the process to register all measurement positions.

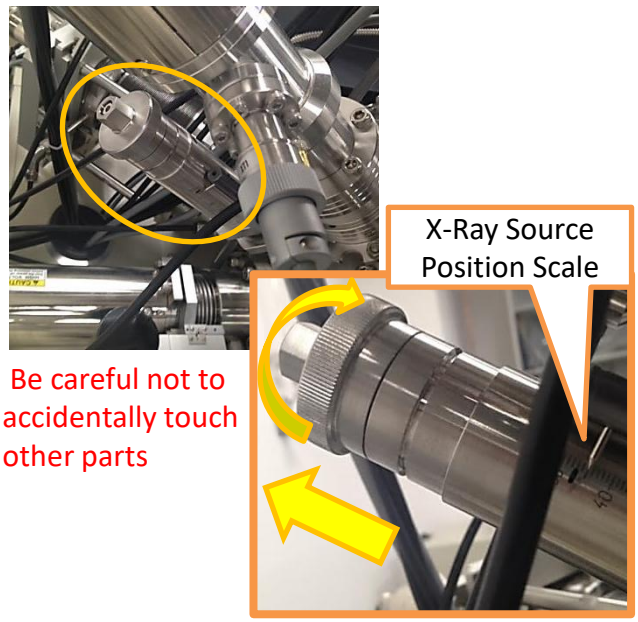




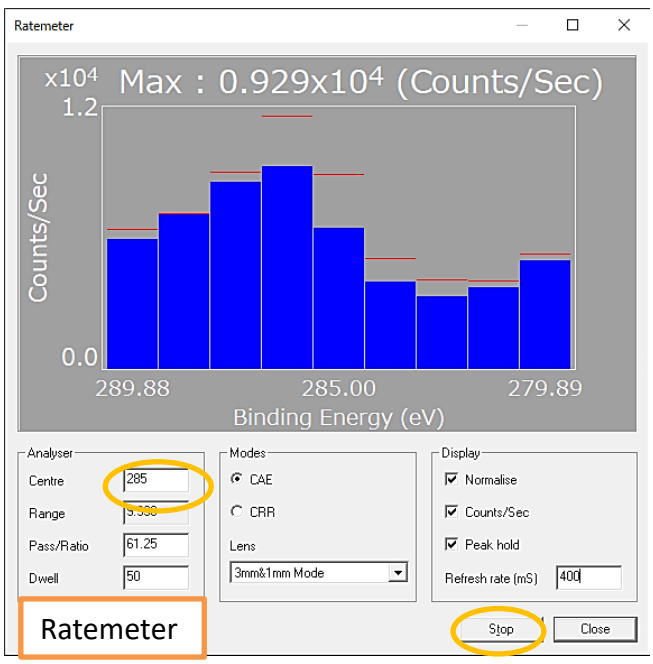
Don’t touch camera body

Setting of monochrome X-ray(Appendix)





Be careful not to accidentally touch other parts



After starting up the monochromatic X-ray, perform the following three steps

<u>Open the spectrometer crystal window</u>

Loosen the locking screw of the switch in the center of the main unit, align the arrow from "baking" to "monochrome," and tighten the screw.

<u>Retract the position of the Standard X-ray source</u>

Turn the knob on the back of the main unit counterclockwise (when viewed from the rear of the device) and set the X-ray source position to 40mm.

<u>Adjust the Z-axis of the stage using the Ratemeter</u>

Tilt the stage to -10° and move the sample to the analysis position. Close the XPS acquisition window, then open the **Ratemeter** window by selecting **Acquire → Ratemeter**. Enter the main peak value of the measurement element into **Centre** and click **Start**. Find the Z-axis value where the peak intensity reaches its maximum and register that position in the stage memory number.

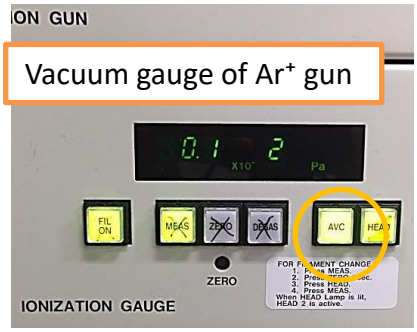
Recommended settings(**Pass/Ratio**: 50 **Dwell**: 400 **Refresh time**: 400)

If the intensity is not sufficient, tilt the stage to -20° to improve measurement. Refer to the "Angle Resolved" high-angle measurement for releasing the tilt restriction.

Setting of Ar^+ etching gun(Appendix)

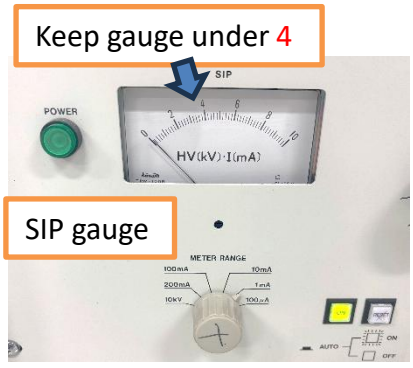
Set up the Ar^+ gun when you want to remove contamination from the sample surface or measure the depth profile. Contact the staff when you use it for the first time.

Use a measurement size of 1 mmϕ or 0.7 mmϕ. Ar^+ etching may affect the chemical state and composition of the sample surface. The etching area is 3 x 3 mm centered on the camera display position.



After transfer the holder, wait until the vacuum in the analysis chamber is less than 10.0×10^{-7} Pa.

Press the AVC button on the ionization chamber vacuum gauge. Slowly open the Ar gas valve from 6 o'clock to 9 o'clock. The vacuum gauge value will barely change until reaching 9 o'clock. Then, slowly continue towards 7 o'clock.

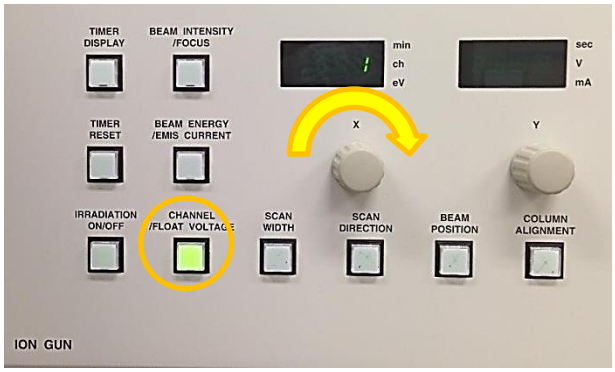


Micro-adjust the Ar gas valve to stabilize the pressure at 10.0×10^{-2} Pa. While opening, watch the SIP gauge (below the vacuum gauge) and keep its needle ≤ 4. If the SIP gauge exceeds 4, close the valve, then reopen slowly toward 10.0×10^{-2} Pa. Wait briefly; if the pressure rises again, tighten the valve and readjust.

Do not exceed 12.5×10^{-2} Pa (error threshold). During analysis, check the SIP gauge regularly. If it approaches 4, pause the run, close the Ar valve to 9 o'clock, wait for vacuum recovery, then resume.

Turn on the auto valve controller, the valve will close, and the vacuum reading will decrease.

Setting of Ar^+ etching gun(Appendix)

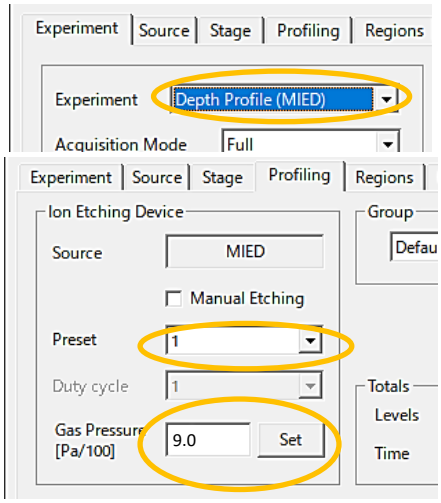


Press the Channel button on the Ion Gun unit and turn the X knob to select the etching condition. The conditions are as shown in the table below.
Etching rate is measured on SiO_2.

