Cold FE-SEM
User Manual(Eng.)

UNIST Central Research Facilities
UNIST Materials Characterization Lab

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Index

Lab Safety

Compliance with lab safety rules

If you do not follow the lab safety rules, access and use of the lab will be restricted, so we ask for your cooperation.

No Food or Drink

Wear Protective Clothes

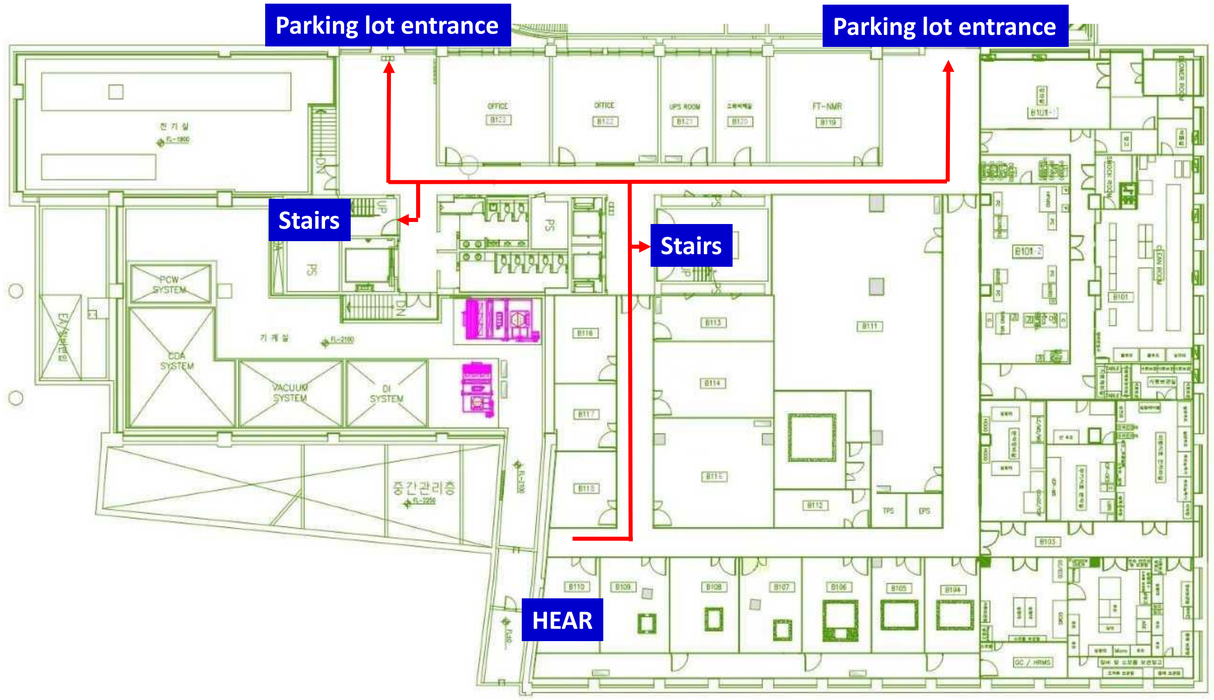
Wear Foot Protection
No Open-Toe Shoes

If you didn't bring your lab coat, please wear a shared lab coat entrance side of the room 101-2 on the first basement floor of 102 building.

Make sure to wear a lab coat and enter the lab of UCRF.

Make sure to keep the lab clothes you wore in their original place.

Lab emergency evacuation



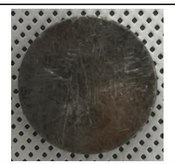
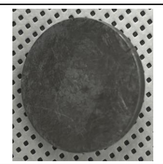
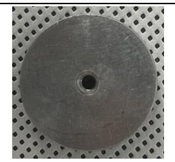
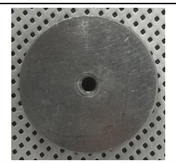
UCRF SEM Feature

	High resolution imaging	EDS analysis	Specimen damage & charge-up	BSE detector	E-SEM	Reservation status	Introduction year	Fee(100%)
Verios FE-SEM	◎	◎	Slight	O	X	Not busy	2024	Undecided
SU8220 cold FE-SEM	◎	O	Slight	X	X	Busy	2013	40,710won /30min
SU7000 FE-SEM	O	O	Serious	X	X	Busy	2021	40,710won /30min
Cold FE-SEM	O	O	Slight	X	X	Not busy	2011	31,320won /30min
Quanta200 FE-SEM	X	△	Slight	O	O	Not busy	2009	26,100won /30min

What to prepare for SEM imaging

1. Fully dried samples

2. A stub suitable for the equipment and measurement purpose

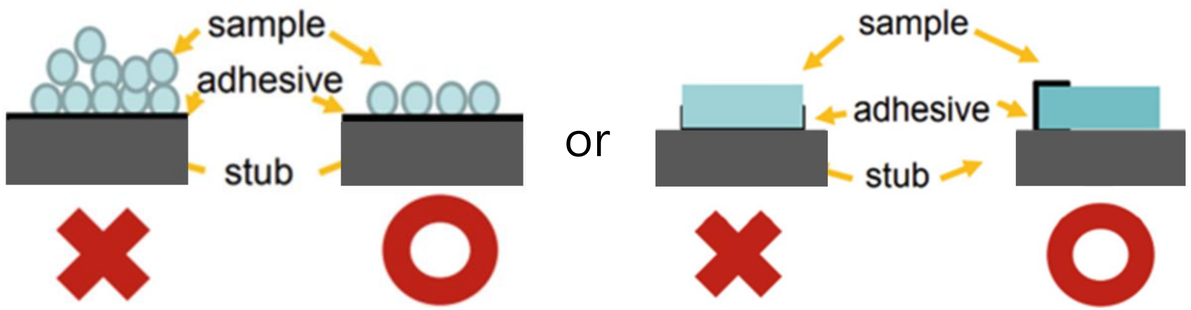
Type	A	B	C	D
Equipment	SU8220 Cold FE-SEM SU7000 FE-SEM Cold FE-SEM		Nano FE-SEM Quanta FE-SEM	
Usage	General	Cross section	General	Cross section
Picture (Front)				
Picture (Back)				

3. Conductive double-sided carbon tape for SEM 

- If you need a special stub other than the stub beside, please discuss with the SEM manager and UDMC.

☎ SEM stub Production
- UDMC Cha Jae-hoon, Jeong Woo-hyun

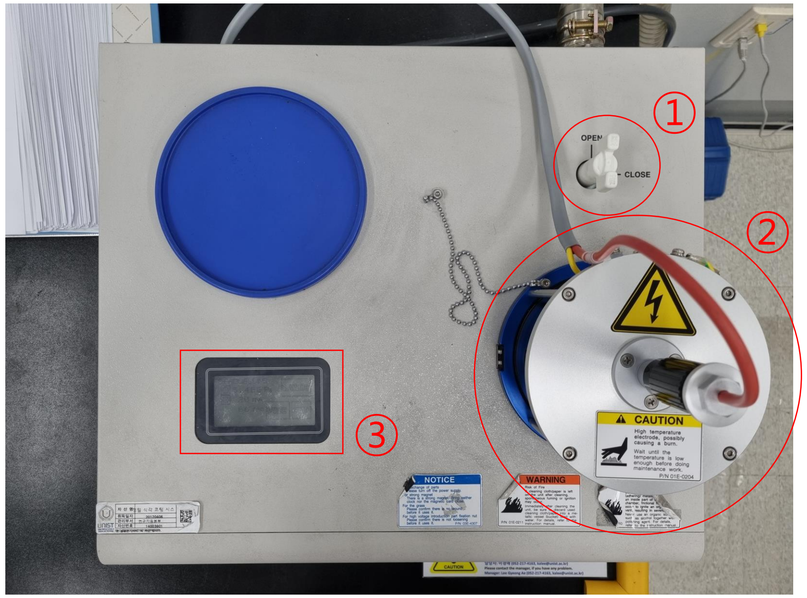
Specimen Preparation



1. Sample preparation should be performed by individual laboratories.★
2. Wear appropriate safety gear.(goggles, gloves, etc.)★
3. Prepare completely dried specimens.
4. Clean the stub with ethanol, etc.
5. Attach conductive double-sided tape to the stub and fix a very small amount of specimen to the tape.(For powder samples, be sure to use carbon tape)★
6. Blow to remove dust from the specimen.★
7. Coat it if necessary.

Coating

- **Hitachi Sputter**



- Coating rate

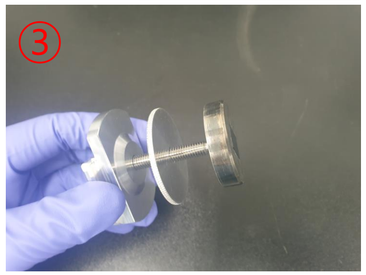
  - Pt target: 15 nm/min

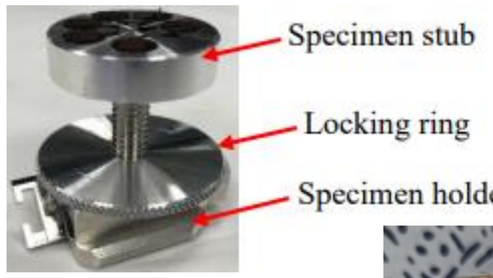
<Condition>

- Sputter current: 40mA
- Distance between target and sample: 20 mm

1. Reserve and use the equipment.(Zero tolerance)
2. Turn the valve open.
3. Open the column part and place the specimen in the center.
3. Make sure the specimen touches the Pt source.
4. Press the touchscreen to view the coating parameters.
5. If you want to change a parameter, press [Change].
6. Tap each parameter and enter the desired value.
7. Press [Enter] - [Back] - [Start].
8. When Processing finished appears on the screen, open the column and remove the specimen.
9. Press [Restart], then press [Stop] after 20 seconds.
10. Turn the valve to [CLOSE].

