

2022/7/29 update

# X-ray Photoelectron Spectroscopy (XPS) Basic manual Acquisition section

Laboratory of XPS analysis

Contact K. Suzuki Ext. 6882

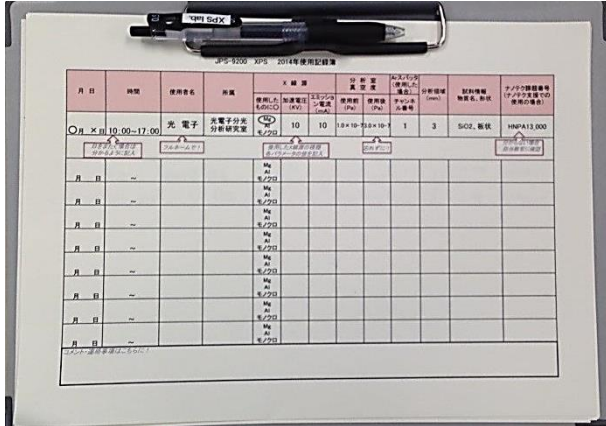
S. Yoshida Ext. 6882

# Caution

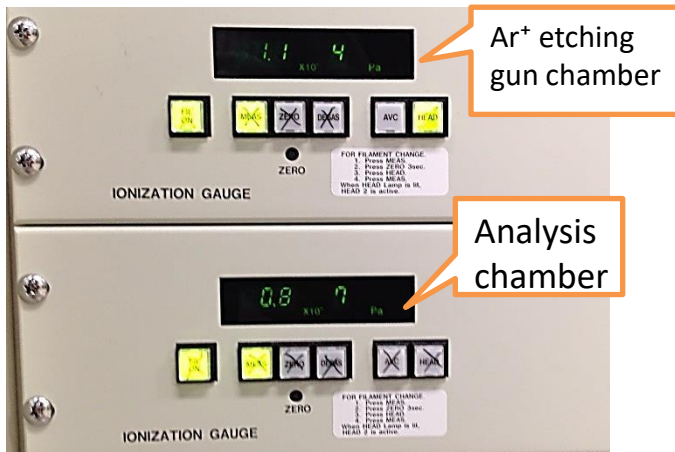
Please keep the following rules.

- Take off your shoes, change into slippers. No food or drink. Clean up the garbage.
- Contact with the staff immediately when you encounter machine trouble. Emergency contacts are mentioned beside the entrance.
- Don't use the machines roughly.
- Don't take out equipments of this laboratory.
- Keep valuables yourself. Lock a door of this laboratory when you leave here.
- Return to original setting after using if you change parameters of software and hardware.
- Don't connect your USB memory to XPS-PC. Use the USB of this laboratory. And take out the data through the PCs for analysis.
- Don't touch with your hand anything which enter XPS chamber. Clean the equipment if you make it dirty.
- Be careful when moving the stage. Do not neglect the visual check.
- Reserve XPS before using by yourself. And use the machine on time. If you want to change your reservation, please cancel by the day before. Cancellation on the day is invalid.
- It is the responsibility of your laboratory if equipment fails due to operation not described in this manual. Students must let your supervisor know.
- If you use first time, contact the facility staff before using and take the training.
- Don't enter fragile sample, too big sample, gas exhaust sample to the XPS chamber without permission. Please contact the staff in advance if you are worried.
- When using the transfer vessel, be sure to contact the facility staff.

# Before using



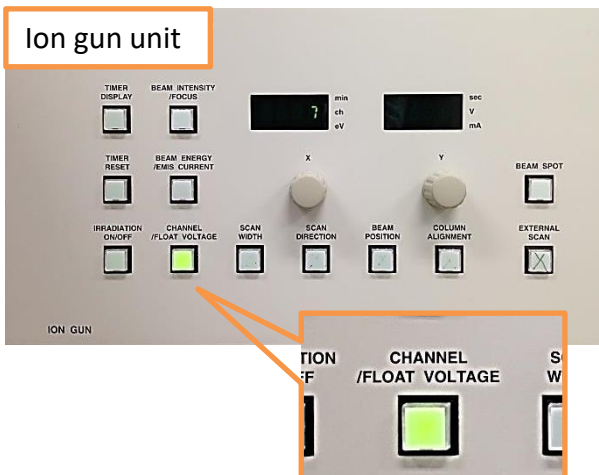
Write your name and start time in the “logbook” and write finish time and parameters after using. Check the analytical chamber vacuum before use and write the value. **Use as scheduled. Change of reservation on the day is invalid.**



Check the Ar<sup>+</sup> etching gun chamber vacuum and the analysis chamber vacuum. Normally,

- ✓ Ar<sup>+</sup> etching gun  $\sim 10^{-4} \text{ Pa}$
- ✓ Analysis chamber  $\sim 10^{-7} \text{ Pa}$

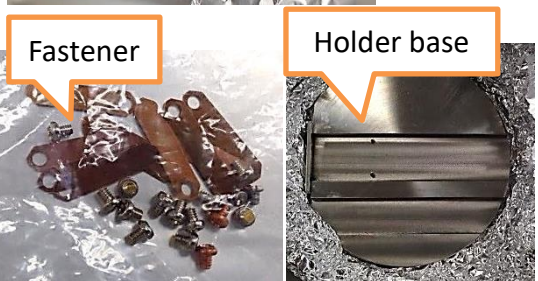
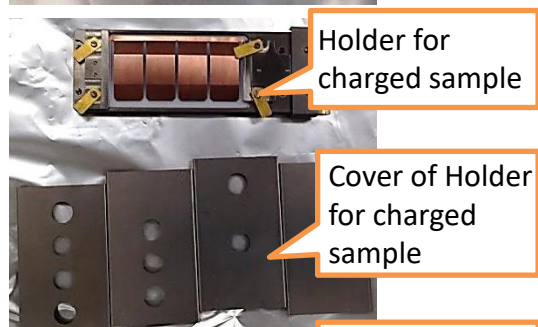
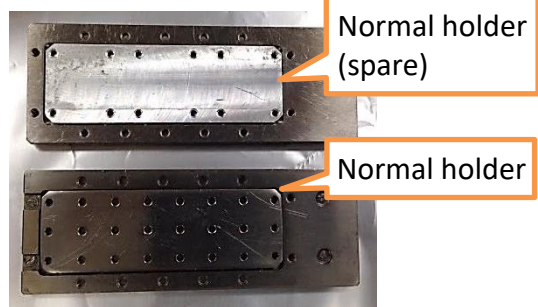
If the degree of vacuum is significantly deteriorated, inform facility staff.



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



Set your sample on the sample holder. Holders are stored in the vacuum desiccator. Use normal holder for normal sample.

Use the large holder for large samples (less than 100mm $\phi$ ). Please contact staff for samples that are worried about setting, such as samples that easily emit gas, are too thick or fragile.

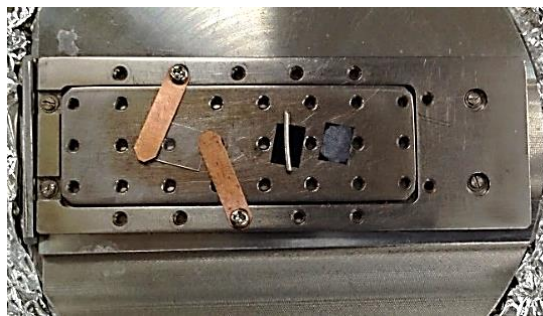
a sample is fixed to the holder using the Fastener or carbon tape. Make sure the sample is completely fixed.

- About powder samples

Use carbon tape or attach (press) to a metal thin film (Al, In, Si, etc.). Blow using blower at least 30 times to completely remove unbonded powder. Vibrate the holder to shake off powder. If even small amount of powder scatter in the chamber, the device will break down. If it causes a breakdown, we will charge a repair fee. Do not put pelletized powder if it is cracked or chipped when setting.



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.

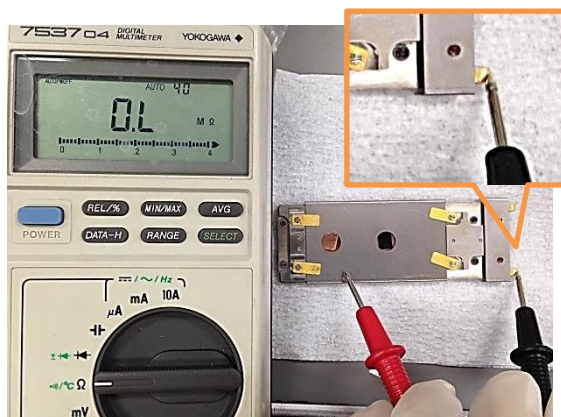
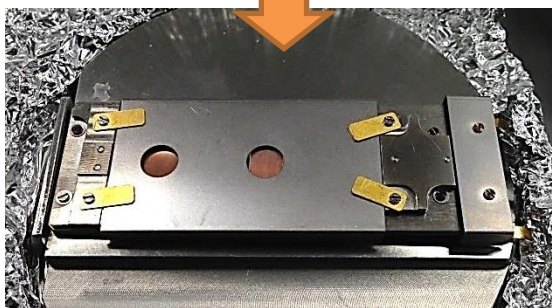


# Sample preparation (Appendix)

## ▪ Holder for charged samples

By applying a bias voltage to a sample, the charge reduction using the neutralizing electron gun is made more effective.

Applying + 100V to the bottom of a sample and applying a potential difference to the cover of the GND allows the neutralizing electrons to be efficiently applied to a sample surface. And it has the effect of suppressing surface potential deviation due to etching when Ar<sup>+</sup> ion etching.