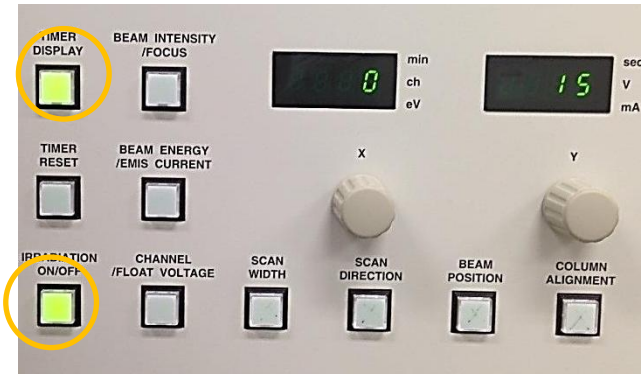
channel	Ch1	Ch2	Ch3	Ch4	Ch5
Beam energy(eV)	3000	2000	1000	500	3000
Emis current(mA)	20	20	20	20	20
Gas Pressure(x10^{-2}Pa)	9.0	8.5	7.7	6.6	9.0
Etching rate(nm/min)	7.5	4.4	1.6	0.3	8.5
Etching rate(nm/min)	5.5	3.6	1.4	0.3	5.3

2025/7/18update

previous rate

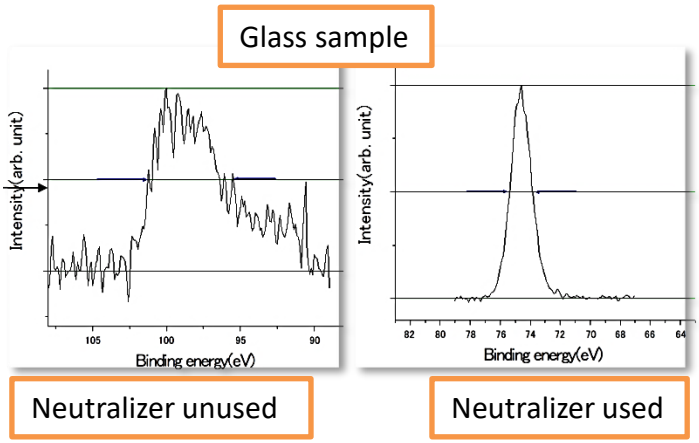


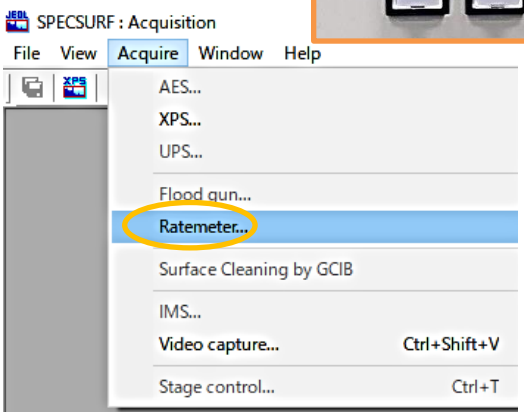
Select “Depth Profile” in Experiment tab, select Profiling tab, enter channel number to be used in Preset, enter Gas Pressure value in above table to Gas Pressure, and click Set.
If you want to do Depth profile, go to "Depth profile" page.



Etching can be started by pressing “Irradiation ON/OFF” button in Ion Gun unit, and the elapsed time of etching will be shown on the display by turning on “Timer Display” (the time will be reset by pressing “Timer Reset”).

Setting of charge neutralizer(Appendix)





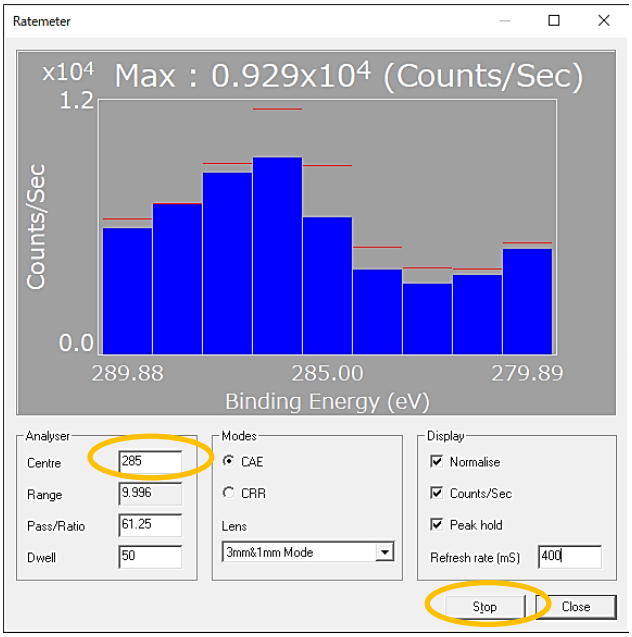
In the case of non-conductive samples, non-uniform charging may occur on the sample surface. The non-uniform charging affects the energy value and the half width. The use of charge neutralizer can alleviate the charging phenomenon by supplying electrons to the sample surface.

It is recommended to use this when a powder sample contains both insulating and conductive materials.

▪How to use charge neutralizer

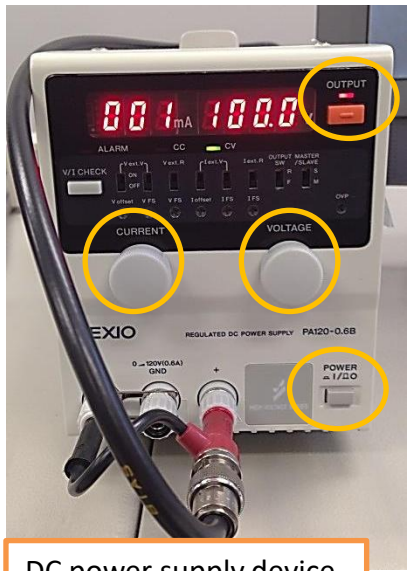
1. Power on charge neutralizer unit
2. Press “SEL” button to change the ADJUST item to “FIL”. and turn the ADJ knob to increase the current value slowly to 6.0A.
3. Select Ratemeter from Acquire in the software menu. If XPS Acquisition is open, close it first.

Setting of charge neutralizer(Appendix)



Find the voltage value that results in not only the highest peak count but also normal peak position and peak full width at half maximum (FWHM). Determine the appropriate voltage value for each sample.

4. Enter the energy value of the highest intensity peak in Centre. Set Pass: 50, Dwell: 400, and Refresh time: 400.
5. Press Start and check the peak shape (if the peak is not visible, perform a wide scan to identify the peak position).
6. Turn on Acc.V. Press the SEL button to change ADJUST to Acc.V, then adjust the voltage using the ADJ knob while monitoring the peak count in Ratemeter. Find the voltage where the peak count is highest (if the peak increases at the screen edge, adjust the Centre value to center the peak).
7. Once the voltage is found, fix the value, close Ratemeter, and proceed with regular measurements.
8. Perform a brief narrow scan under several electron gun conditions, compare the differences, and determine the optimal condition.



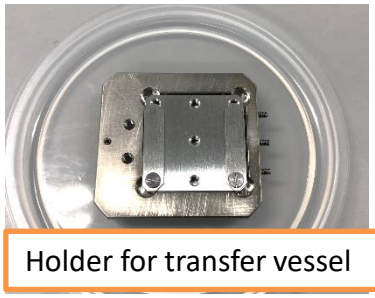
DC power supply device

<u>When using the holder for charged samples</u>

Before setting up the neutralizer, turn on the device as shown in the left figure and apply voltage to the holder.

1. POWER On, and OUTPUT On.
2. Turn the current knob to apply a small amount of current.
3. Turn the VOLTAGE knob to set to 100.0V.

Setting of transfer vessel(Appendix)

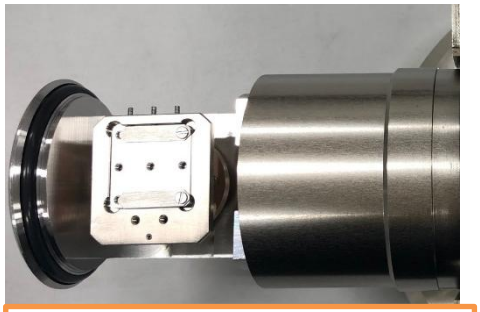


Holder for transfer vessel

Do not touch the left part from here with your bare hands!