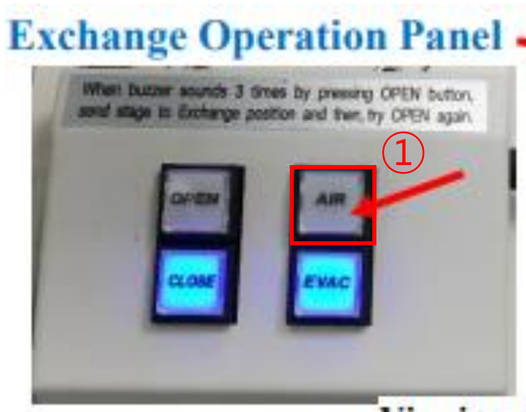
Specimen Preparation

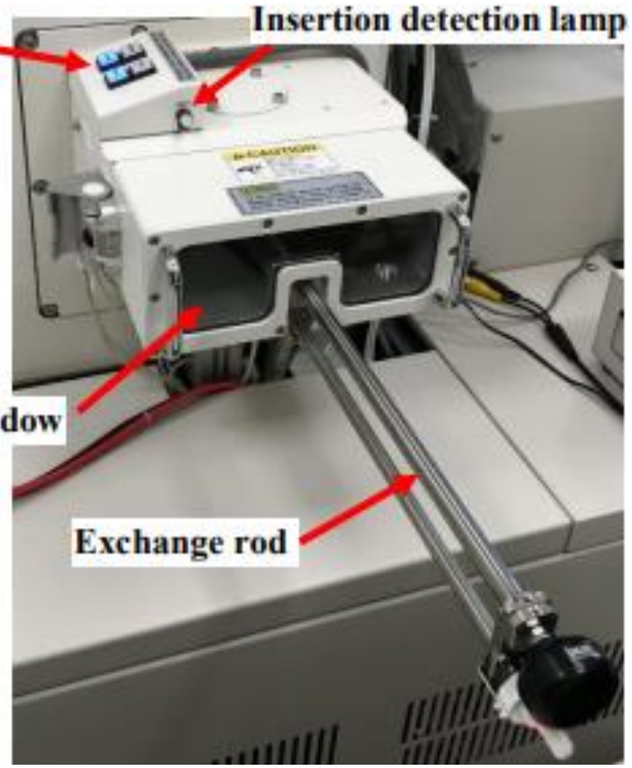


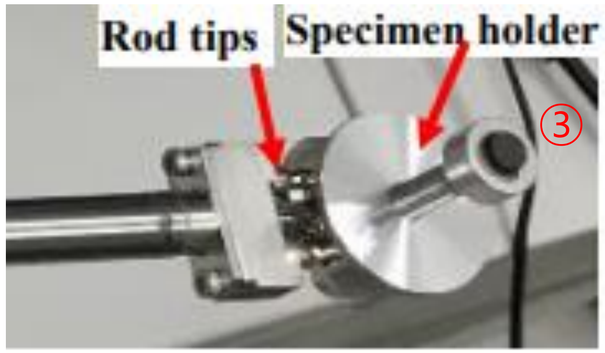


1. Prepare 4 items needed to assemble the specimen holder.(If the screw does not come off from the holder, wrap tissue paper around the screw and loosen it with a long nose.)
2. Assemble the holder in the order shown in the photo below.
3. Check if the top and bottom direction of the specimen holder is correct.★
4. Check if the screw protrudes from the bottom.★
5. Adjust the specimen height so that the highest part of the specimen touches the height checker.★

Loading the Holder







1. Press [AIR], wait until the buzzer sounds.
2. Open the exchange chamber door.

   (Do not hold the exchange rod to open the door.)
3. Insert the specimen stage onto exchange rod
4. Turn the knob counterclockwise to lock.(LOCK←)★

Loading the Holder

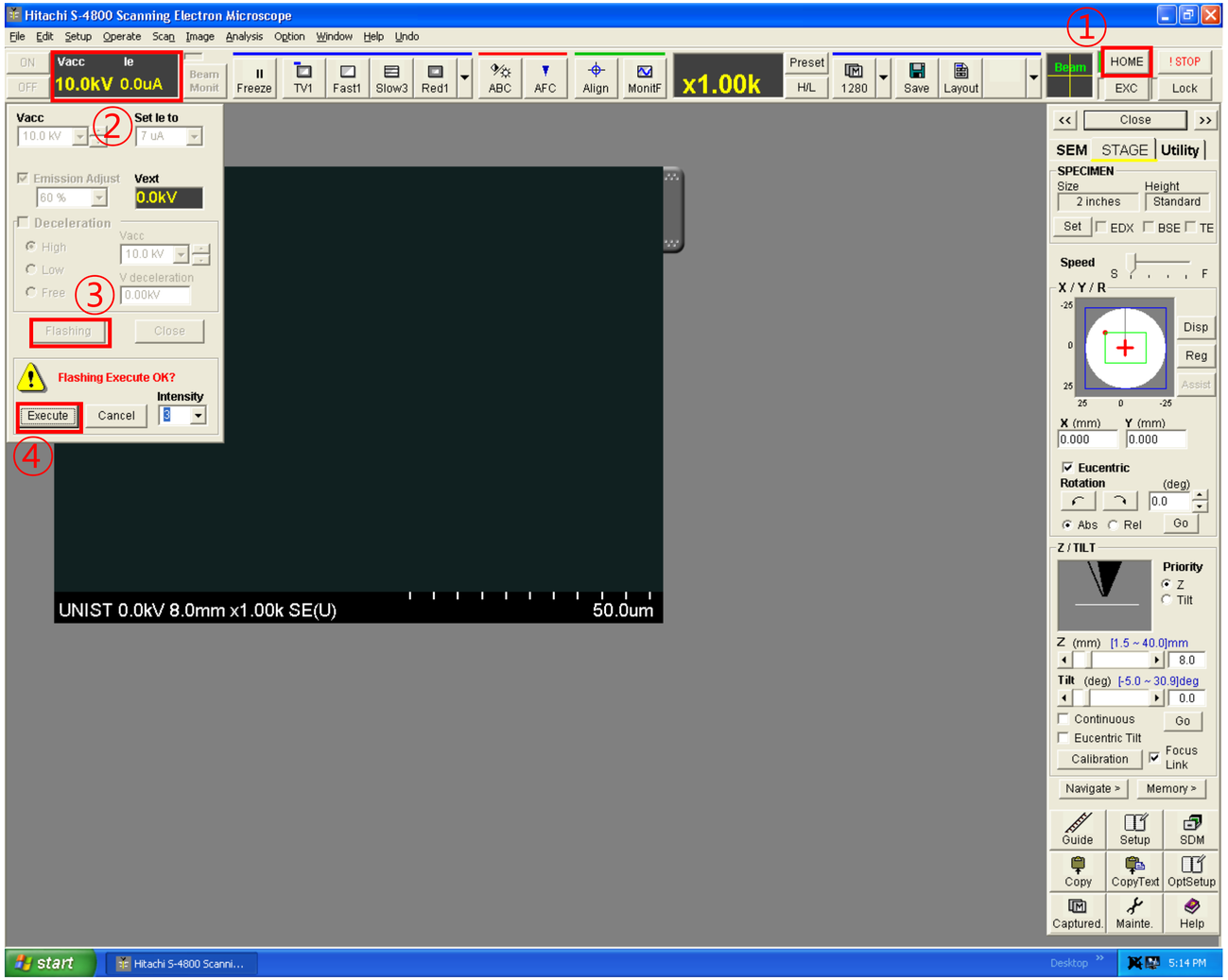
5. Press to close the specimen chamber door.(Do not hold the exchange rod.)
6. Press [EVAC], press the chamber door.★
7. Wait until buzzer sounds.
8. Press [OPEN], wait until buzzer sounds.
9. Push the rod into the chamber.

   (Do not forcibly insert the exchange rod.★)