Mount the sample as shown in the photo, cover and fix. The sample thickness is limited to 1.3mm. Set the cover so that do not touch the copper plate of the holder. Make sure a sample surface is below the cover. Confirm that there is no conduction between the holder cover and terminal.

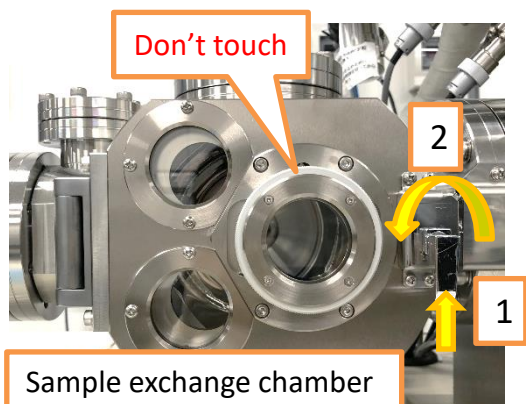
## ▪ About contamination

To prevent surface contamination in the air, samples prepared by the dry process should be wrapped in aluminum foil or stored in a vacuum device immediately after preparation. Samples prepared by the wet process are washed with pure water after washing with acetone. Immediately blow with air duster and wrap in aluminum foil.

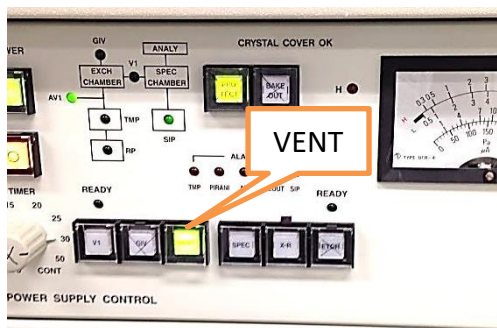
## ▪ About reference peaks

It is convenient to prepare reference peaks in a sample to correct the charge shift of a sample. Samples containing carbon may be more necessary because they may not be available for reference because C1s peak of the contamination and the carbon peak from a sample overlap. For example, Au thin film or mesh is placed together for measurement, or a sample is coated with Au and measured together, or in the case of a powder, other powder whose chemical state is known is mixed and measured together. It is better to do.

# Set into the sample exchange chamber



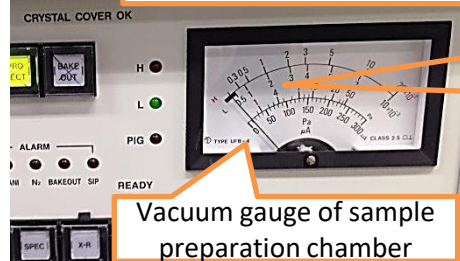
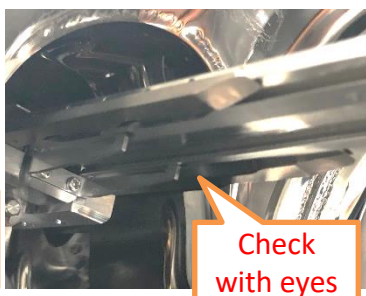
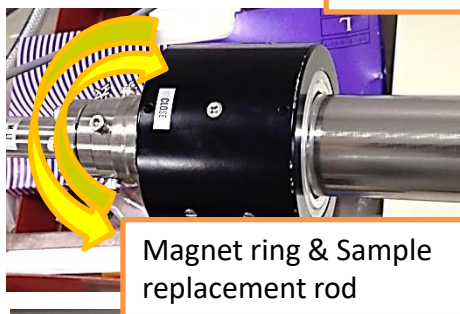
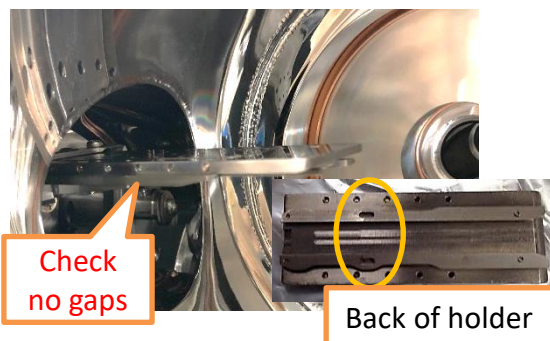
Set the holder into the sample exchange chamber and vacuum evacuate. Unlock the chamber and press "VENT" button to return the chamber to atmospheric pressure. Put the holder horizontally on the rail in the chamber, and close it to the back.



Turn(any direction) the magnet ring on the sample replacement rod to change from "OPEN" to "CLOSE". Turn the ring to raise the hook under the rail. **Look into the bottom and check that the hook is in the hole on the back of the holder.**

Check that the ring is retracted to the rear. Close the door, lock it and press the "VENT" button again to evacuate.

If this setting fails, the holder cannot be introduced into the analysis chamber. Check it is set properly.



Evacuation of the sample takes about 30 minutes for metal plates and about 2 hours for powder samples using carbon tape. Startup XPS device while waiting.

# Startup equipment

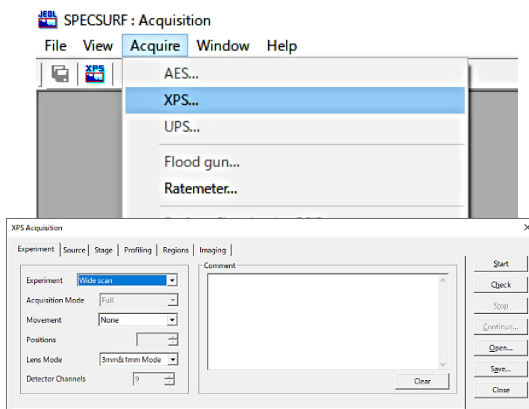
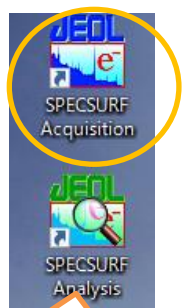


Turn on the chiller first. Next, press “SPEC” button and “X-R” button to light up.

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)

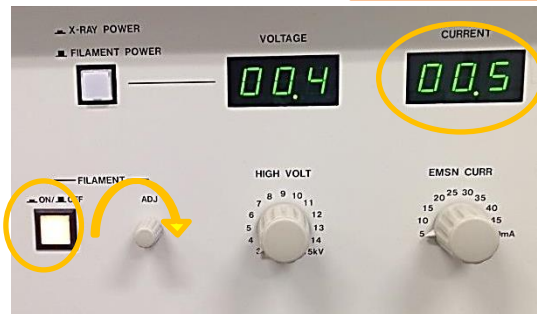
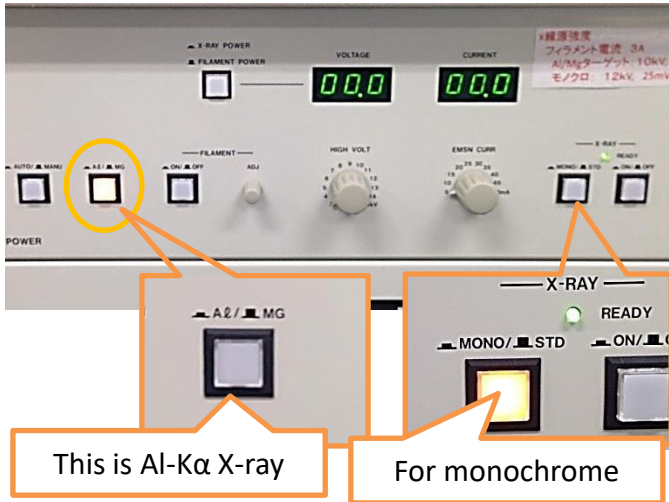


# Startup equipment

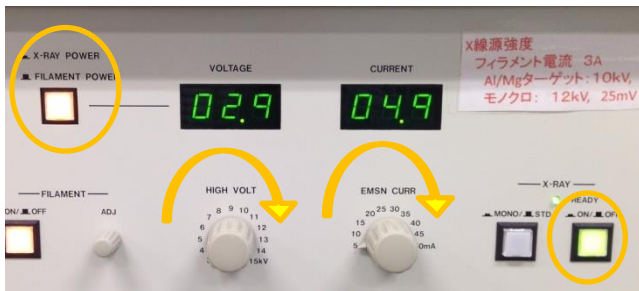
First, select the type of X-ray source from the following list.

- Mg-K $\alpha$  X-ray
- Al-K $\alpha$  X-ray
- Monochrome X-ray (Al)

Turn on the “Al/Mg” button for Mg, and turn it off for Al. For monochrome X-ray, select Al and press the “MONO/STD” button to turn it on.

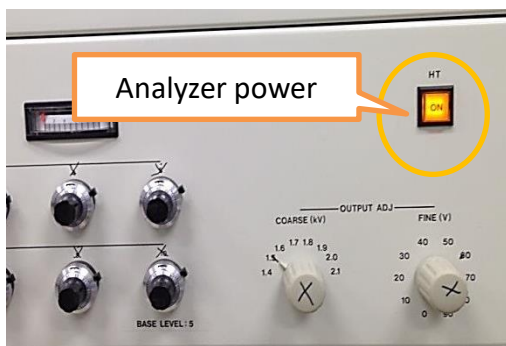


After selecting the X-ray source, turn on the power of FILAMENT, and turn the ADJ knob slowly to increase the current to **3.5 A**. Wait for 5 minutes after increasing.