Transfer vessel

Opening/closing knob

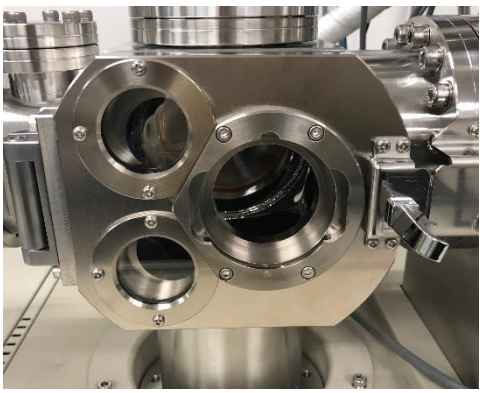


Fix the holder in this orientation.

- ✓ Always contact facility staff when using a transfer vessel. Do all work carefully.
- ✓ Do not give any shock to the transfer vessel. Make sure that there is no dust on the O-ring. Be careful when transferring the holder.

After fixing the sample to the holder in the glove box, mount the holder in the transfer vessel. Pay attention to the direction of the holder when mounting it (see the figure on the left). Turn the opening/closing knob to tighten it firmly.

Remove the lock of the sample preparation chamber door and remove the blank flange from the door after VENT.



Blank flange
Do not touch the inner part with your bare hands!

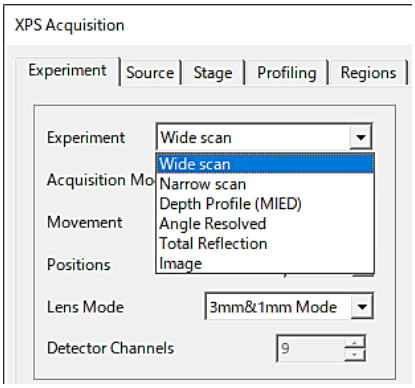
Setting of transfer vessel(Appendix)

Align the pins to attach the transfer vessel to the door and turn the fixing ring to fix it to the door. Lock the door and press the VENT button to evacuate.

When the vacuum in the sample preparation chamber exceeds 0.5×10^{-3}Pa, carefully turn the opening/closing knob to open the vessel little by little. Opening the vessel too large will cause the pump to malfunction. Check the vacuum level and open the vessel carefully.

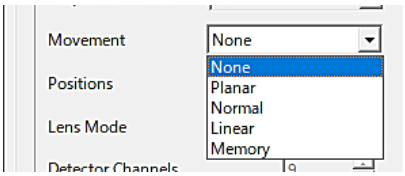
Once the vacuum in the vessel is established, fully open the opening/closing knob. Operate the black ring to retrieve the holder, then close the vessel (but leave it slightly open). Once the vacuum in the sample preparation chamber is sufficiently established, open V1 and introduce the holder into the analysis chamber. Finally, close V1.

Wide scan

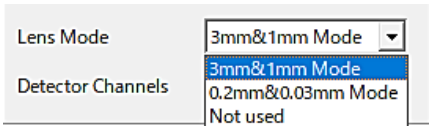


There are a total of six different measurement methods. It is recommended to start with a wide scan for all measurements.

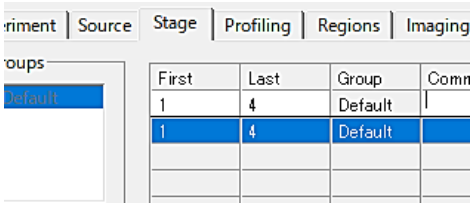
Select “Wide scan” in Experiment.



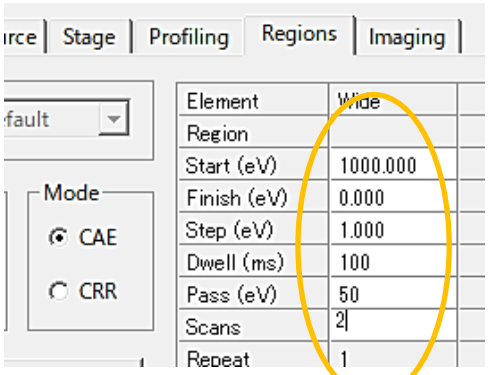
Select “None” for Movement to measure at the current stage position or select “Memory” to use the memory position of the stage controller.



Select the measurement size in Lens Mode.

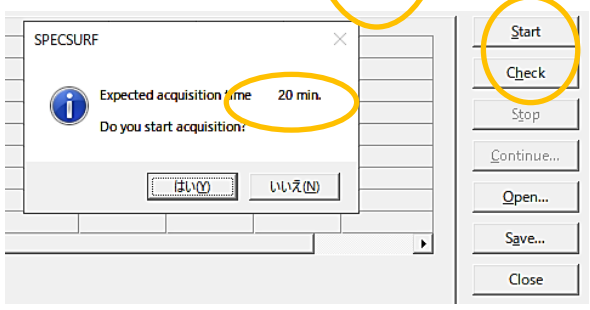


Input the first number (First) and the last number (Last) of the memory number for continuous measurement in the Stage tab (when Memory is selected). Set each condition of Wide scan in Regions tab.



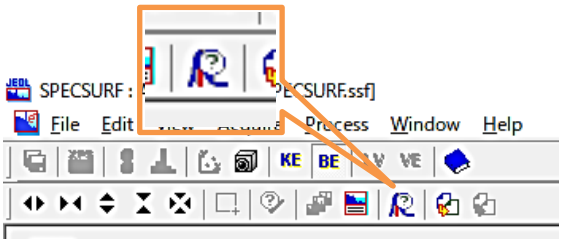
Pass (eV) affects both energy resolution and intensity. A smaller value improves resolution but decreases intensity, with approximately 5 eV being the limit. The Step (eV) interval should ideally be set to about 1/100 of the Pass (eV) value. Scans can be adjusted based on measurements. The background noise decreases by 1/√N with the number of scans.

Recommended conditions for wide scans are as follows: Start: 1200 eV for Mg-Kα or 1400 eV for Al-Kα Finish: 0 eV or -10 eV Step: 1.0 eV Dwell: 100 ms Pass: 50 eV Scans: 1–4 times

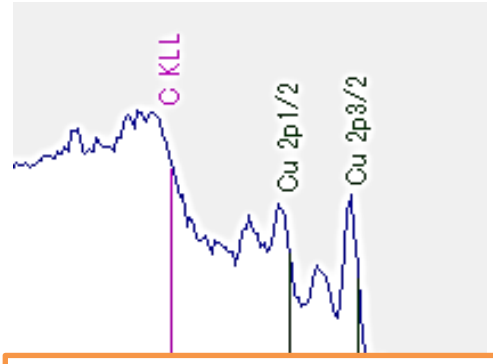


Click “Start” to start measurement and click “Check” to display the estimated measurement time.