Loading the Holder

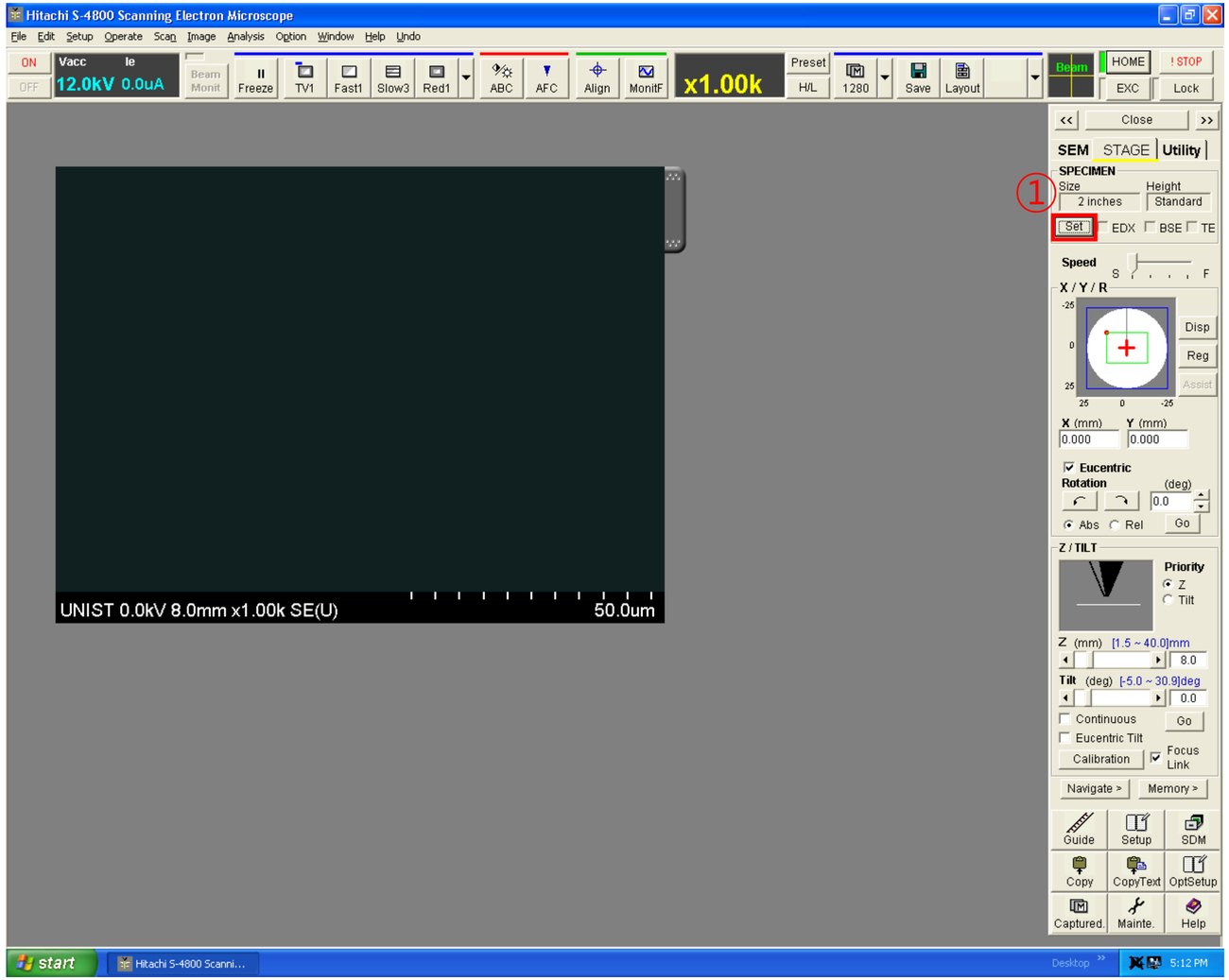
10. Turn the specimen holder lock/unlock knob to UNLOCK position.
11. Pull out the rod all.★
12. Press [CLOSE], wait until the buzzer sounds.

Starting Instrument



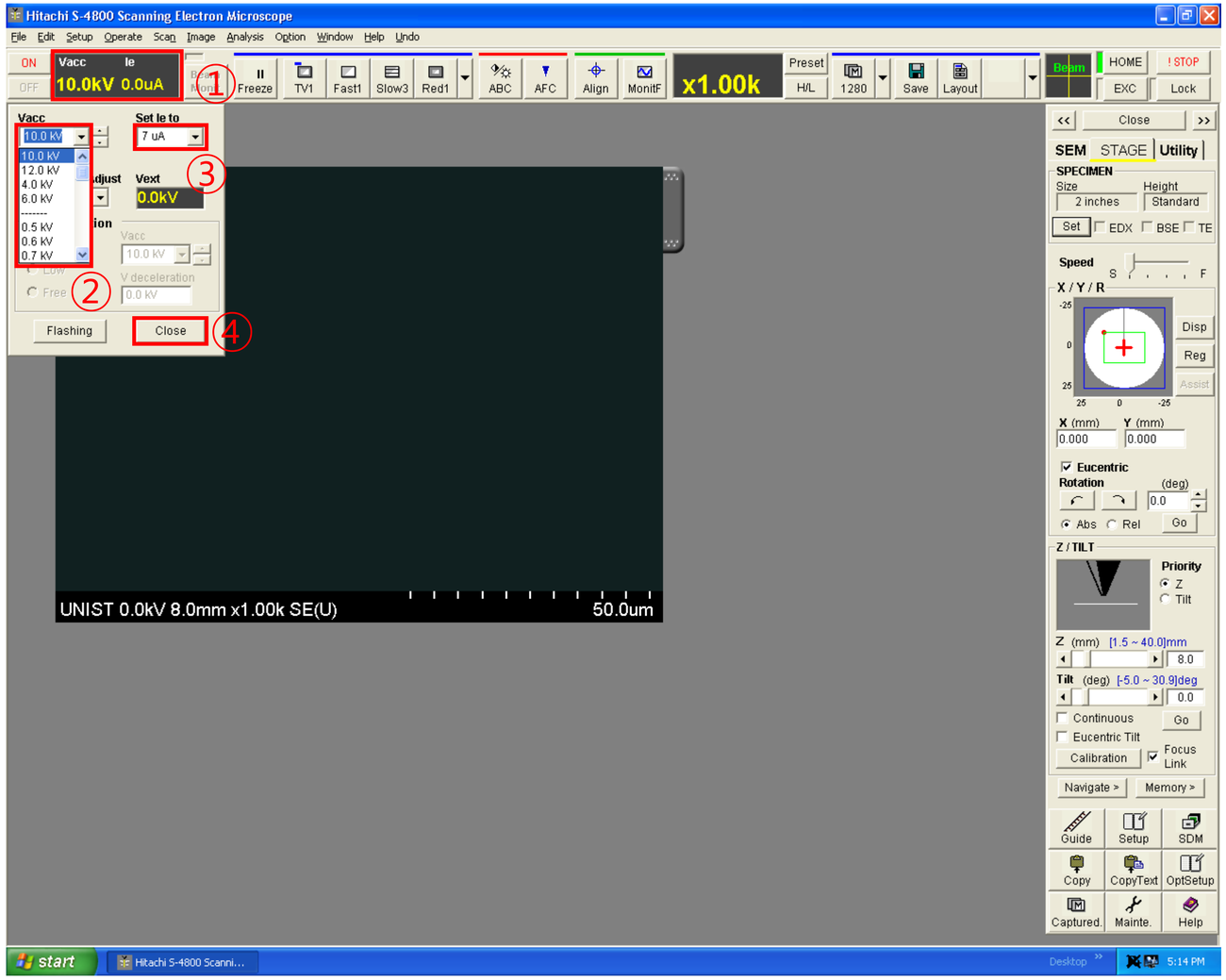
1. Click [PC_SEM].
2. Enter your lab ID and PW.
3. Click [HOME].
4. If a flashing message “Please Flash!!” appears, click the electron beam window.
5. Click [Flashing].
6. Click [Execute].

Size & Height of Stub



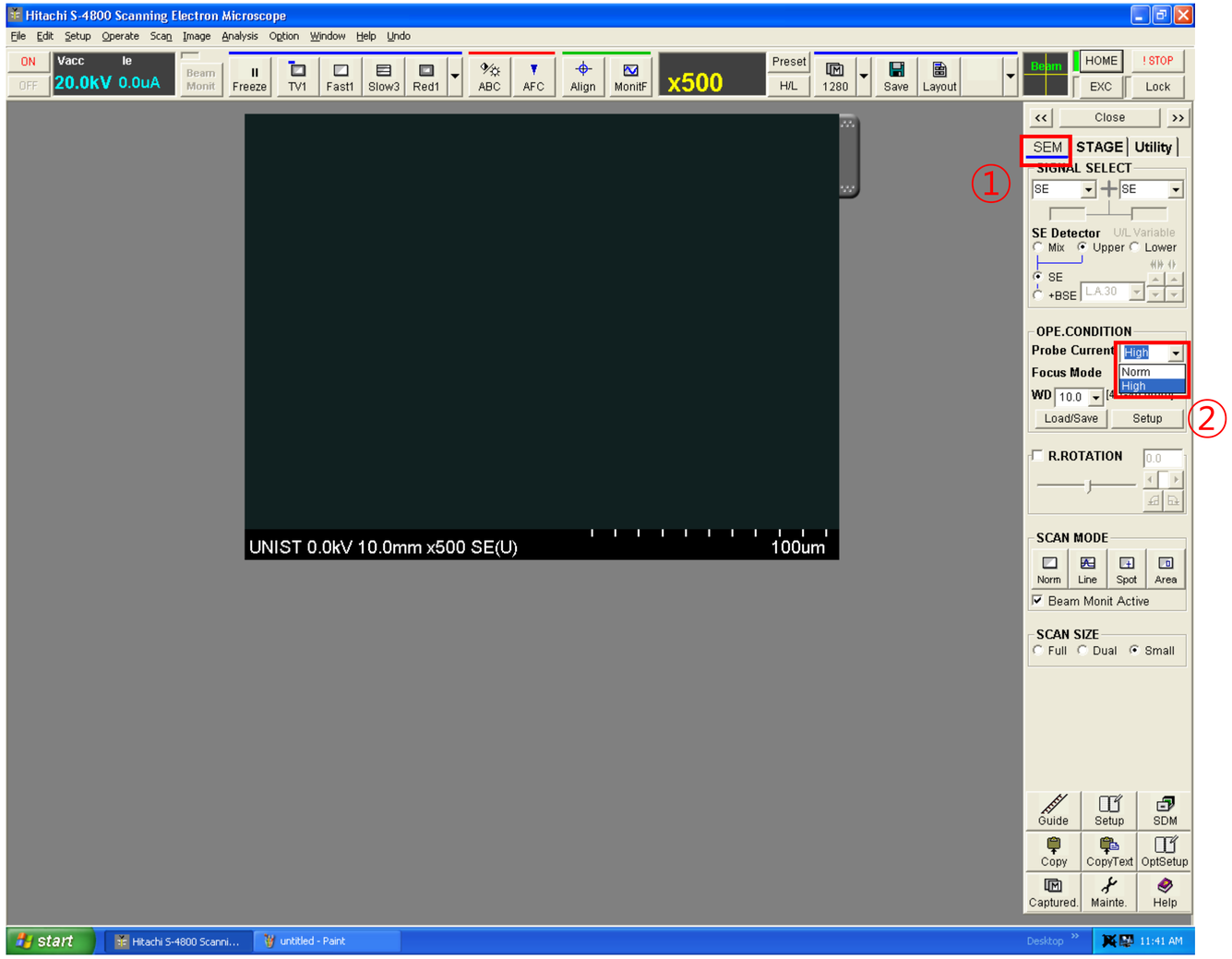
1. Click [Set].
2. For [Size] setting, select the diameter of the stub one size larger.★
3. For Height setting, select Standard.
4. Click [OK].