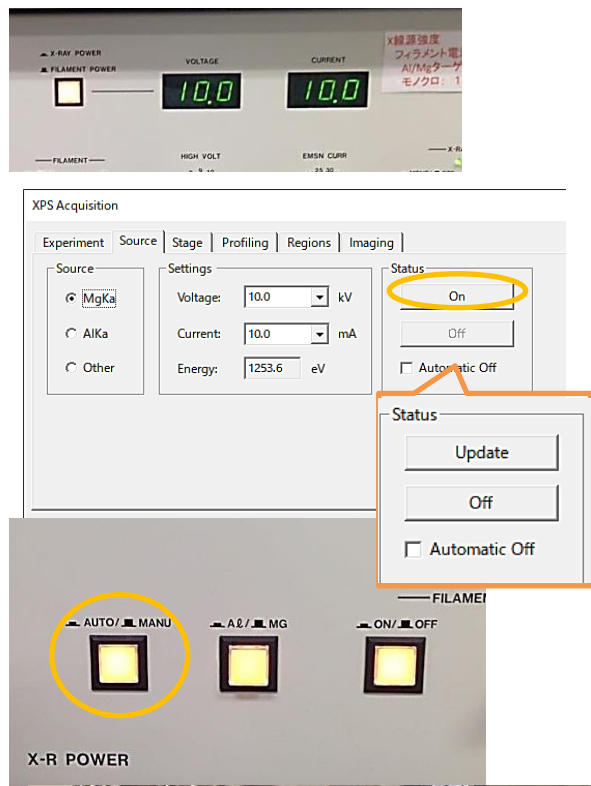
Switch the display to “X-RAY POWER” and turn on the X-RAY power(Rightmost button). Increase the voltage knob **by 1 kV every 3 minutes to 10 kV**. After another 3 minutes, increase the current knob **by 5 mA to 10 mA**. For monochrome X-rays, increase these by one **every 3 minutes to 12kV and 25mA**.

Turn on analyzer power.





# Startup equipment



Select the Source tab from XPS Acquisition in SPEC SURF, enter the type of X-ray source and voltage and current values, and click “On” in Status.

When the Status display changes to “Update”, press the “AUTO/MANU” button on the X-R POWER unit to switch to “AUTO”.

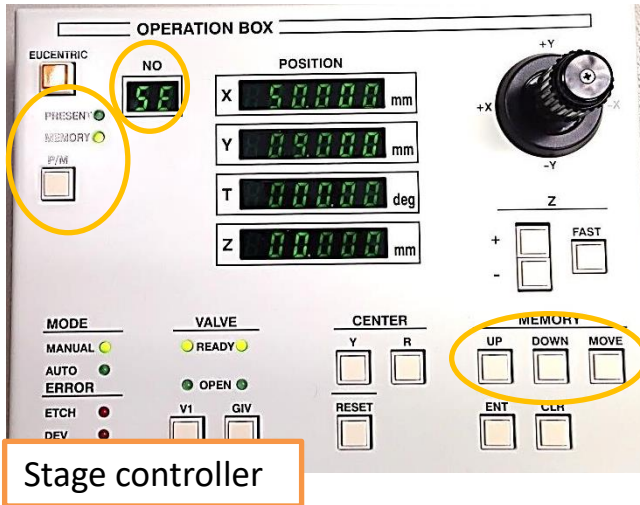


The measurement area can be set by using the two apertures shown in the left picture. Loosen the locking screw and turn the aperture in the CLOSE direction to decrease the number, or in the OPEN direction to increase it to match the number in the table on the left. **Be careful not to turn it too far. 36 is 0 after one turn in the OPEN direction.**

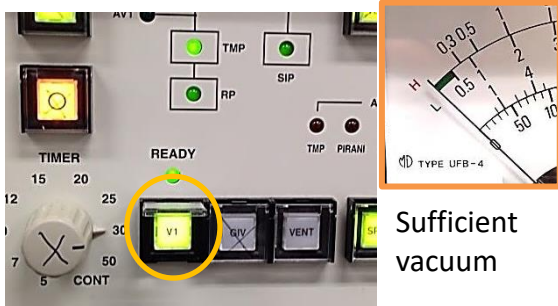
Measure size	left	right
3.0mmφ	36	36
1.0mmφ	24	12
0.7mmφ	12	10
0.2mmφ	12	8
0.03mmφ	4	1

For depth profile, please set 1 mmφ or 0.7 mmφ.

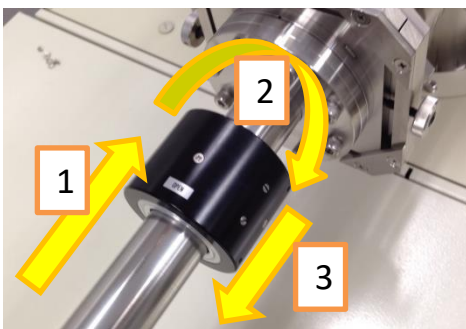
# Transfer into analysis chamber



Move the stage to the sample exchange position. Press “P/M” button on the stage controller to switch the display to MEMORY, then use “UP” and “DOWN” buttons to select the memory No. SE. Press “MOVE” button to move the stage to the displayed position. Basically, the stage will not move because it is already, but be sure to check it.



After the sample preparation chamber is sufficiently evacuated, press the “V1” button. Move the magnet ring to the right to transfer the holder to the analysis chamber. Send it while checking the analysis chamber from the viewing port. When you put it to the end, turn the ring to change it to “OPEN” and return the ring to the left end. Press “V1” button again. If you notice anything unusual, please call facility staff.



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

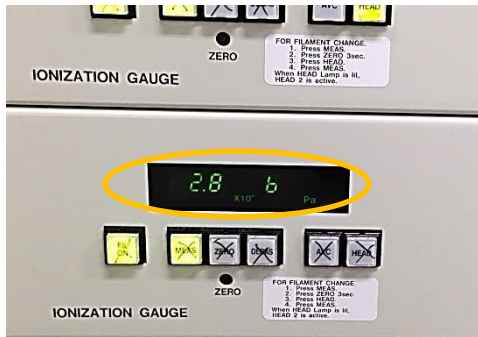


Move it to a position where it cannot move.

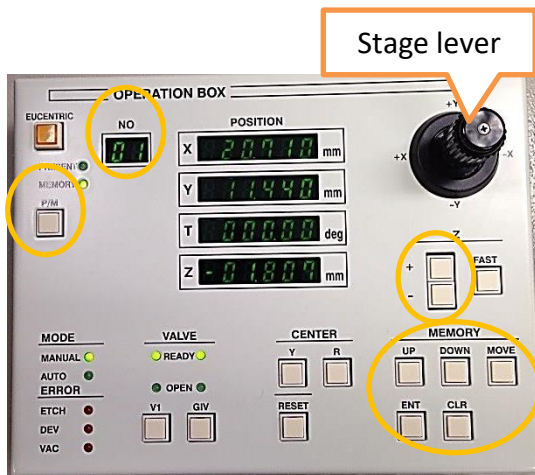


Turn the magnetic ring and pull it out.

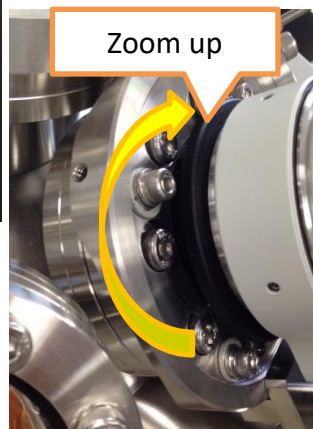
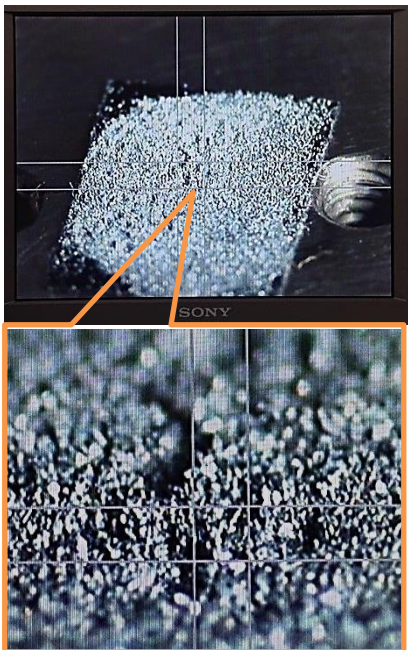
# Transfer into analysis chamber



After the V1 valve is closed, check the vacuum level in the analysis chamber. If the vacuum is worse than  $5.0 \times 10^{-6} \text{ Pa}$ , return the holder to the sample preparation chamber immediately, continue vacuuming for a while, and then retransfer the holder. Wait until the vacuum decreases  $10.0 \times 10^{-7} \text{ Pa}$  or lower.



Operate the stage lever to move the holder. The center of measurement is the cross on the camera monitor. When there are several samples, use the position memory function. Switch to MEMORY mode with "P/M" button, select the No. to be memorized with "UP" and "DOWN" buttons, and press the "ENT" button to memorize the current position.



When aligning the measurement position, be sure to align the Z-axis as well. Zoom the camera to find the Z value that the camera will focus on.