Markers

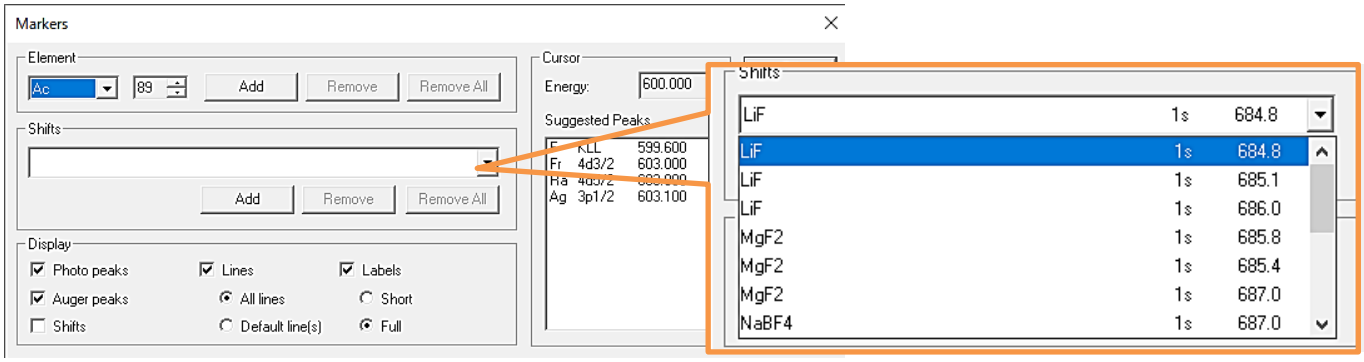


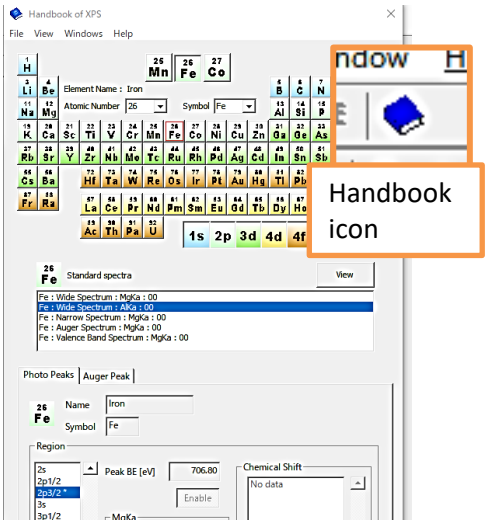
Use markers to identify the peaks that appear in the wide scan spectrum. Click Markers icon on the left.



The position of the marker is based on the standard, so it may be different from peak position of the actual sample.

A line will appear on the spectrum. When you put the line to the peak position, a list of elements will be displayed in “Suggested Peaks”. Select an element from the list and click “Add” to stamp the element name on the graph. And you can select from “Element” item. Some elements also have a Chemical Shift list that can be stamped as well. Clicking OK at the end will leave the stamp.



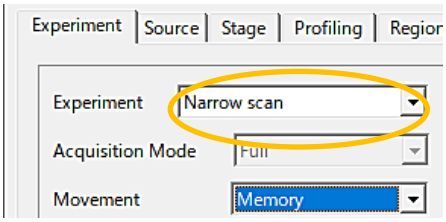


Handbook icon

You can see the spectrum of the standard sample in the XPS handbook, so please refer to it.

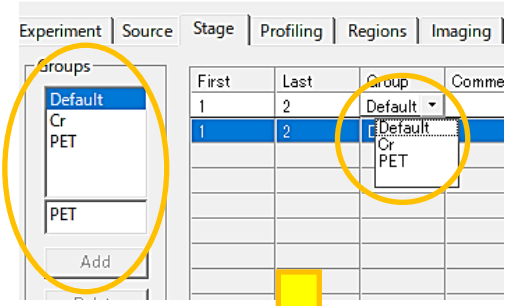
In addition to the photoelectron peak, various satellite peaks appear in the spectrum. In particular, the K-$\alpha_{3,4}$ satellites appear at 10 eV lower energy than the photoelectron peak. And when using Mg-Kα, Satellite peaks called "ghost peaks” derived from O-Kα(730eV higher), Al-Kα(230eV lower), Cu-Kα(320eV higher) appear. When using Al-Kα, same satellites appear.(960eV higher(O-Kα), 230eV higher(Mg-Kα), 560eV higher(Cu-Kα))

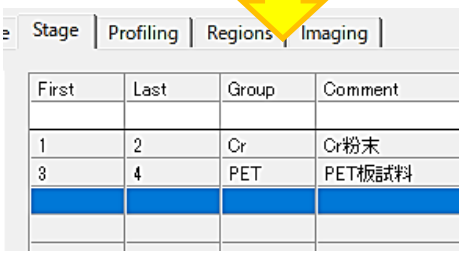
Narrow scan



After the wide scan, acquire spectra near the main peaks of each element in high-resolution mode for quantitative and chemical state analysis. Select “Narrow scan” in the Experiment menu.

Refer to "Wide scan" for Movement and Lens Mode settings.

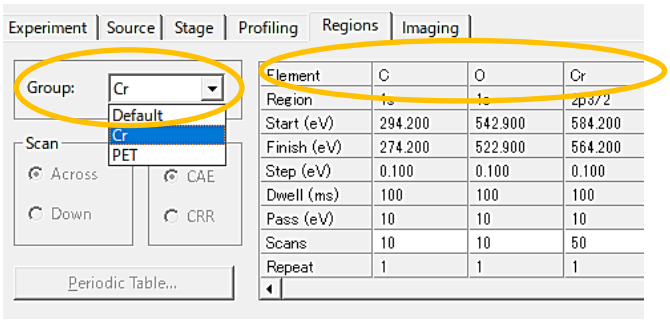




Set the memory number in the Stage tab (refer to "Wide scan" when using Memory). If the measurement content differs depending on the sample, you can group the samples here. Enter the group name in the Groups field and click Add. Input a set of numbers in “First” and “Last”, and select the name from “Group”.

Adding information in the Comment field is convenient.

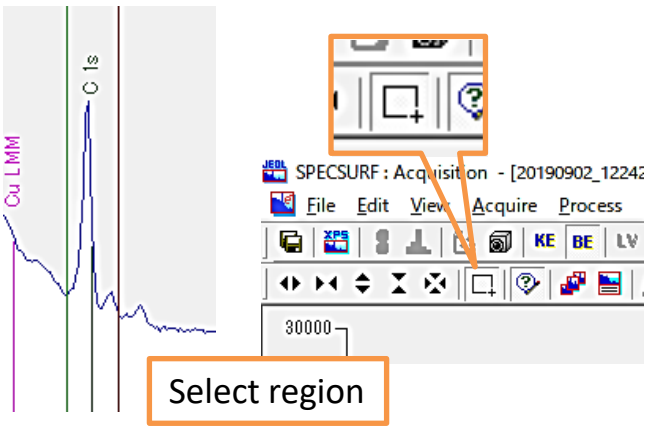
In the example on the left, Memory No. 1 and 2 are registered under the Cr group, while No. 3 and 4 are under the PET group.



In the Regions tab, select “Group” and choose the element to be measured from “Element”.

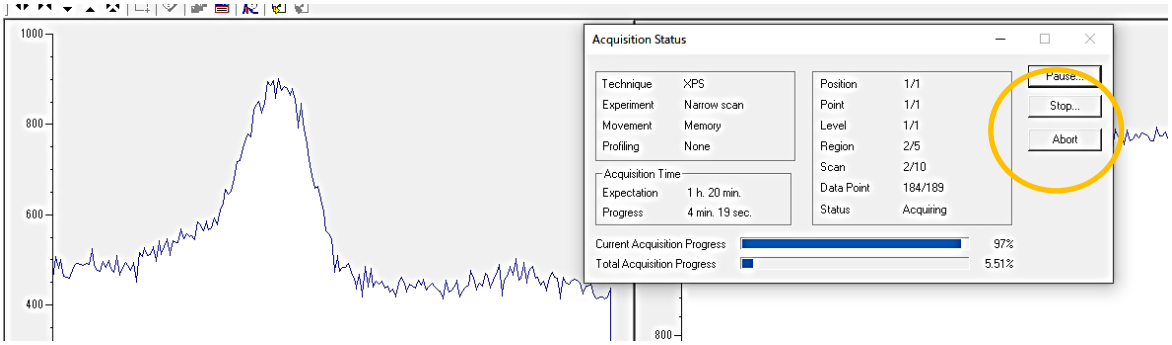
Open the Wide scan spectrum and click the icon shown on the left. This will display the Start line and Finish line on the spectrum. Adjust the line positions to align with the peak, and the measurement range conditions will be updated accordingly.

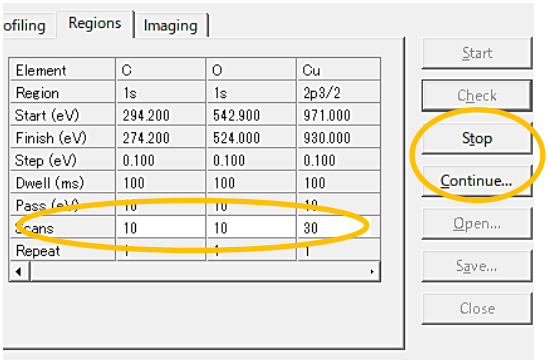
Make sure to check this for all Wide scans.