Acceleration Voltage



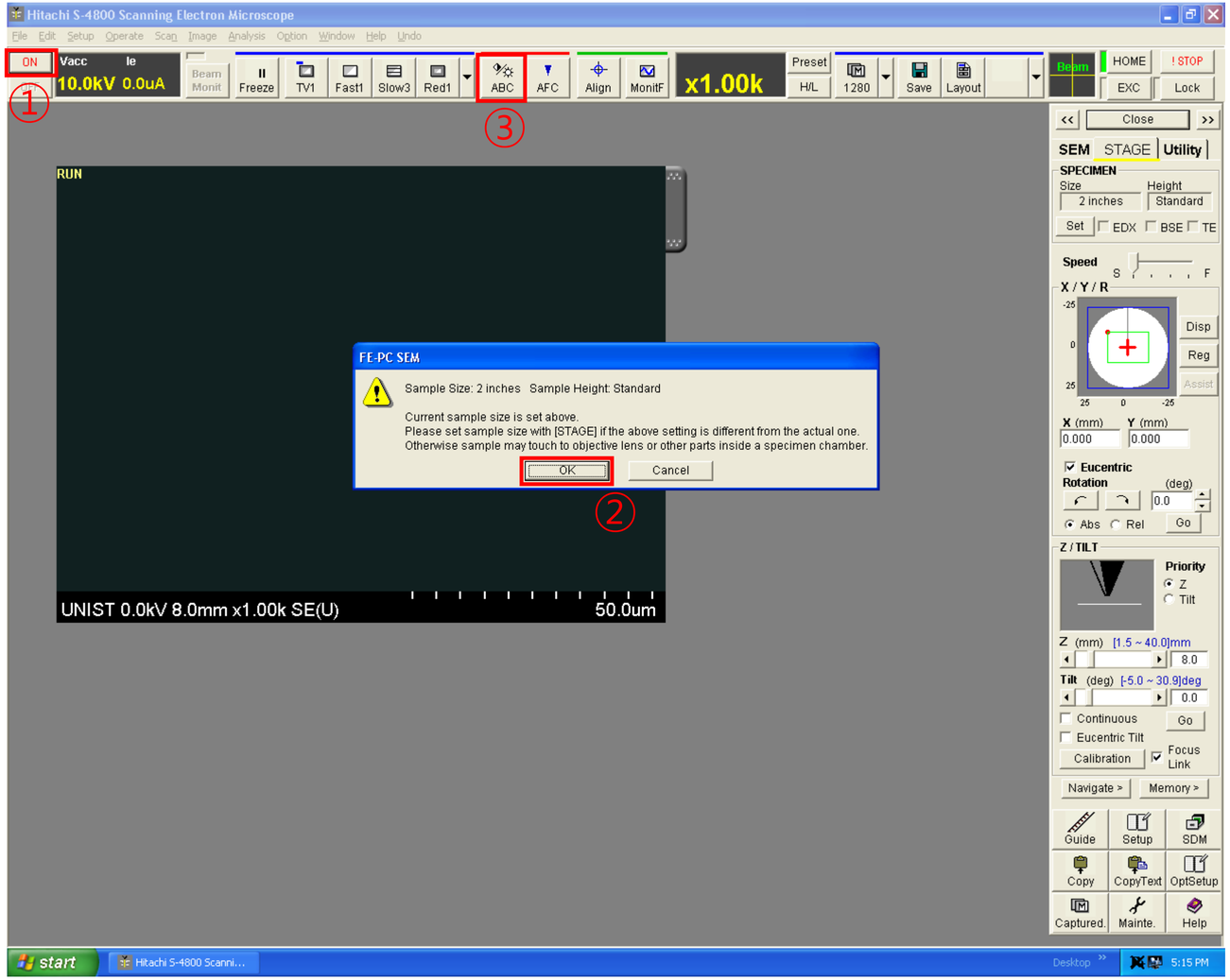
1. Click the [Electron Beam].
2. Select the acceleration voltage in [Vacc].
3. Select beam current (7~10) in [Set Ie to].
4. Click [Close].

Probe Current



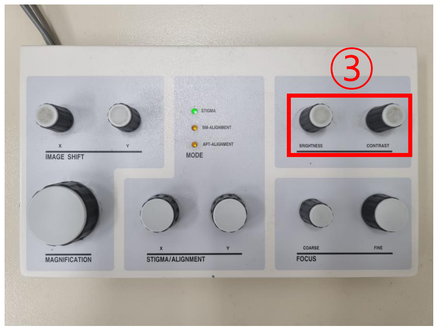
1. Click the [SEM].
2. Select [Probe Current] appropriately.

Beam On

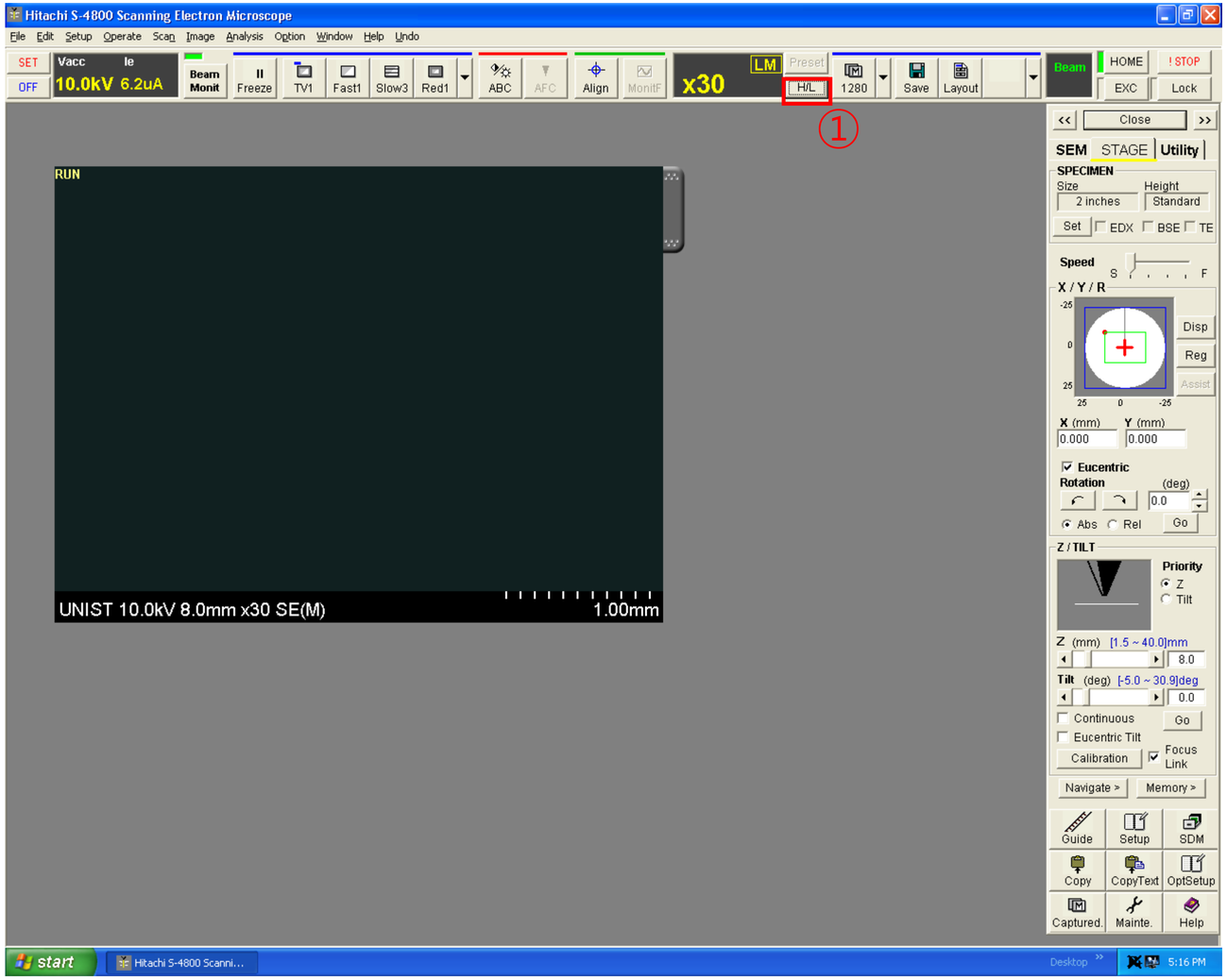


1. Click [ON].
2. Click [OK].
3. If the screen is too dark or too bright, press [ABC].(You can manually adjust the [Brightness] or [Contrast] knob individually.)

   → This process can be performed at any time during image observation.