Don't touch camera body

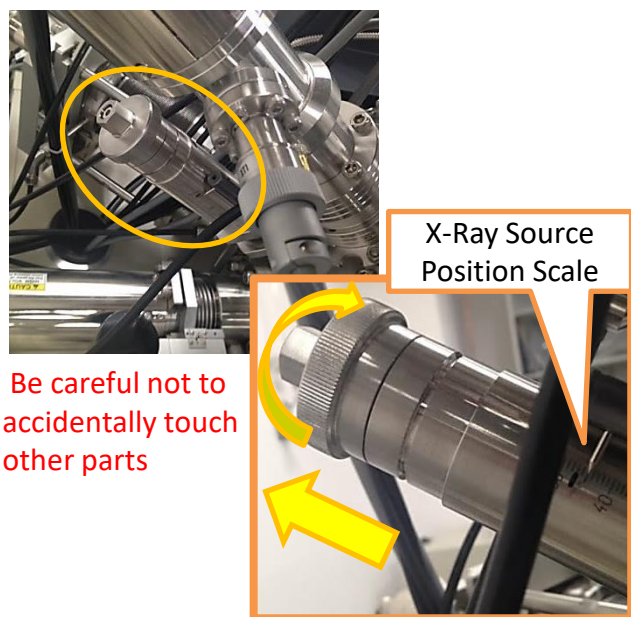


# Setting of monochrome X-ray(Appendix)



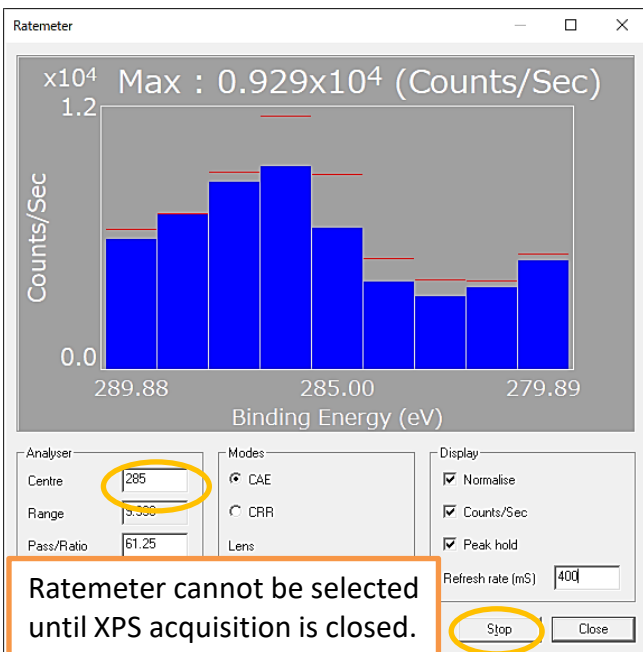
Increase the monochrome X-ray output to **12 kV** and **25 mA** in advance. Contact the staff when using the unit for the first time.

Loosen the lock screw on the left picture switcher in the center of the main unit and change it to monochrome.



Change the position of the X-ray source from **45 mm** to **40 mm** by turning the knob shown in the left photo at the back of the device.

After transfer the holder, move it to the analysis position, change stage tilt to **-10°**. Select "Acquire" → "Ratemeter" in the software, enter the main peak value of the element to be measured in Centre in the Ratemeter window, and click "Start". Find the value on the stage Z axis where the peak intensity is highest, register that position in stage controller. Change the Ratemeter values for "Pass, Dwell, Refresh time". so that you can easily see the increase or decrease in peak intensity with Z movement. If high intensity is not possible, there is a way to tilt up to **-20°**. Please refer to "Angle Resolved" to remove the tilt limitation. Contact staff when using.

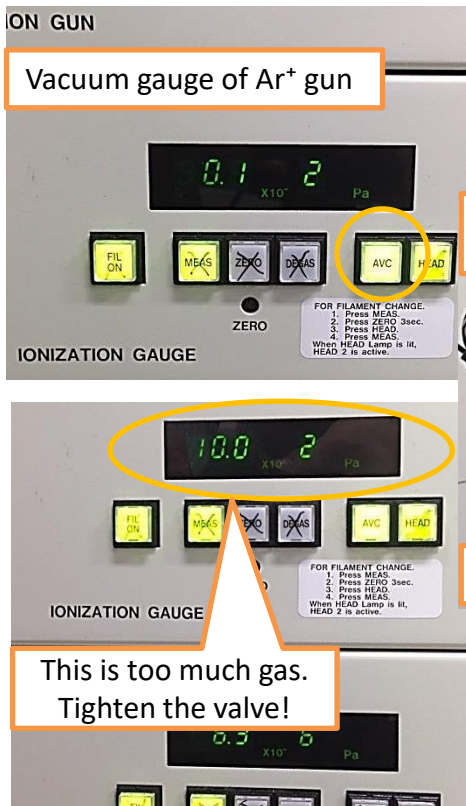




# Setting of Ar<sup>+</sup> etching gun(Appendix)

Set up the Ar<sup>+</sup> 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.

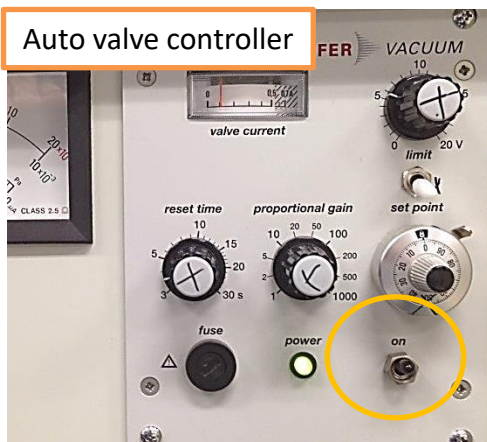
Use a measurement size of 1 mmφ or 0.7 mmφ. Ar<sup>+</sup> etching may affect the chemical state and composition of the sample surface. The etching area is 3 x 3 mm centered on the camera display position.



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

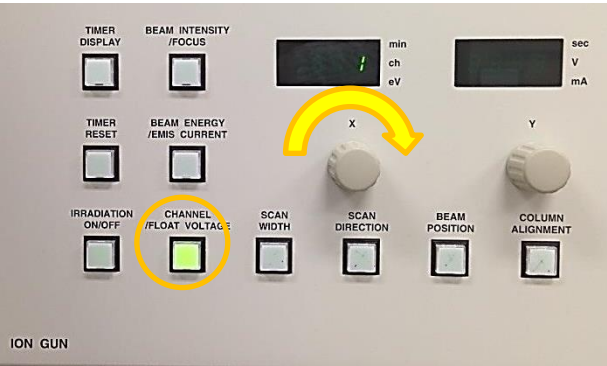


Press "AVC" button on the vacuum gauge of Ar<sup>+</sup> gun. Turn the Ar gas valve on the left picture slowly counterclockwise to adjust the vacuum to  $9.5 \times 10^{-2} \text{ Pa}$ . The valve always has a marker at 6 o'clock direction. About one turn will adjust the vacuum to  $9.5 \times 10^{-2} \text{ Pa}$ . Watch the vacuum gauge and turn it carefully. Too much Ar gas will damage the equipment. Wait 10 minutes after equilibrium is reached.



Turn the lever of the auto valve controller to ON. the Ar gas valve will start to tighten and the vacuum of the Ar<sup>+</sup> gun will drop temporarily.

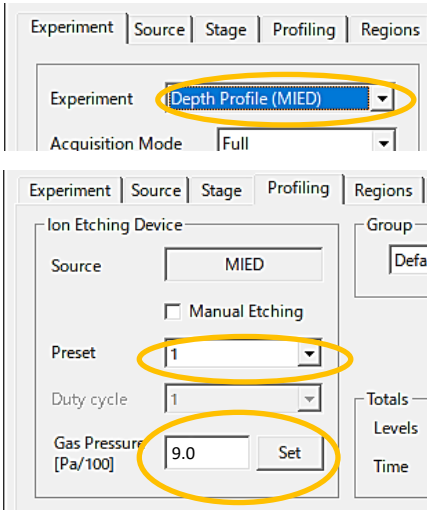
# Setting of Ar<sup>+</sup> etching gun(Appendix)



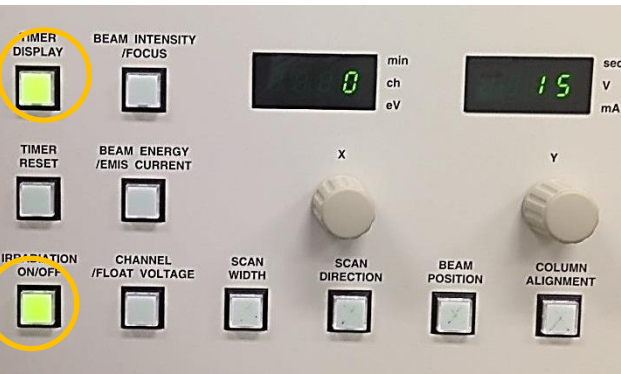
Press the Channel button on the Ion Gun unit and turn the X knob to select the etching condition. The conditions are as shown in the table below.  
Etching rate is measured on SiO<sub>2</sub>.

channel	Ch1	Ch2	Ch3	Ch4	Ch5 For monochrome
Beam energy(eV)	3000	2000	1000	500	3000
Emis current(mA)	20	20	25	25	20
Etching rate(nm/min)	6.0	5.0	2.0	0.5	6.0
previous rate	7.5	4.6	1.6	0.4	6.2

2022/7/29update  
previous rate

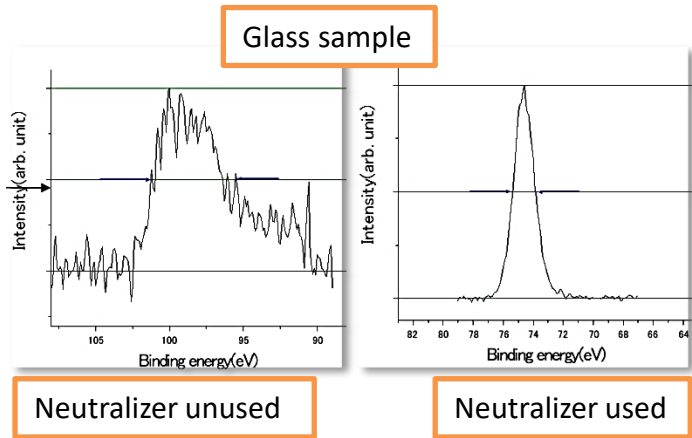


Select “Depth Profile” in Experiment tab, select Profiling tab, enter channel number to be used in Preset, enter “9.0” (6.0 when using ch5) in Pressure, and click Set.  
If you want to do Depth profile, go to “Depth profile” page.