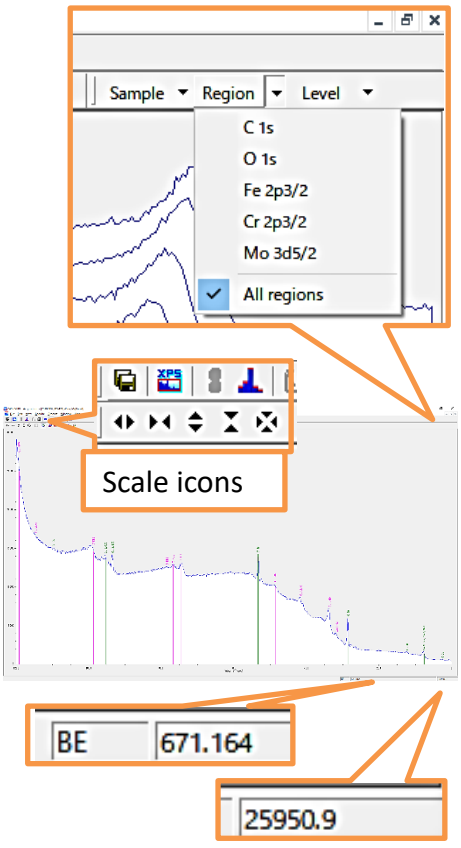
Set the remaining parameters as appropriate (refer to the recommended conditions on the next page).

Narrow scan





If you click “Pause” in “Acquisition Status”, the measurement will be interrupted after one scan, and only the number of scans can be changed. After changing, click “Continue” to restart the measurement. Pressing “Stop” will end the measurement after one scan cycle, “Abort” is force termination.



▪About the spectrum window

When the mouse cursor is placed on the spectrum, the energy position and intensity are displayed in the lower right of the spectrum window. In the upper left corner, there are icons to adjust the display scale. The spectrum obtained by continuous measurement is stored in one window. You can change the display of each sample, each region, and each level from the menu at the top right of the spectrum window.

Save, Open, etc. of data are also done in each window. In the spectrum window, sample numbers are assigned from 0. Don't make a mistake with the sample number.

▪Recommended Conditions for Narrow Scan

Start & Finish: Set the start and finish points at least ±10 eV around the target peak. Always verify that the entire measurement range can be covered across all samples.

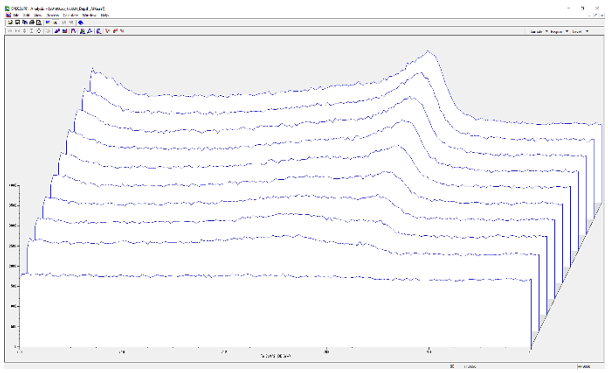
Step: 0.1 eV or 0.05 eV. For Auger peaks, 0.2 eV is acceptable.

Dwell: 100 ms

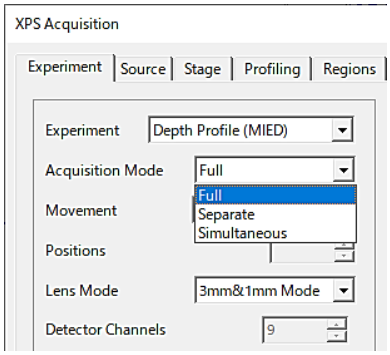
Pass: 10 eV or 5 eV. For the measurement of minor components, set the pass to 20 eV, and adjust the step and dwell to 0.2 eV and 200 ms. The FWHM will degrade by about 20% compared to 10 eV, but sensitivity will increase by 4 times. If performing relative quantification calculations, the pass and dwell should be consistent across all measurements.

Scans: 10–100 scans.

Depth Profile

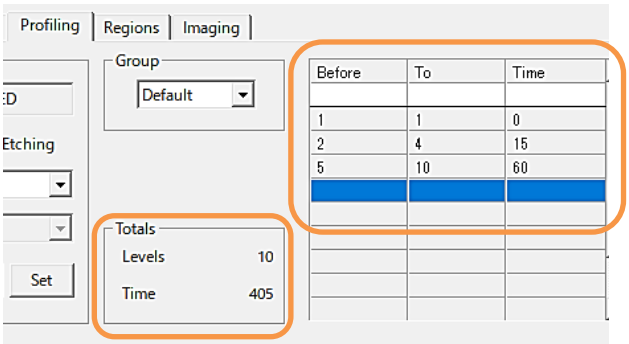


By repeating the Ar^+ etching and measurement alternately, the depth profile can be obtained. Contact the staff when you use it for the first time.



Select “Depth Profile” in Experiment and adjust Ar gas pressure of the etching gun (see “Setting of Ar^+ Etching gun"). Select “Full” in Acquisition Mode.

If you select “Separate” or “Simultaneous”, you can set the energy positions of Peak and Background and acquire only their differential intensities. Since no spectrum is acquired, the measurement can be performed at high speed. (See "Image & Linescan")



In the upper image, the etching time is set to 0 seconds before the first measurement (in the unit of level), each 15 seconds before the second to fourth measurements, and each 60 seconds before the fifth to tenth measurements. The total etching time is 405 seconds.

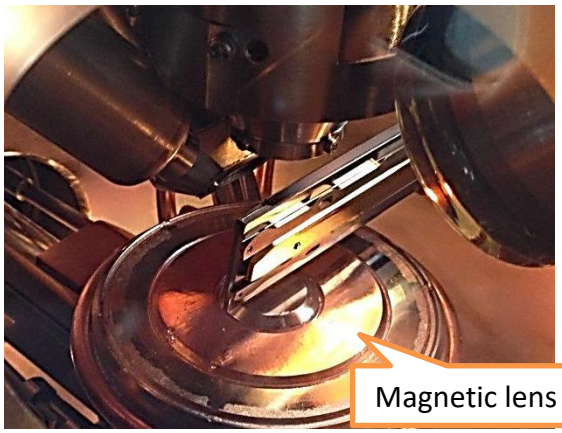
Set the etching time in “Profiling” tab.

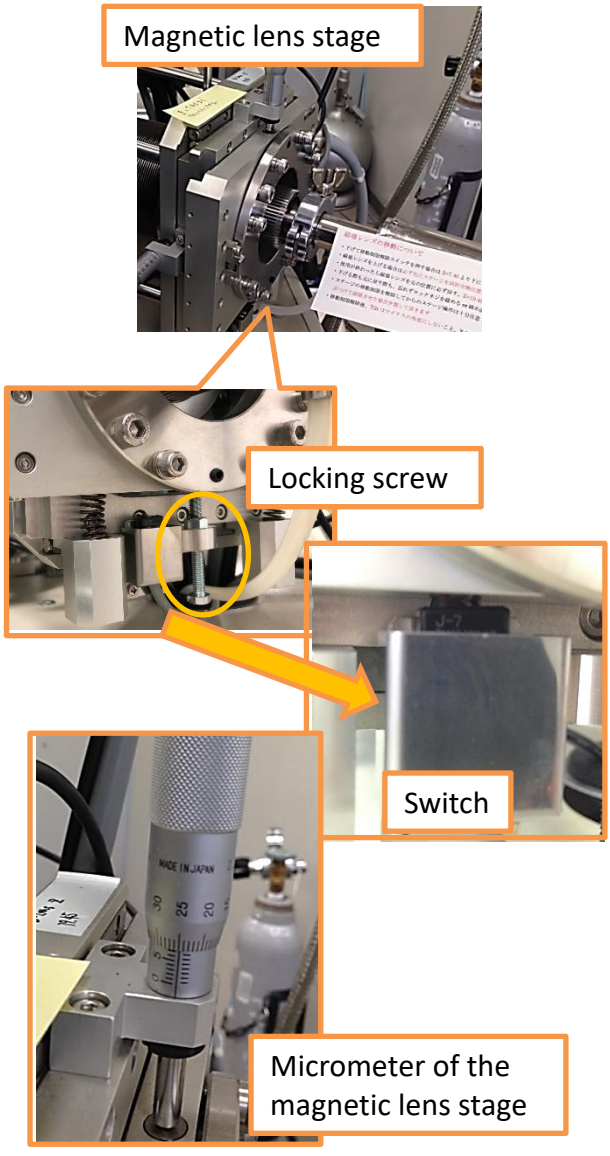
“Regions” settings are the same as "Narrow scan".