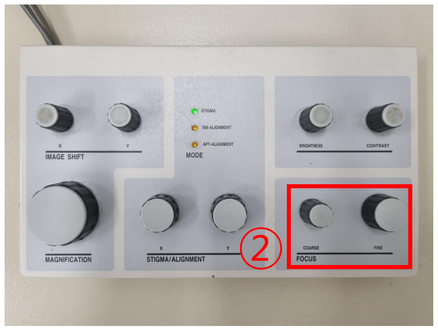


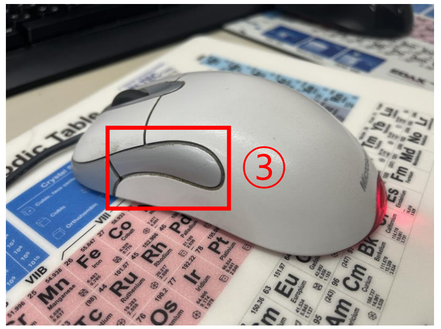
Finding the Specimen



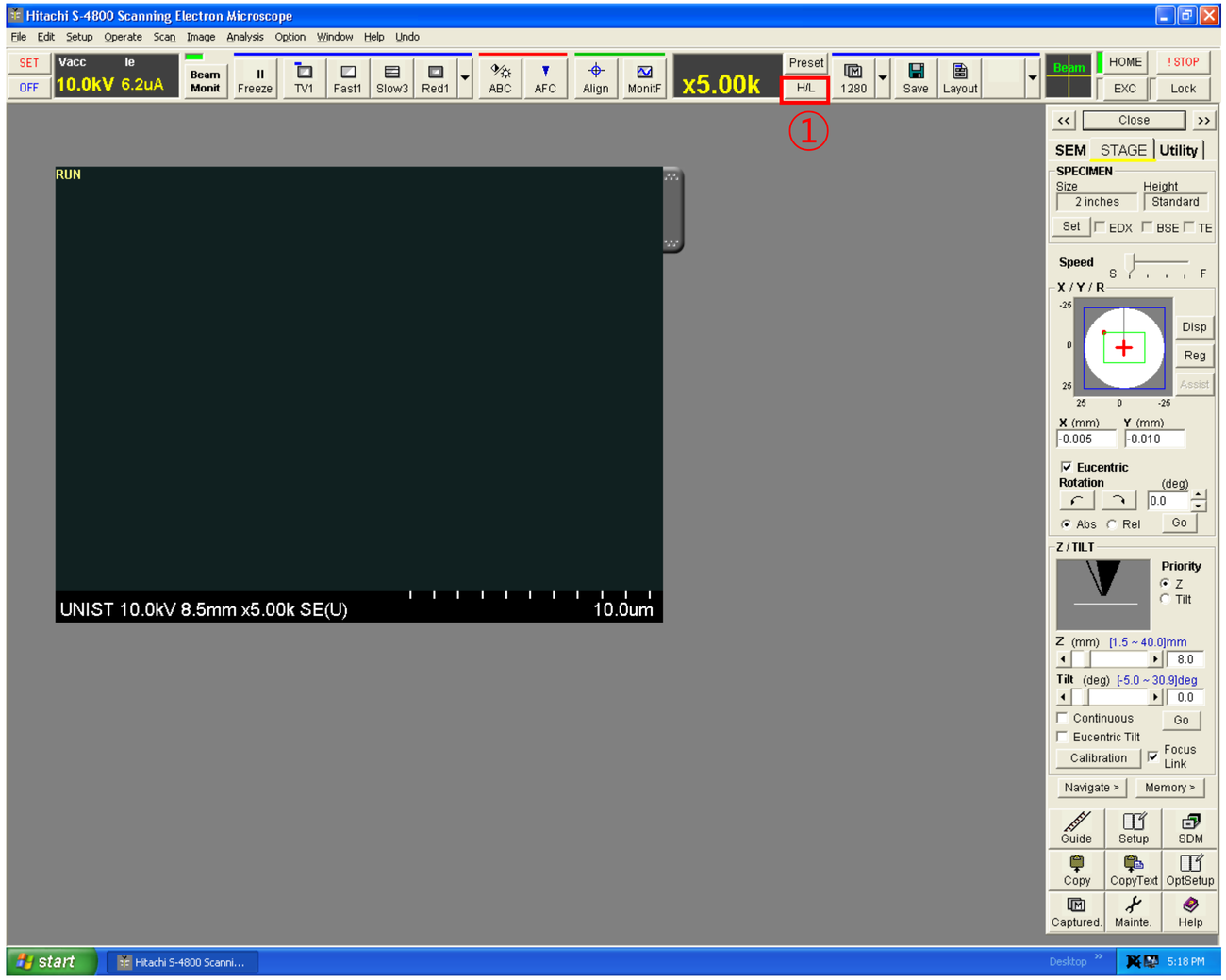
1. Press the [TV1].

   (SCAN SPEED: TV1>Fast1>Slow3)
2. Click [H/L].
3. If the image is blurry, use the [FOCUS] knob to adjust the focus.
4. Press the left silver mouse button four times.
5. Double click or drag to the desired area.

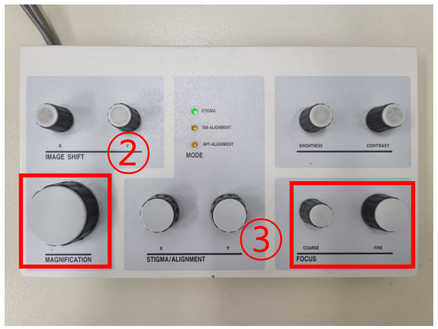




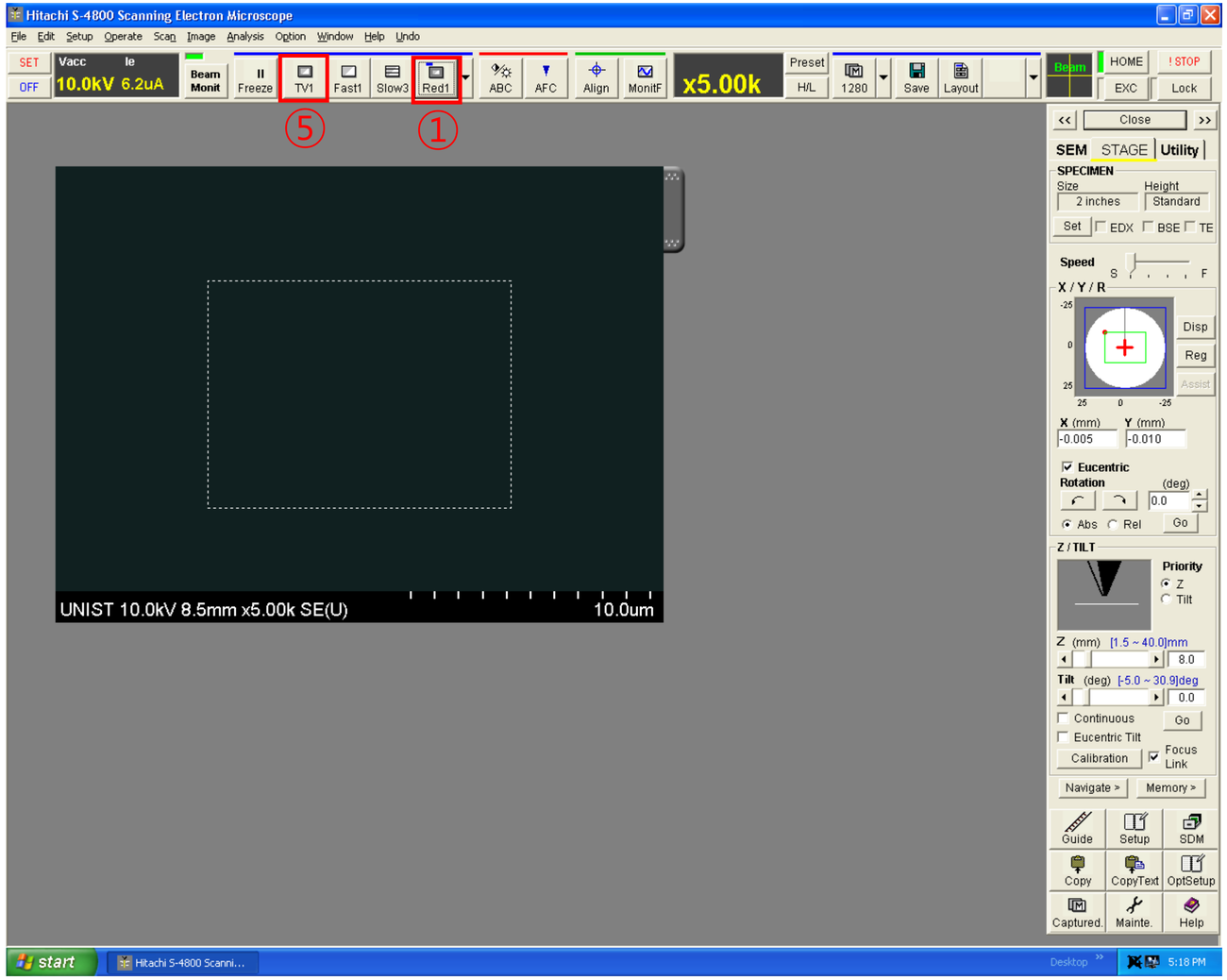
Focus



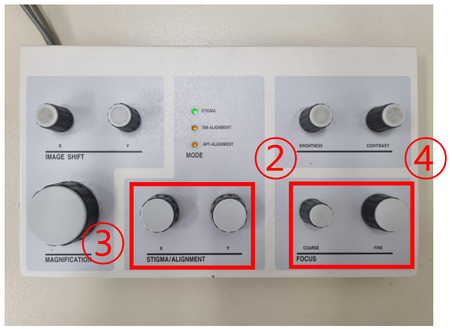
1. Click [H/L].
2. Move the stage to the location you want to observe.
3. Use the magnification knob to slowly raise the magnification to 5000 times and adjust the focus in between.
   - COARSE: fast adjustment
   - FINE: fine adjustment