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)

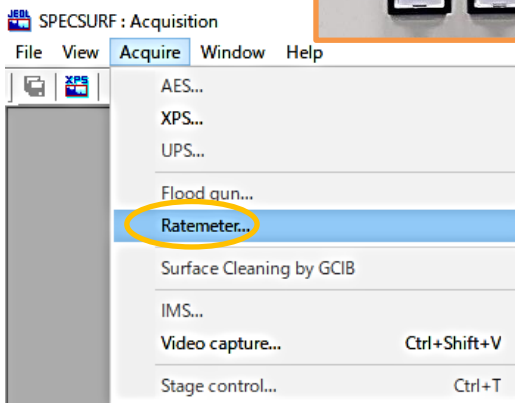


In the case of non-conductive samples, non-uniform charging may occur on the sample surface. The non-uniform charging affects the energy value and the half width. The use of charge neutralizer can alleviate the charging phenomenon by supplying electrons to the sample surface.

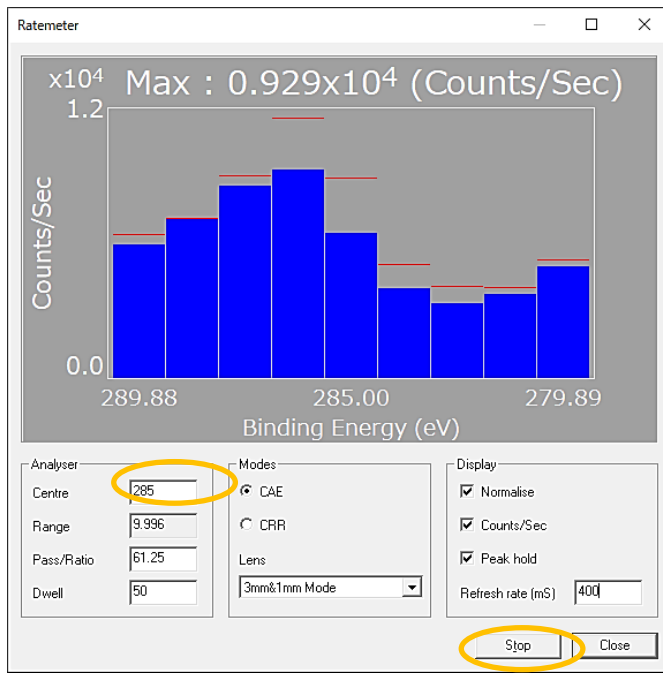


## ▪ How to use charge neutralizer

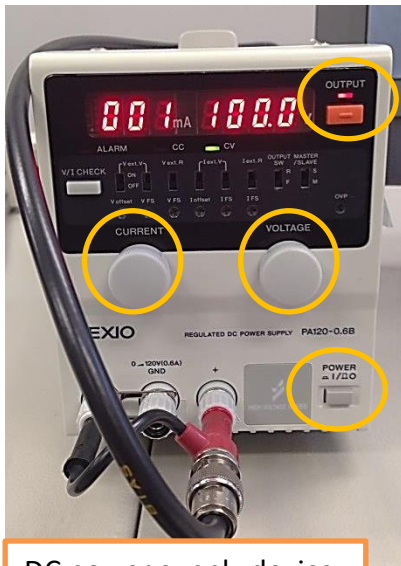
1. Power on charge neutralizer unit
2. Press “SEL” button to change the ADJUST item to “FIL”. and turn the ADJ knob to increase the current value slowly to 6.0A.
3. Select Ratemeter from Acquire in the software menu. If XPS Acquisition is open, close it first.



# Setting of charge neutralizer(Appendix)



- Enter the photoelectron peak position of the sample in "Centre", and change "Pass, Dwell, and Refresh time" to appropriate values to make it easier to see the peak shape. Click "Start" and check Intensity value.
- Turn on "Acc.V" on the unit, change the ADJUST item to Acc.V using the "SEL" button, and turn the ADJ knob to change the voltage value while searching for the voltage value that has the highest peak count and the proper peak position.
- Once the voltage value is determined, close the Ratemeter and measure wide and narrow scan. The appropriate voltage value is different for each sample.



DC power supply device

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

- POWER On, and OUTPUT On.
- Turn the current knob to apply a small amount of current.
- 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



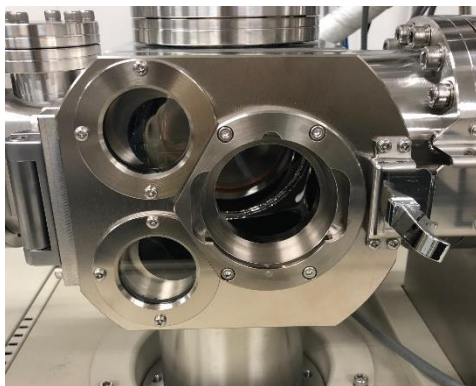
Fix the holder in this orientation.

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

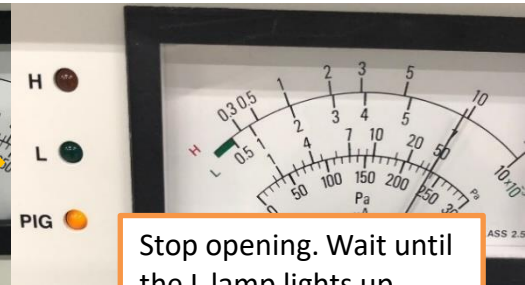
Do not touch the inner part with your bare hands!

# 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  $0.5 \times 10^{-3} \text{Pa}$ , 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.**



Open the vessel slightly.

Stop opening. Wait until the L lamp lights up.

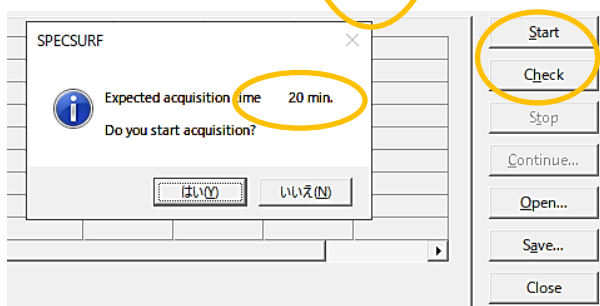
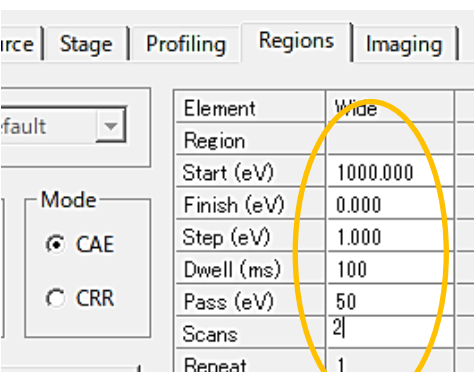
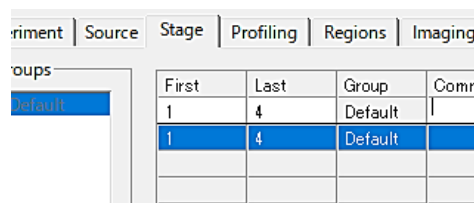
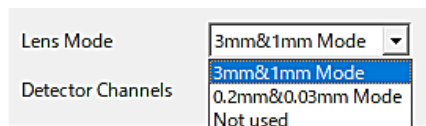
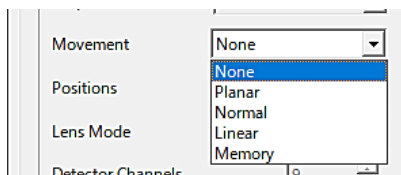
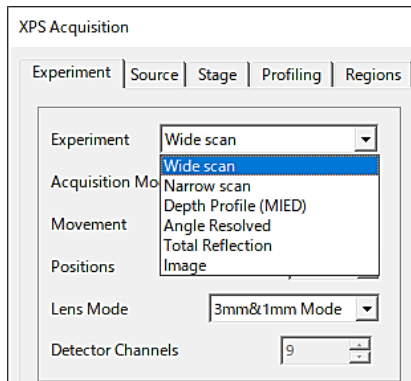


Once the vacuum in the vessel is drawn, fully open the opening/closing knobs. Operate the magnetic ring to receive the holder and close the vessel. When the vacuum in the sample preparation chamber is drawn, open V1 and transfer the holder into the analysis chamber.



Close V1 valve.

# 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.

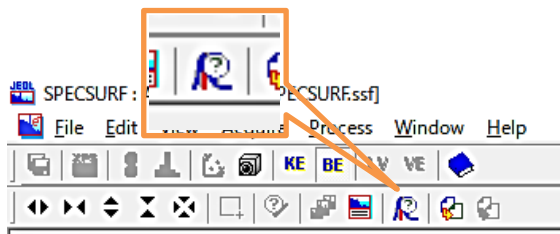
Select the measurement size in Lens Mode.

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.

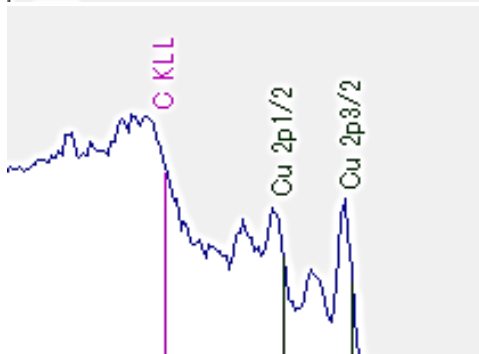
Mainly check the Start(eV) value and the number of scans. If you want to see peaks on the high energy side, increase the start value, and if you want to see small peaks, increase the number of scans.