Etching changes the state of the surface potential and chemical state, and the peak position may move significantly, so you should measure a little wider.

Atomic mixing, selective sputtering, etc. may need to be taken into account in the analysis.

Angle Resolved





By measuring the spectrum while the stage is gradually tilted toward the analyzer axis, the depth profile of the sample can be obtained nondestructively. Contact the staff when you use it for the first time.

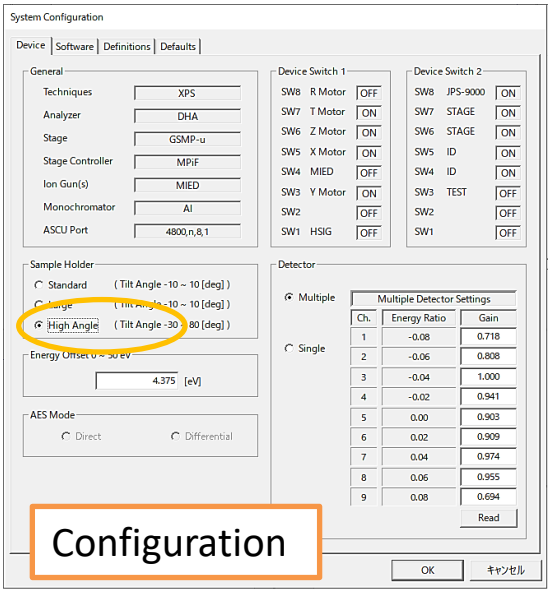
The stage can be tilted up to 80° to obtain a depth profile from 6 nm to 1 nm. A high tilt angle extends the area of the surface to be measured in the Y-axis direction, increasing the amount of signal. It can be applied to the analysis of minute amounts of components.

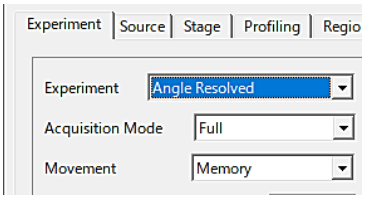
The stage will be moved a lot, so please be very careful. After analysis, be sure to restore the settings of both hardware and software. And, be sure to delete the positions registered in the stage memory.

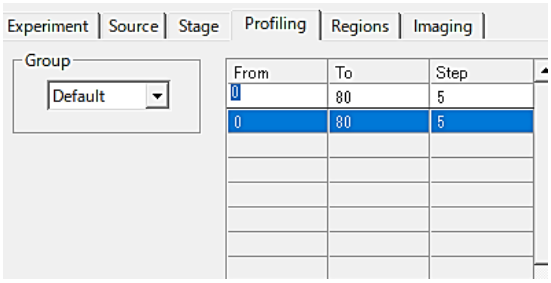
・Setup of the XPS main unit

Transfer the holder to the analysis chamber. Be sure to fix the sample firmly as it will be tilted. Loosen the locking screw and turn the micrometer of the magnetic lens stage to lower it from Z=17.95 to Z=8.5. Make sure the switch at the bottom is pressed (you should hear a small click. Be careful not to lower it too far or it will break). Tighten the loosened locking screw again.

Angle Resolved







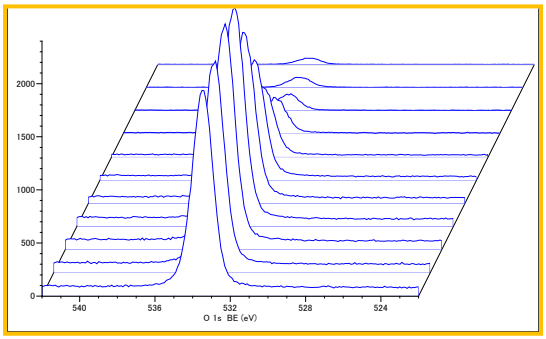
・Setup in Spec Surf software

Go to "View" -> "Configuration" to launch "System Configuration", and select "High Angle" for "Sample Holder". the tilt limit will be released, allowing you to move the stage from -30° to 80°. After that, please operate the stage with great care. If you only want to do wide and narrow scan at high angle, you can continue as usual.

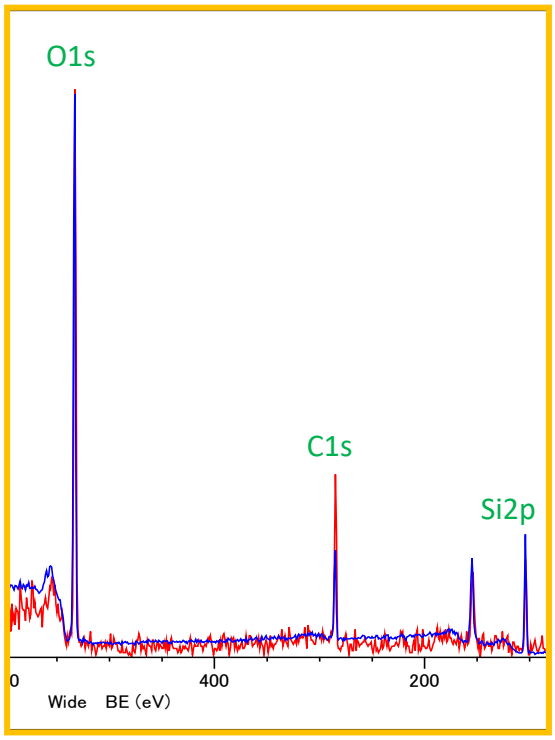
Select "Angle Resolved" in the Experiment tab, and set the tilt angle of the stage in the Profiling tab. In the left picture, XPS acquires spectra at each level while tilting from 0° to 80° in 5° steps. If you want to tilt the stage more than -10°, be sure to move the position of the X-ray source back by 5 mm (see "Setting of monochrome X-ray").For the rest of the settings, refer to "Wide Scan" and "Narrow Scan".

← If the holder is tilted in the minus direction, there is a risk of it hitting the X-ray source. Basically, it is prohibited. Please consult with staff.

Total Reflection



check the tendency of the signal-to-noise ratio.



Normal wide scan Total Reflection scan

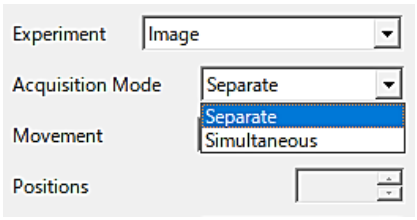
You can see that the intensity of the contamination C1s peak is about twice as large as in the normal measurement.

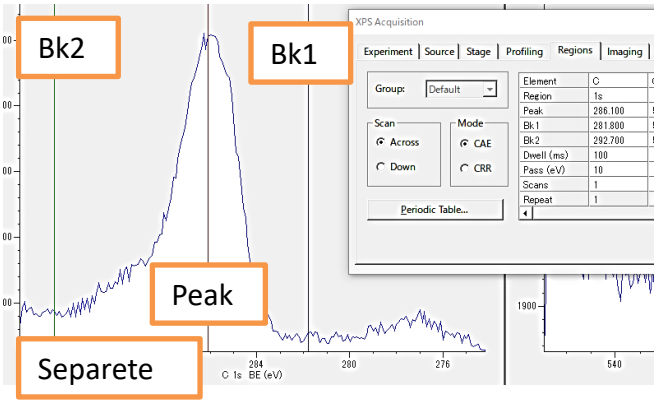
Total Reflection uses monochrome X-rays, and by adjusting the position of the stage so that total reflection of X-rays occurs, it is possible to obtain spectra that are about two-thirds shallower than the normal measurement depth, and with a better signal-to-noise ratio due to the no penetration of X-rays. It is mainly used to evaluate contamination on the surface of a sample. Only samples with smooth surfaces (such as Si wafers) can be measured. Contact the staff when you use it for the first time.