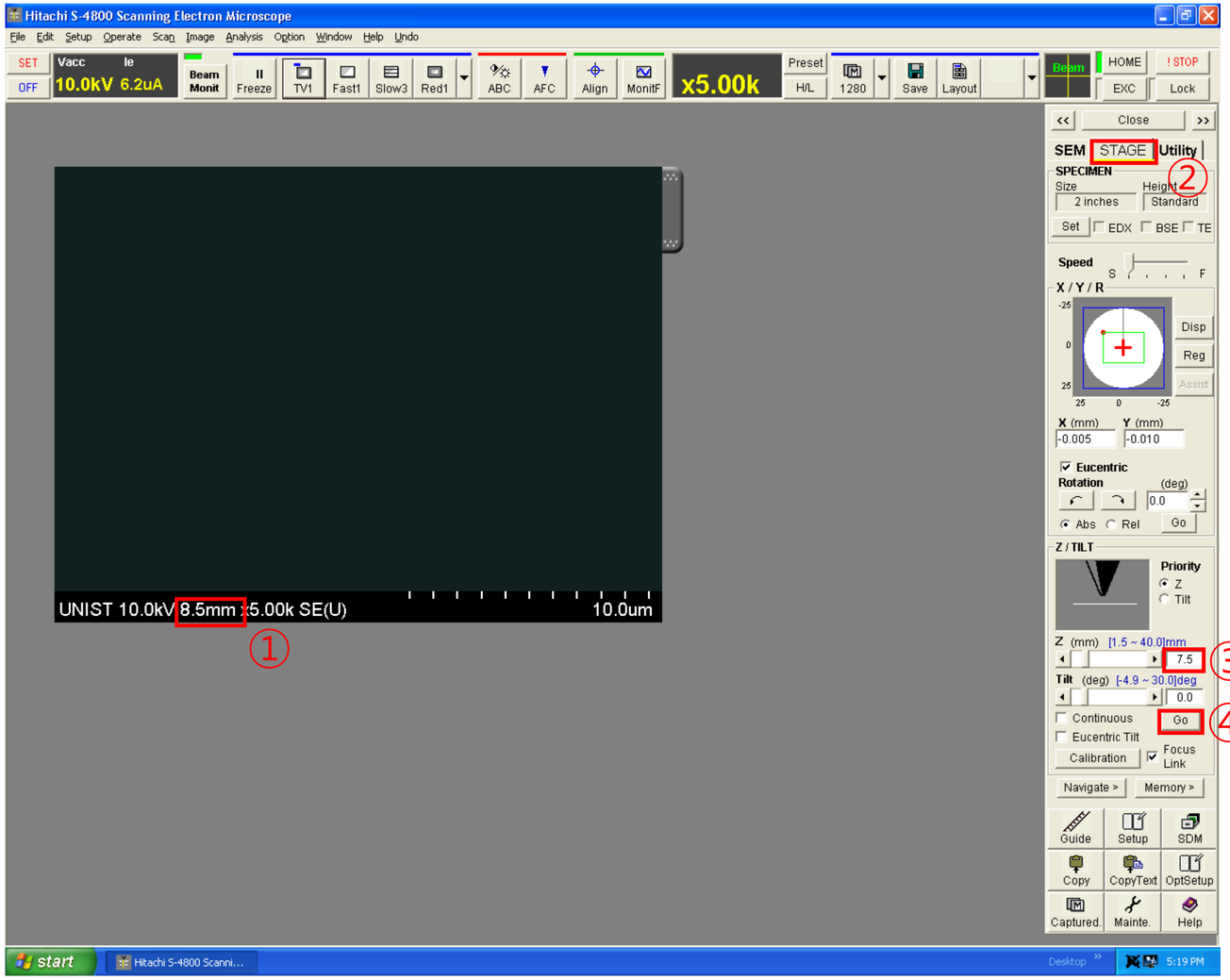
Stigmator

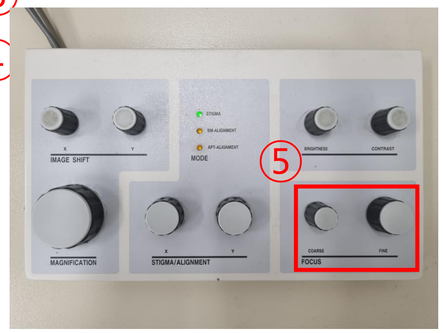


1. Press [Red1].
2. Adjust the focus.
3. Adjust the Stigma X and Y knobs one by one.
4. Adjust the focus.
5. Press [TV1].



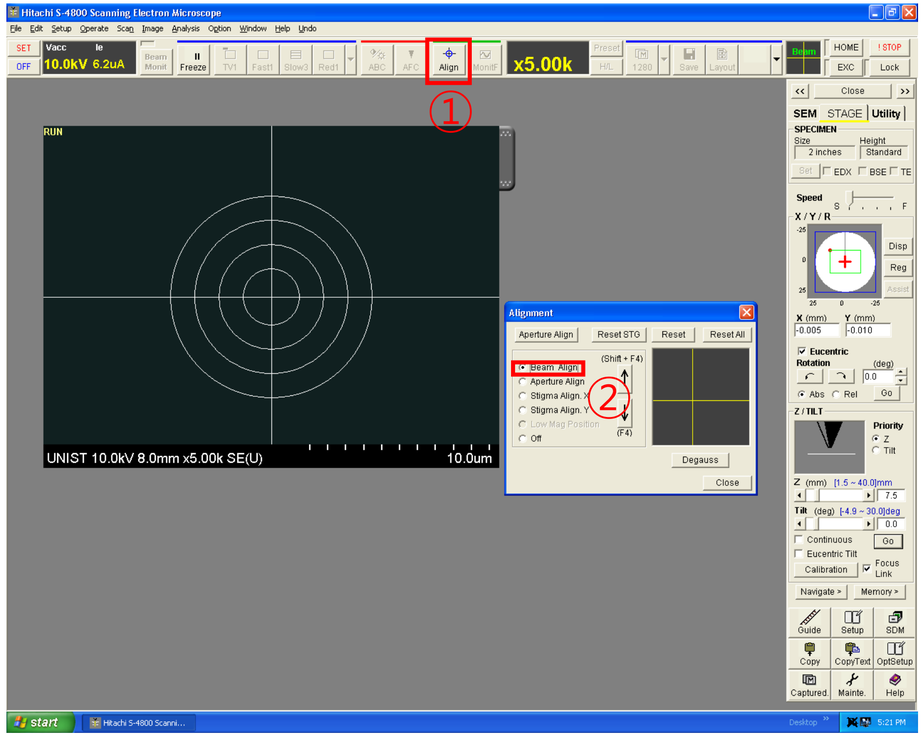
Working distance

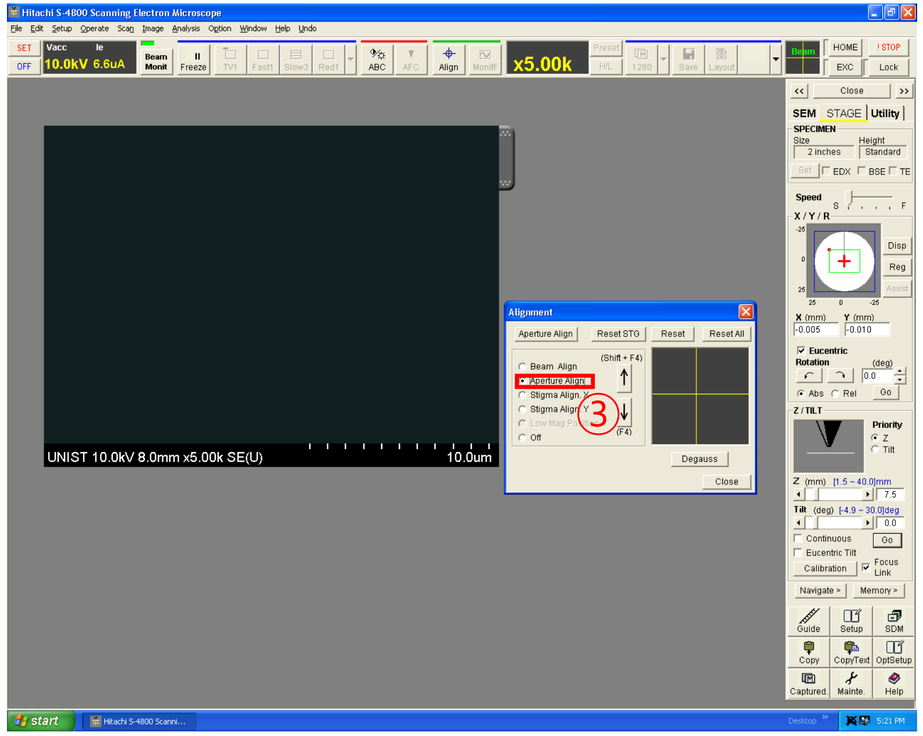




1. Check WD at the bottom of the screen.
2. Subtract the WD found in 8, and subtract that value from the [Z] value.
3. Enter the calculated value in [Z].
4. Click [Go].
5. Adjust the focus.