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

# Markers

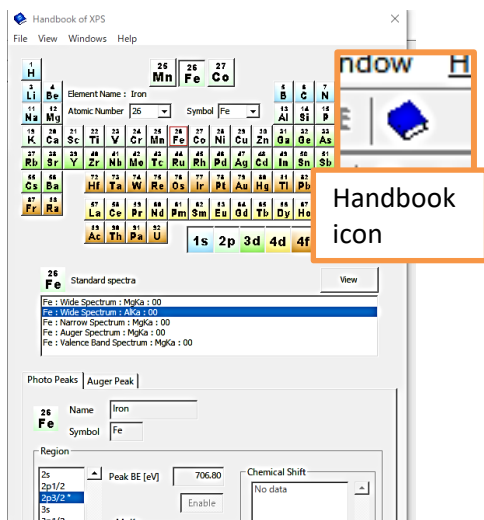
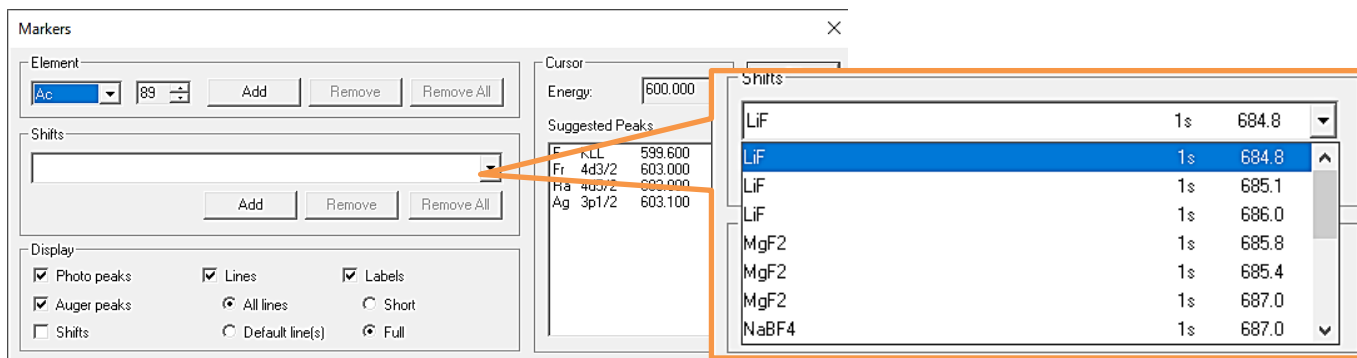


Use markers to identify the peaks that appear in the wide scan spectrum. Click Markers icon on the left.



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.

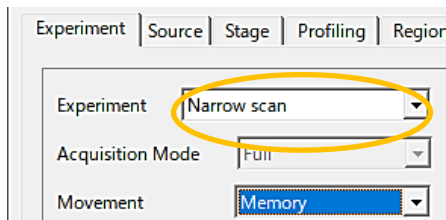


You can see the spectrum of the standard sample in the XPS handbook, so please refer to it.

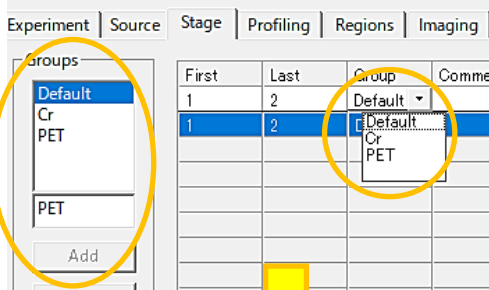
In addition to the photoelectron peak, various satellite peaks appear in the spectrum. In particular, the K- $\alpha_{3,4}$  satellites appear at 10 eV lower energy than the photoelectron peak. And when using Mg-K $\alpha$ , Satellite peaks called "ghost peaks" derived from O-K $\alpha$ (730eV higher),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



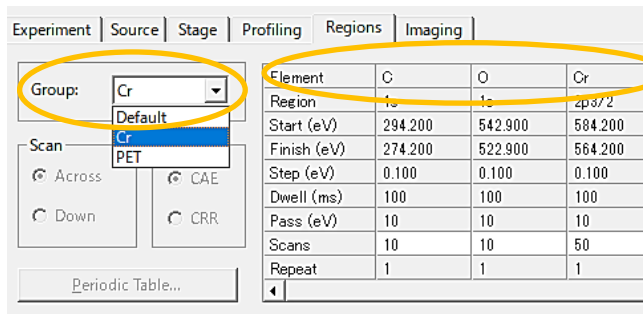
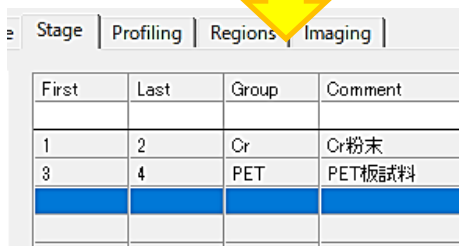
# Narrow scan



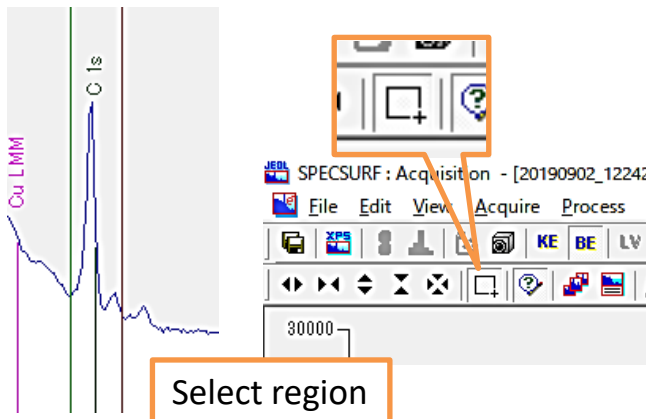
It has a high energy resolution and mainly acquires the spectrum of the main peak of each element. Select Narrow scan in Experiment. For Movement and Lens Mode, refer to "Wide scan".



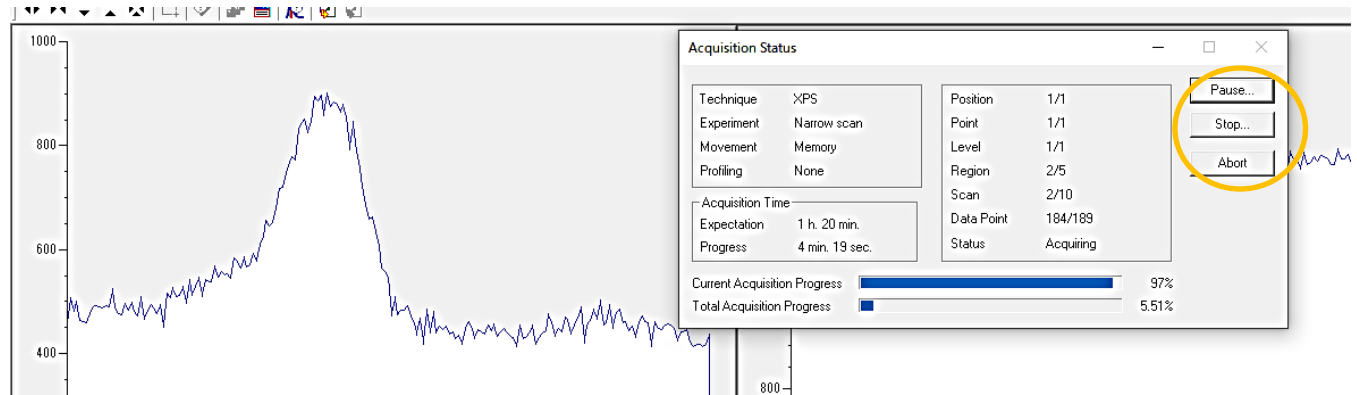
Set the memory number in the Stage tab (when using Memory position, see "Wide scan"). If the measurement contents are different for each sample, you can set the group. Enter a group name in "Groups", click "Add", enter the memory number in First and Last, and select a name from "Group".



Select "Group" in the "Regions" tab (when using the Group function), select the element to be measured from "Element". Open the measured wide scan spectrum and click the "Select region" icon to display Start (green) and Finish (red) lines on the spectrum. Adjust the line position according to the peak to change the measurement range. Set the number of scans to about 10~100. More scans will take more time, but will reduce the noise.



# Narrow scan



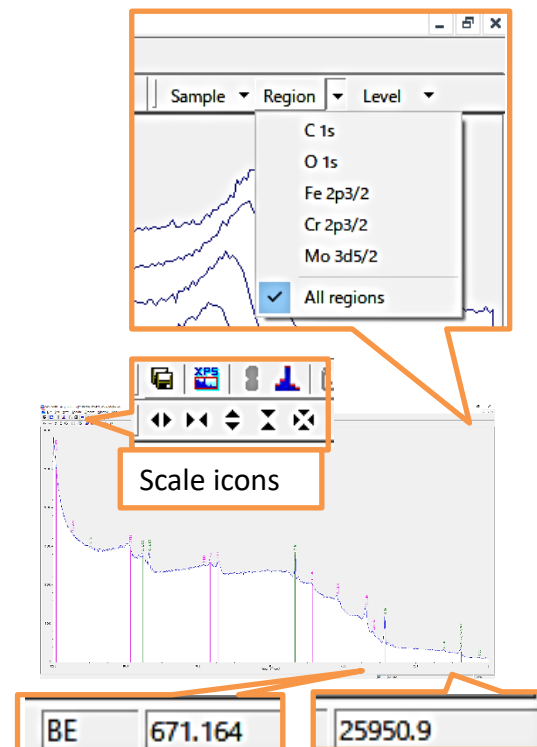
Element	C	O	Cu
Region	1s	1s	2p3/2
Start (eV)	294.200	542.900	971.000
Finish (eV)	274.200	524.000	930.000
Step (eV)	0.100	0.100	0.100
Dwell (ms)	100	100	100
Pass (eV)	10	10	10
Scans	10	10	30
Repeat	1	1	1

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.