・Measurement Method

1. Place the sample in the holder so that the sample surface is parallel to the stage, and transfer the holder.
2. Set up the monochrome X-ray and adjust the Z position while checking the photoelectron spectrum of the sample with Ratemeter (see " Setting of monochrome X-ray ").
3. Select "Total Reflection" in "Experiment", tilt 0.2° from -10° to -5° , and acquire the spectrum, and check the tendency of the signal-to-noise ratio. (see " Setting of Total Reflection")
4. Check the spectrum with Ratemeter by 1° from -4° to -1° , and fine adjust Z again to get the intensity.
5. After the Z adjustment, measure at the angle with the best S/N ratio. Basically, -1° .

Image & Linescan





Element	C
Region	1s
Centre	287.100
Range	9.800
Channels...	Default
Dwell (ms)	100
Pass (eV)	60.05
Scans	1
Repeat	1

Channels

Channel	Energy	Setting
1	291.886	Background
2	290.787	Peak
3	289.532	Peak
4	288.415	Peak
5	287.100	Peak
6	285.875	Peak
7	284.626	Peak
8	283.365	Peak
9	282.086	Background

Group: Default

Region: C 1s

Acquire the intensity at each energy position with 9 detectors, and subtract the background intensity from the peak intensity to draw the mapping.

Image can obtain elemental mapping images up to 5.0 x 1.8 cm in size. If chemical shifts are available, it is possible to map the distribution of oxides and metals, for example. Contact the staff when you use it for the first time.

First, obtain a Narrow scan of the element you want to measure. Select "Image" in "Experiment", and select "Separate (PB separate method, easy to set up)" or "Simultaneous (PB simultaneous method, fast measurement)" in "Acquisition Mode".

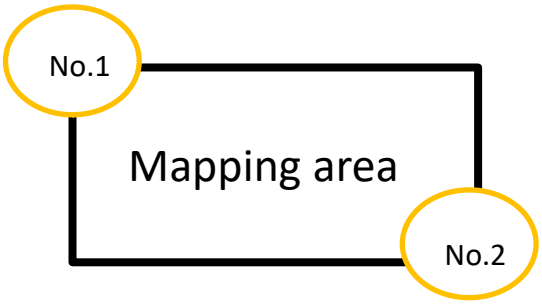
・Separate

Set the values of Peak, Bk1, and Bk2 based on the spectrum of Narrow scan acquired in the Regions tab. Only the intensity at that energy positions are acquired.

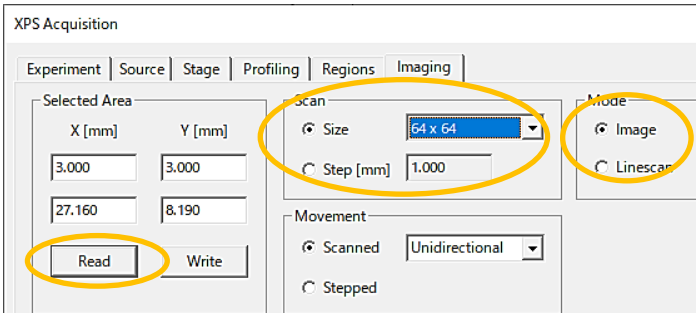
・Simultaneous

Click the Channels column and select whether the intensity to be acquired by each channel (detector) should be Background or Peak. Change the Pass value appropriately so that the high energy side Background, Peak, and low energy side Background of the element to be measured.

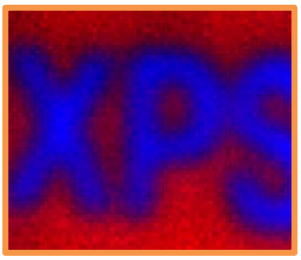
Image & Linescan



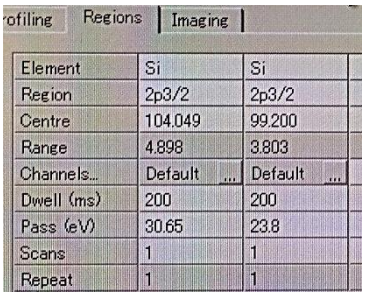
Register the upper-left stage position to memory No.1 and the lower-right stage position to memory No.2 of the sample area for which you want to acquire the image using the stage controller.

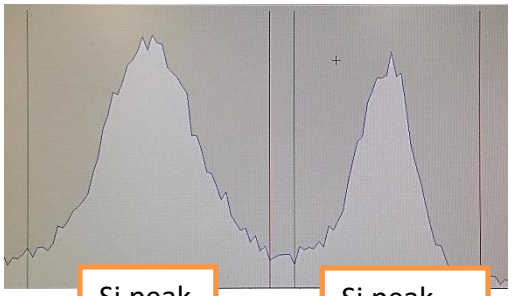


In the Image tab, click "Read" to read the memory positions." In Scan, select either Size or Step and set the resolution of the image. For "Mode", select "Image".

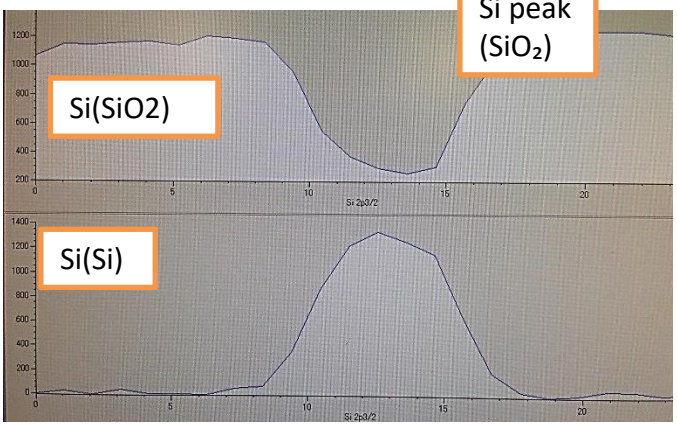


←Mapping image obtained by "Image". red:Si and blue:Au. The image of each element in the mapping result can layered in different colors.