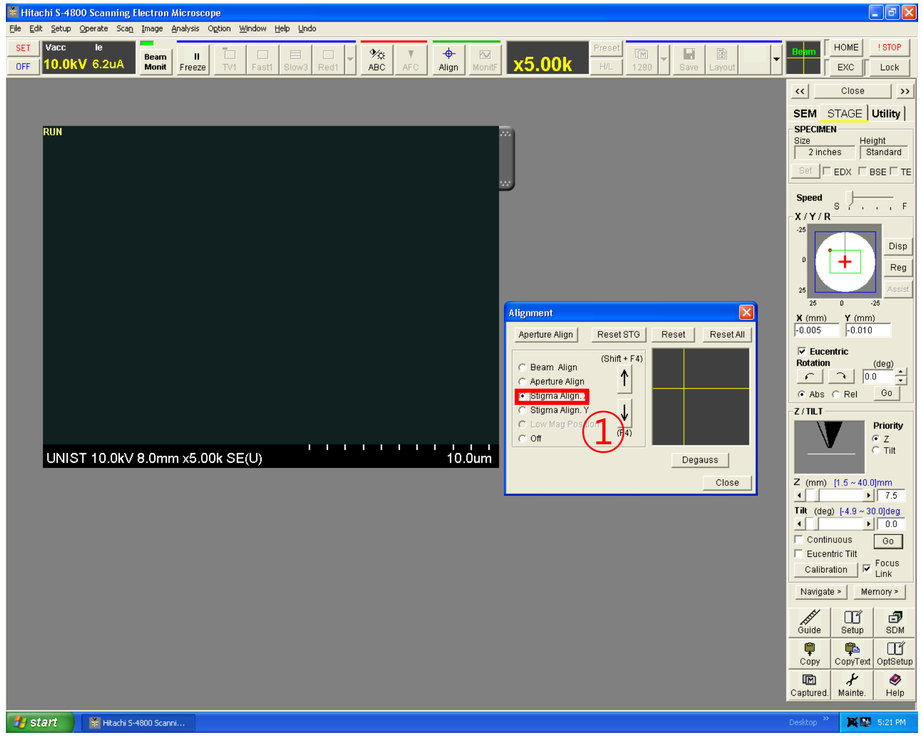
Beam Alignment

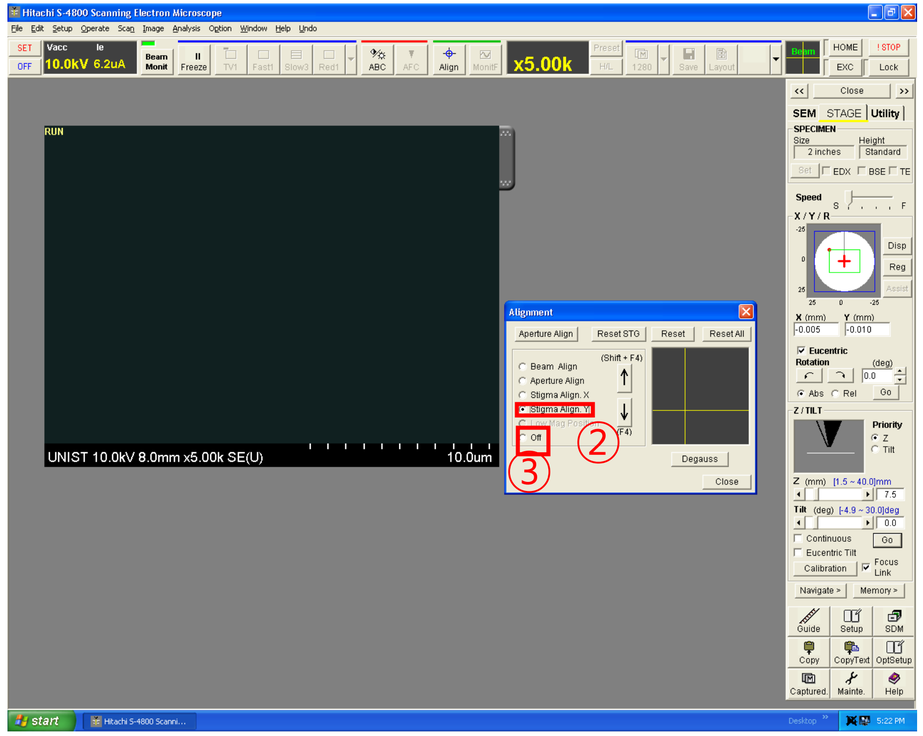




1. Click [Align] - [Beam Align] and adjust the Stigma X and Y knobs one by one to bring the beam to the center.
2. Click [Aperture Align] and adjust the stigma X and Y knobs one by one so that the specimen moves in place.(STIGMA X: ↔, STIGMA Y: ↕)

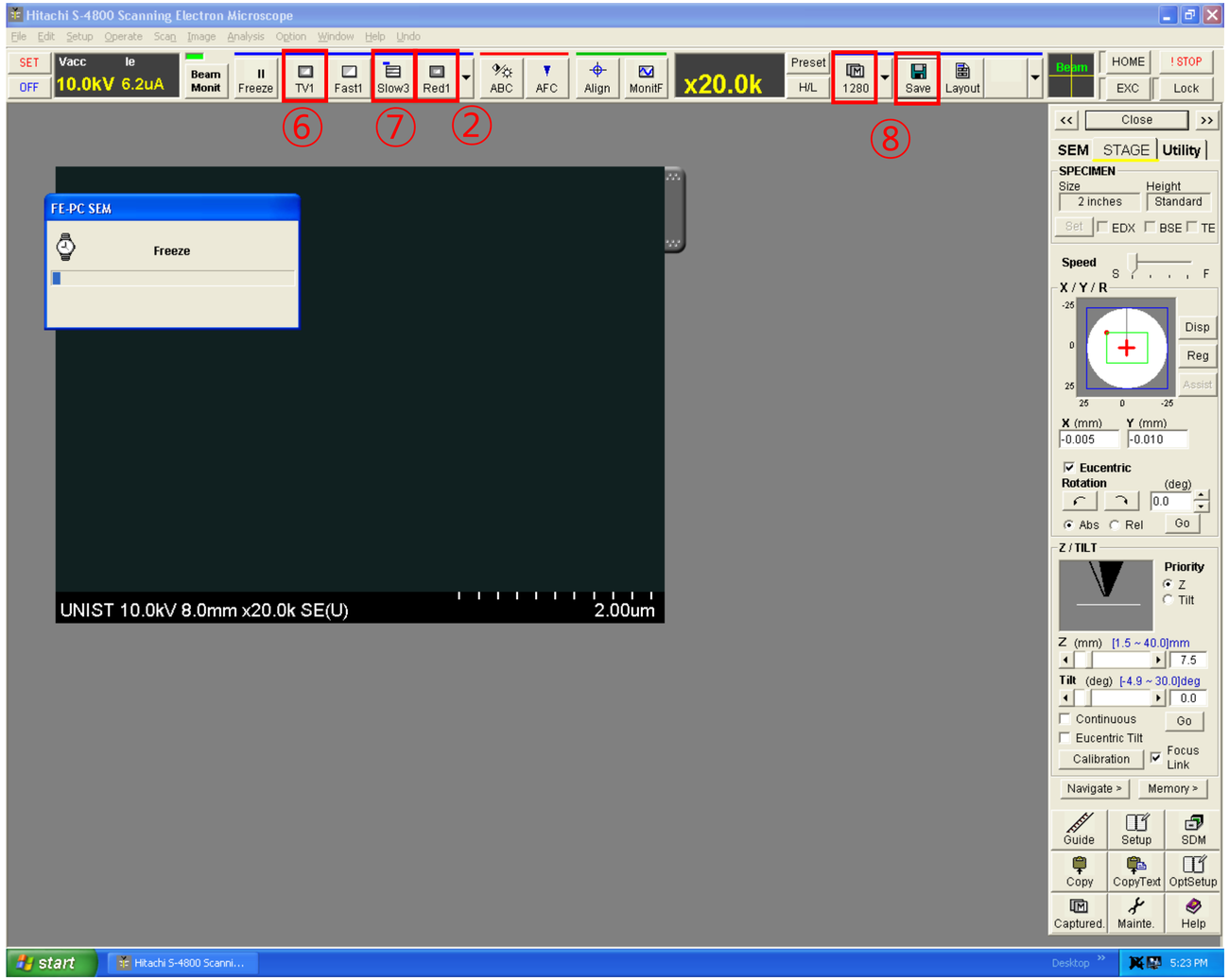
Beam Alignment



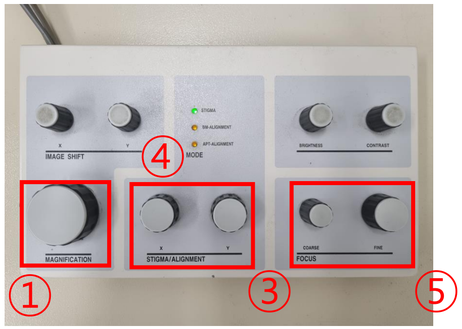


1. Click [Stigma Align.X], and adjust the stigma X and Y knobs one by one so that the specimen moves in place.(STIGMA X: ↖ ↘, STIGMA Y: ↗ ↙)
2. Click [Stigma Align.Y], and adjust the stigma X and Y knobs one by one so that the specimen moves in place.(STIGMA X: ↖ ↘, STIGMA Y: ↗ ↙), and click [Close].