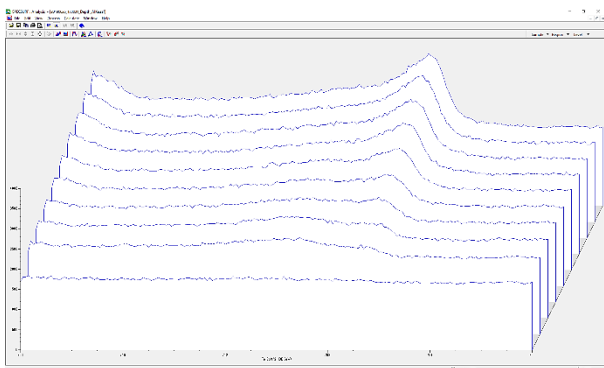
## ▪ About the spectrum window

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.



# Depth Profile



XPS Acquisition

Experiment | Source | Stage | Profiling | Regions

Experiment: Depth Profile (MIED)

Acquisition Mode: Full

Movement: Full

Positions: Separate

Lens Mode: 3mm&1mm Mode

Detector Channels: 9

Profiling | Regions | Imaging

Group: Default

Before	To	Time
1	1	0
2	4	15
5	10	60

Totals

Levels: 10

Time: 405

Set

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.

By repeating the Ar<sup>+</sup> 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<sup>+</sup> 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")

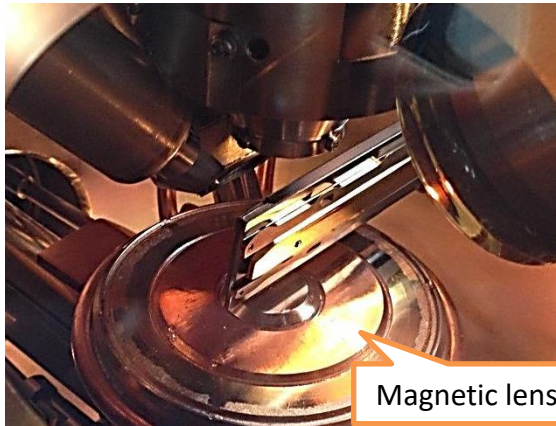
Set the etching time in "Profiling" tab.

"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

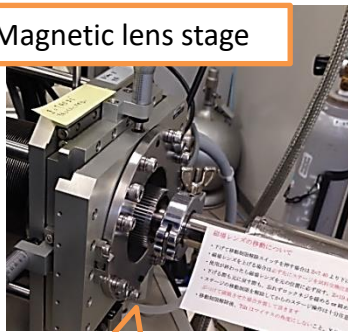


By measuring the spectrum while the stage is gradually tilted toward the analyzer axis, the depth profile of the sample can be obtained nondestructively. Contact the staff when you use it for the first time.

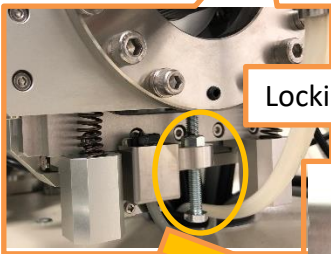
The stage can be tilted up to  $80^\circ$  to obtain a depth profile from 6 nm to 1 nm. A high tilt angle extends the area of the surface to be measured in the Y-axis direction, increasing the amount of signal. It can be applied to the analysis of minute amounts of components.

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. After measurement, be sure to delete the positions registered in the memory function.

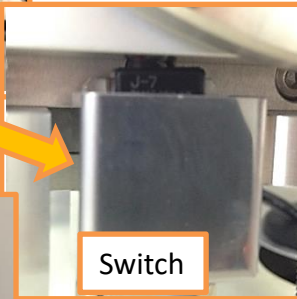
Magnetic lens stage



Locking screw



Switch



Micrometer of the magnetic lens stage

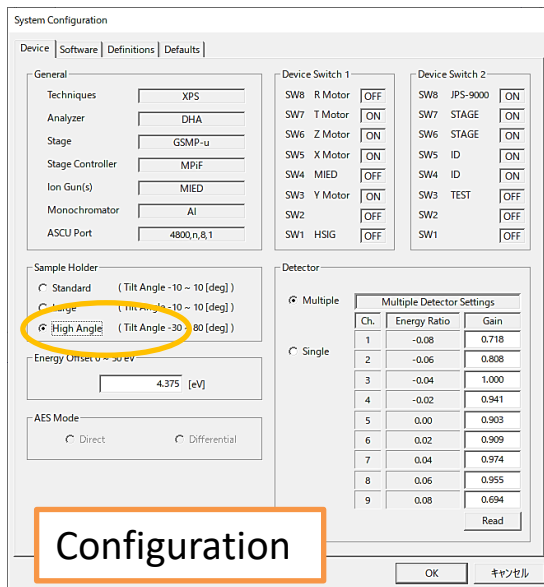


## ▪ Setup of the XPS main unit

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  $Z=18.80$  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.

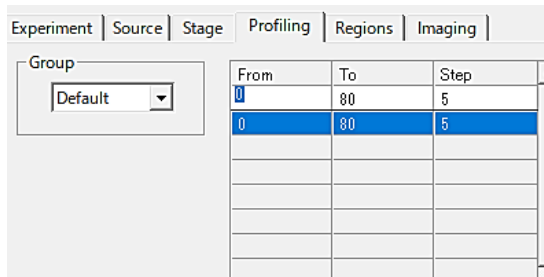
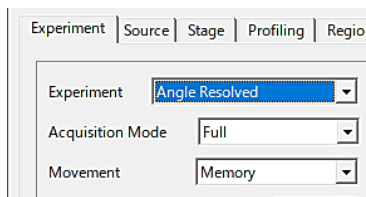


# Angle Resolved



## • 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.

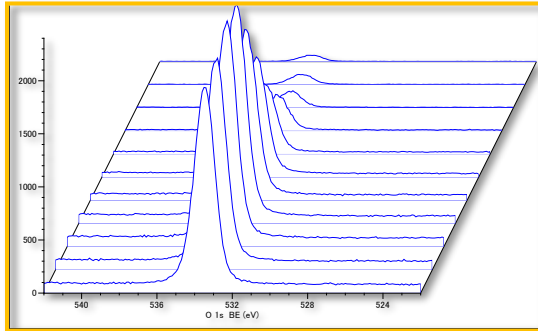


Select "Angle Resolved" in the Experiment tab, and set the tilt angle of the stage in the Profiling tab. In the left picture, XPS acquires spectra at each level while tilting from 0° to 80° in 5° steps. If you want to tilt the stage more than -10° , be sure to move the position of the X-ray source back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

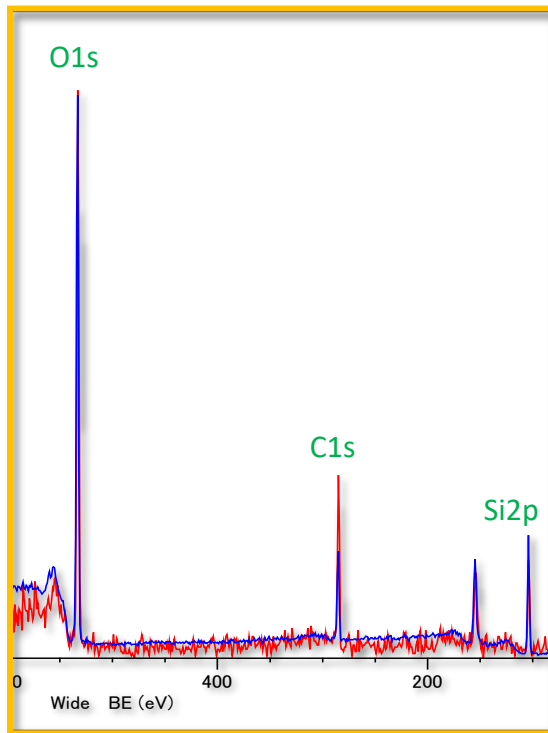


← If the holder is tilted in the minus direction, there is a risk of it hitting the X-ray source. Basically, it is prohibited. Please consult with staff.

# 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  $0.2^\circ$  from  $-10^\circ$  to  $-5^\circ$ , 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  $1^\circ$  from  $-4^\circ$  to  $-1^\circ$ , 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,  $-1^\circ$ .

# Image & Linescan

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.