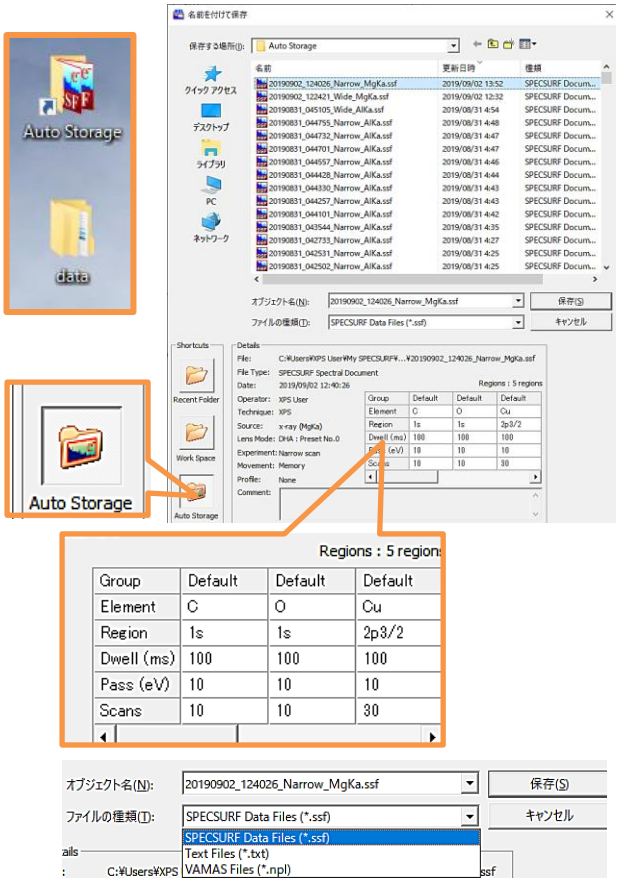


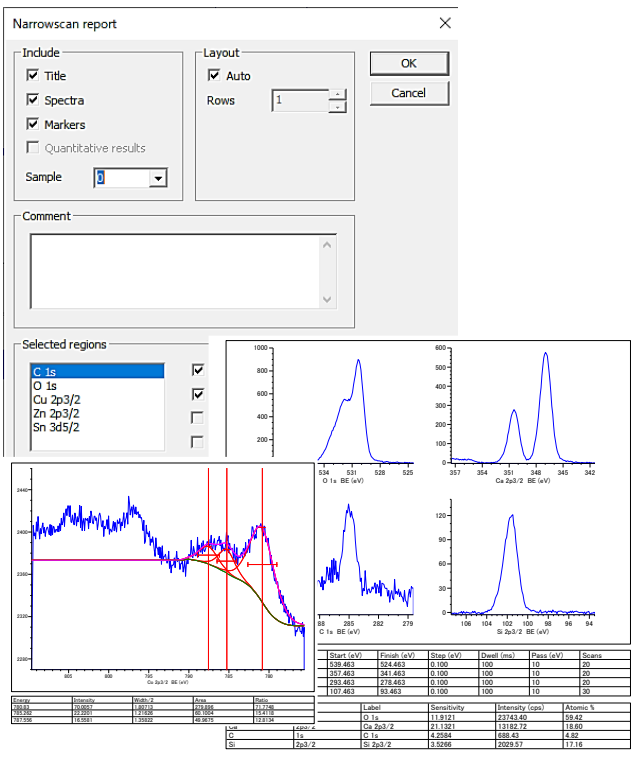
Select "Linescan" for "Mode" to perform line profile from memory No.1 to No.2 position.



←Linescan results by Simultaneous method. This is a Si wafer etched with Ar^+ in the center.

Saving data





All measured spectrum data are automatically saved with the name of the date, measurement method, and X-ray source in “Auto Storage” folder. Select the Auto Storage folder from "File" -> "Open", select the data, the details of the data will be displayed at the bottom of the save window.

To save the spectrum in a different format, select “File” -> “Save as” and choose the file type. In addition to SpecSurf data, text format and VAMAS format can be output.

For users who have indicated "Yes" for data provision in the ARIM usage application form, please ensure to save and take the data in VAMAS format. This will be used for data upload.

You can output the spectrum as an image in report format by clicking "File" -> "Report". Quantification results and waveform separation results can also be compiled into a report.

If you want to keep the data on this XPS-PC, please save them in a folder “data”. Please keep a folder for each laboratory.

Basically, please bring back the measurement data by yourself. We cannot guarantee the preservation of the data.

End procedure

If you have any of the following uses, please perform the following end procedure before the normal end procedure.

・Using the holder for charged samples

Return the voltage and current values of DC power supply device to 0.
OUTPUT OFF.
POWER OFF.

・Charge neutralizer

Return the voltage and current values to 0.
Acc.V OFF.
Unit Power OFF.

・Using Ar^+ etching gun

Auto Valve Controller OFF.
Close the Ar gas valve clockwise to about 9 o'clock (1/4 turn).
Wait for 3 minutes.
Close the Ar gas valve clockwise to about 6 o'clock (3/4 turn).
AVC of Ar^+ etching gun chamber vacuum gauge OFF.
On the ion gun unit, select "Timer Display" and press the "Timer Reset" button.
On the ion gun unit, select "Channel" and turn the X knob to reset it to "7."

・When Angle Resolved measurement carried out

Move the stage to the sample exchange position.
Return the X-ray source position to 45mm(if it has been changed).
Return the Z-axis of the magnetic lens to Z=17.95 and retighten the locking screw.
Reset the holder setting in “Configuration” to “Standard”.
Delete all position registered in the stage memory.

・Using monochrome X-rays

Move the stage to the sample exchange position.
Return the X-ray source position to 45mm.
Return the switcher device to “Baking” and tighten the locking screw.
After 15 minutes have passed since you performed the “X-ray shut down" procedure on the next page, press the "MONO/STD" button to return to "STD".
If the stage tilted above -10 ° , perform the procedure described in “When Angle Resolved Measurement carried out”.

・When using the transfer vessel

Return the holder to the sample preparation chamber, re-seal the holder in the transfer vessel.
Unlock the door and VENT. After VENT, remove the transfer vessel and reattach the blank flange to the door.
Lock the door and draw a vacuum in the sample preparation chamber.

End procedure

This is a common procedure for all measurements. Please be sure to follow the procedure.

・X-Ray shutdown

Press “Auto/Manu” button to change to “Manual”.
Reset X-Ray current to 5 mA and voltage to 3 kV.
Turn off X-Ray power.
Change the display to show “FILAMENT POWER”.
Turn the ADJ knob of FILAMENT to set the current back to 0.
Turn off FILAMENT power.
Click “Off” in the Source tab of XPS acquisition in Spec Surf.

・ Take out the holder

Press the P/M button on the stage controller to change to MEMORY mode.
Press UP and DOWN buttons to display No.SE.
Press the MOVE button to move to the sample exchange position.
Press the V1 button to open the V1 valve.
Turn the magnetic ring to the “OPEN” position and push it forward.
Turn the magnetic ring to the “CLOSE” position.
Pull the black ring out to the end and return the holder to the sample exchange chamber. Press the V1 button again to close the V1 valve.
Open the lock of the sample exchange chamber.
Press the VENT button to return the sample exchange chamber to atmospheric pressure. Turn the magnetic ring to “OPEN” and take out the holder.
Lock the sample exchange chamber and press the VENT button to draw vacuum.

・Finish XPS equipment & other tasks

Remove the sample from the holder, clean the holder, and place it in the vacuum desiccator to draw vacuum. Clean up the workbench.
Turn off analyzer power HT.
Turn off the camera, camera monitor and stage lamp.
Transfer measurement data to PC for analysis using the USB memory of this Lab.
Shut down Spec Surf, PC and turn off display.
After 15 minutes from X-ray shutdown (when using monochrome X-ray, switch MONO/STD to STD), turn off the X-R and SPEC buttons.
Turn off the power of the chiller.
Enter the vacuum level of the analysis chamber and the end time in the logbook.
When using the laboratory at night, turn off the lights and lock the door.

Q & A

In principle, please consult with the staff.

•V1 does not open even after pressing the V1 button
→ Push the black ring of the sample introduction rod all the way back. If the secondary pressure of the nitrogen cylinder has dropped to 0, the cylinder needs to be replaced. Please contact the staff.
•The vacuum is not progressing or the vacuum level is poor
→ Check for debris on the O-ring of the sample preparation chamber door. If the issue is due to the sample, try reducing the number or volume of samples as much as possible. If heating is possible, heat the sample to remove moisture before introducing it. If the vacuum level does not improve, you may have to wait. Please complete as much preparation as possible to ensure the sample is dry.
•No signal is detected even after starting the measurement
→ Check the HT button, verify the aperture value in the measurement range settings, and confirm the status of the monochro/standard button on the X-ray source. If the wrong X-ray source is selected, turn off the X-ray, wait 15 minutes, and then restart with the correct source.
•The peak intensity of the target element is too weak to perform a narrow scan
→ Reassess the sample, review the sample mounting method (e.g., embedding in In foil), check the analysis position, and confirm the peak position. Adjust the measurement conditions to a pass of 20 eV, dwell time of 200 ms, and step of 0.2 eV, and increase the number of scans as much as possible. Note that energy resolution will decrease.
•P-H error is displayed on the vacuum gauge after opening the Ar gas valve
→ The valve was opened too much. Turn off the auto valve controller, rotate the Ar gas valve clockwise one turn to return to its original position, and press the "MEAS" button on the vacuum gauge of the Ar ion gun to clear the error. Then press the "FIL ON" button to restore the display. Open the Ar gas valve slowly and carefully this time.
•Unknown peaks appear, or the spectral shape looks abnormal
→ If contamination is not suspected, try using the neutralizing electron gun as the issue might be due to charging. Additionally, try changing the type of X-ray source and remeasure. If the peak disappears, it may be caused by an Auger peak or ghost peak.