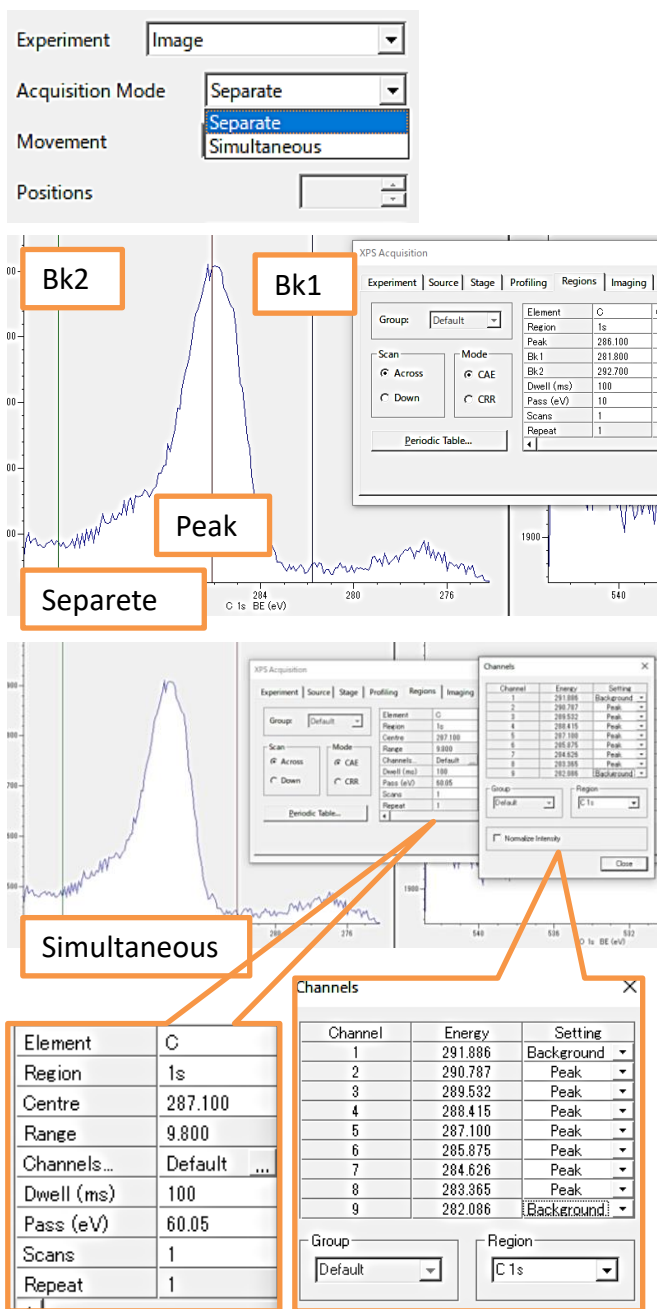
First, obtain a Narrow scan of the element you want to measure. Select "Image" in "Experiment", and select "Separate (PB separate method, easy to set up)" or "Simultaneous (PB simultaneous method, fast measurement)" in "Acquisition Mode".

- 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.



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

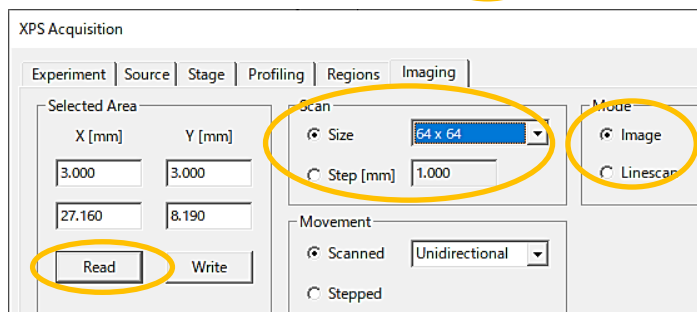
# Image & Linescan

No.1

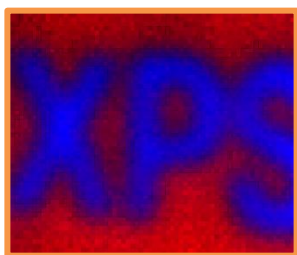
Mapping area

No.2

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.

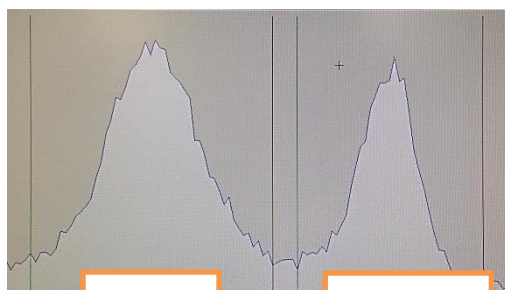


In the Image tab, click "Read" to read the memory positions." In Scan, select either Size or Step and set the resolution of the image. For "Mode", select "Image".

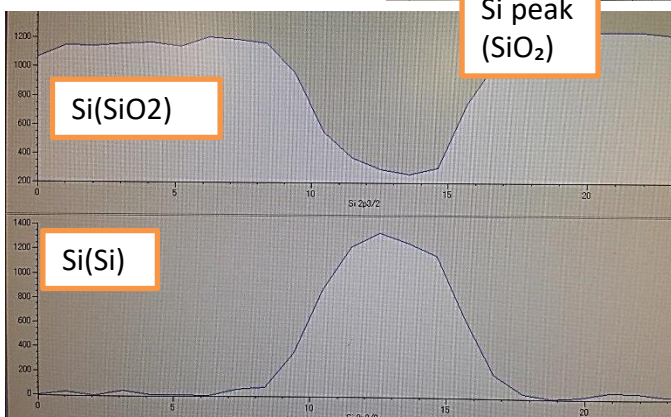


← Mapping image obtained by "Image". red:Si and blue:Au. The image of each element in the mapping result can be layered in different colors.

Element	Si	Si
Region	2p3/2	2p3/2
Centre	104.049	99.200
Range	4.898	3.803
Channels...	Default	Default
Dwell (ms)	200	200
Pass (eV)	30.65	23.8
Scans	1	1
Repeat	1	1



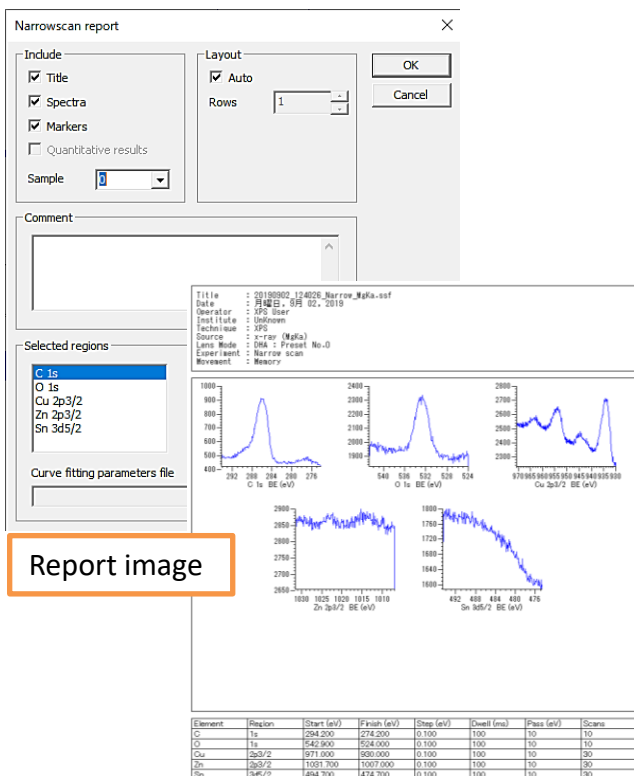
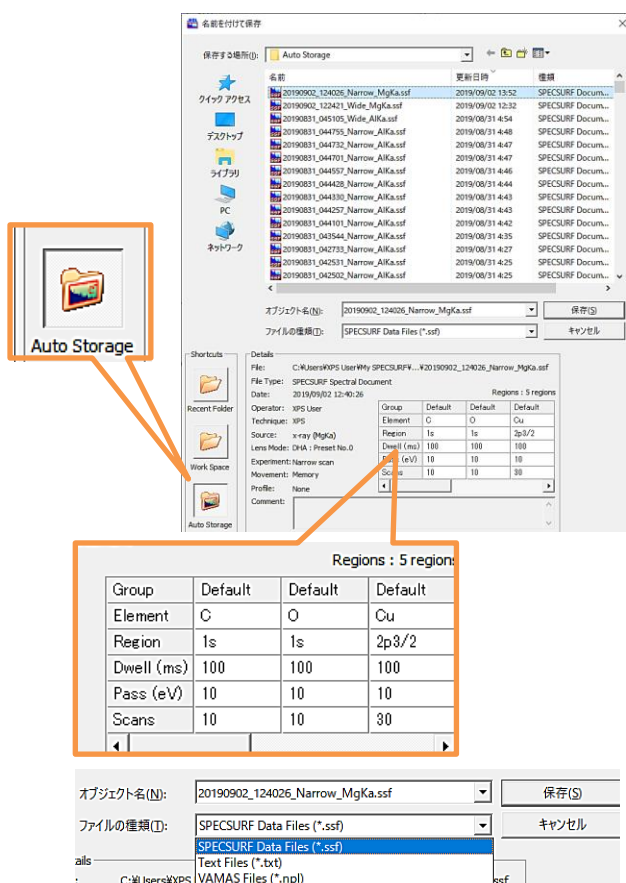
Select "Linescan" for "Mode" to perform line profile from memory No.1 to No.2 position.



← Linescan results by Simultaneous method. This is a Si wafer etched with Ar<sup>+</sup> in the center.



# Saving data



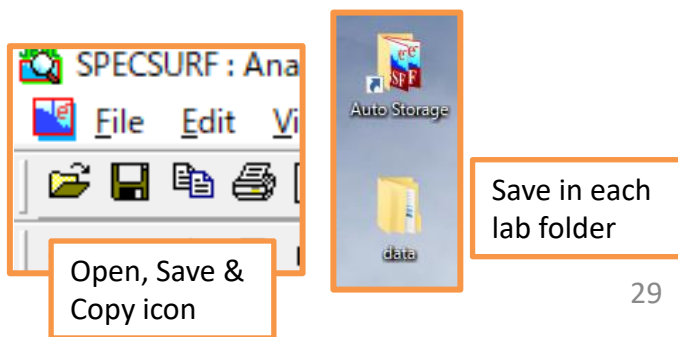
All measured spectrum data are automatically saved with the name of the date, measurement method, and X-ray source in "Auto Storage" folder. Select the Auto Storage folder from "File" -> "Open", select the data, the details of the data will be displayed at the bottom of the save window.

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.

You can output the spectrum as an image in report format by clicking "File" -> "Report". You can also paste the spectrum as an image by clicking "copy" on the spectrum window.

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<sup>+</sup> 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<sup>+</sup> etching gun chamber vacuum gauge OFF.

## ▪ 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=18.80 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.