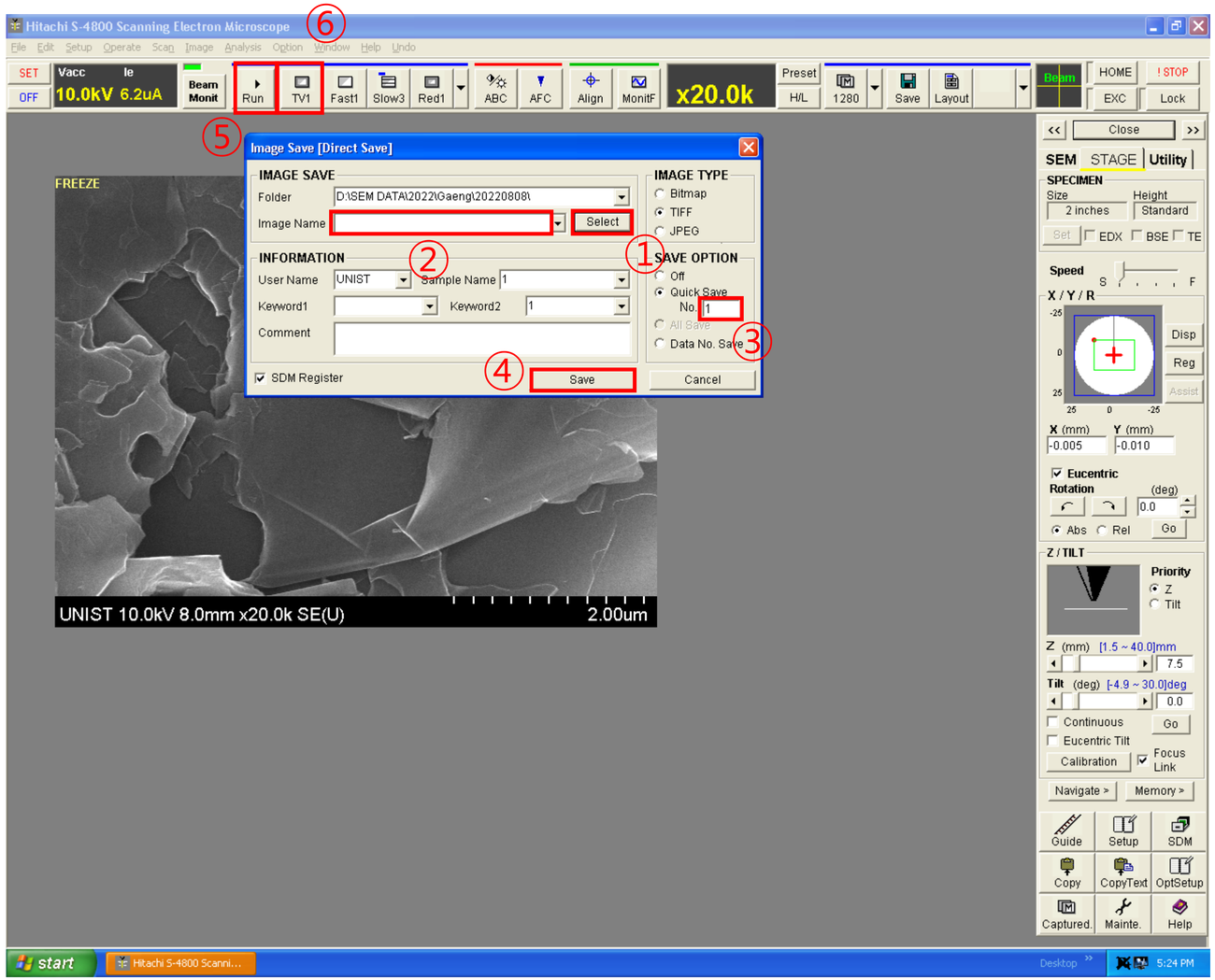
Saving the Image



1. Move to the location you want to observe.
2. Adjust the magnification slightly higher than the magnification you want to observe.
3. Click [Red1].
4. Adjust the knobs [FOCUS]-[STIGMA X, Y]-[FOCUS].
5. Adjust to desired magnification.
7. Click [Slow3].
8. Click [Save] or [1280].

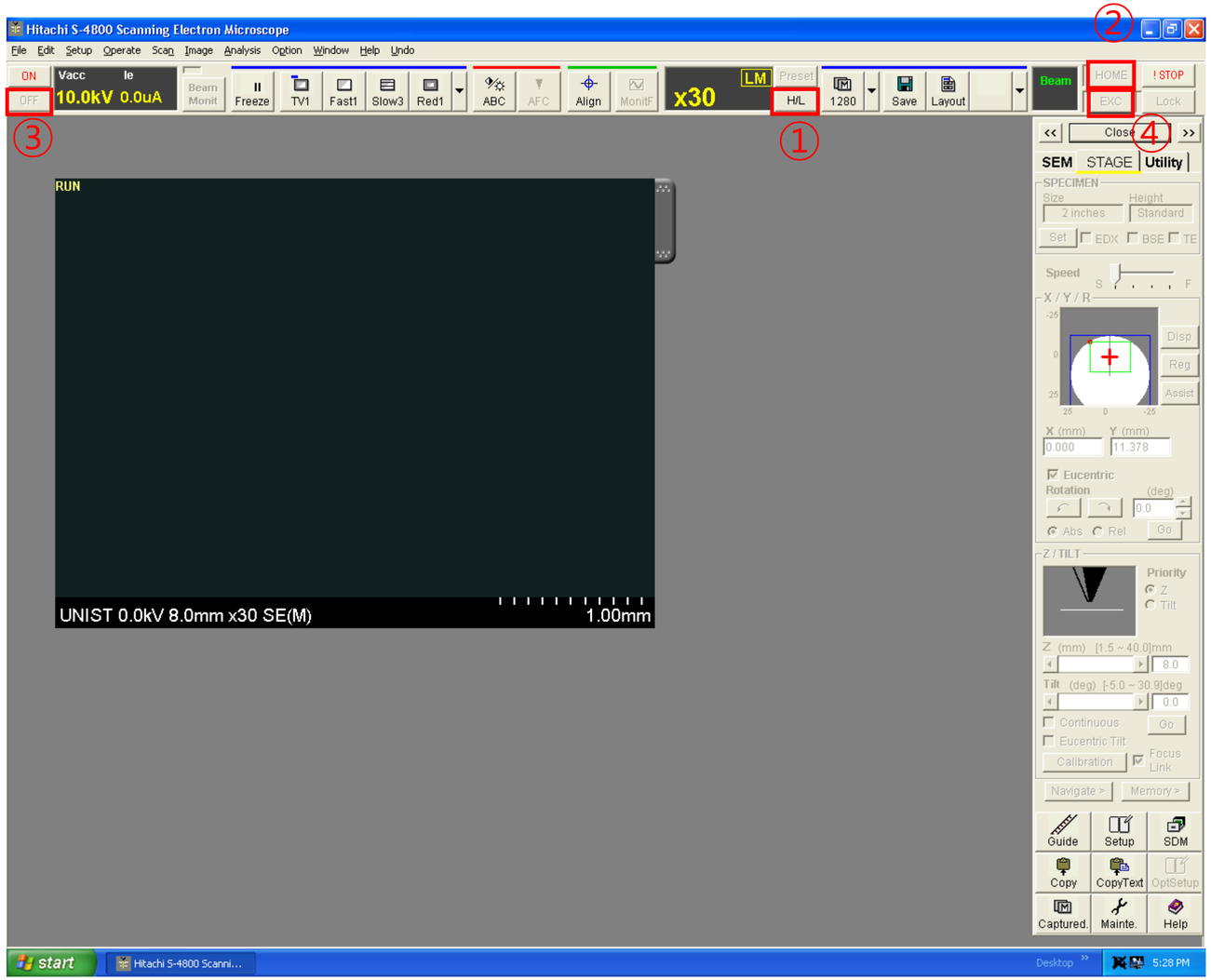


Saving the Image



1. Click [Select].
2. Create a folder.(Desktop-SEM data-Analysis year-Professor folder-Personal folder-Date folder)
3. Enter a sample name in [Image Name].
4. Enter 1 in [Quick Save No.]
5. Click [Save].
6. Click [Run]-[TV1].
7. Repeat steps 1 in the previous slide to 6 in this slide.

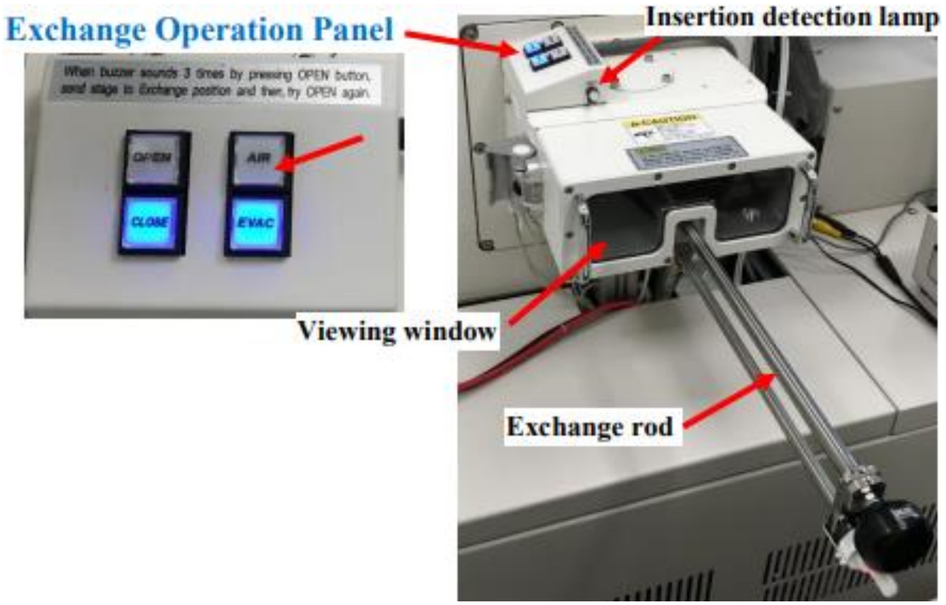
Finish



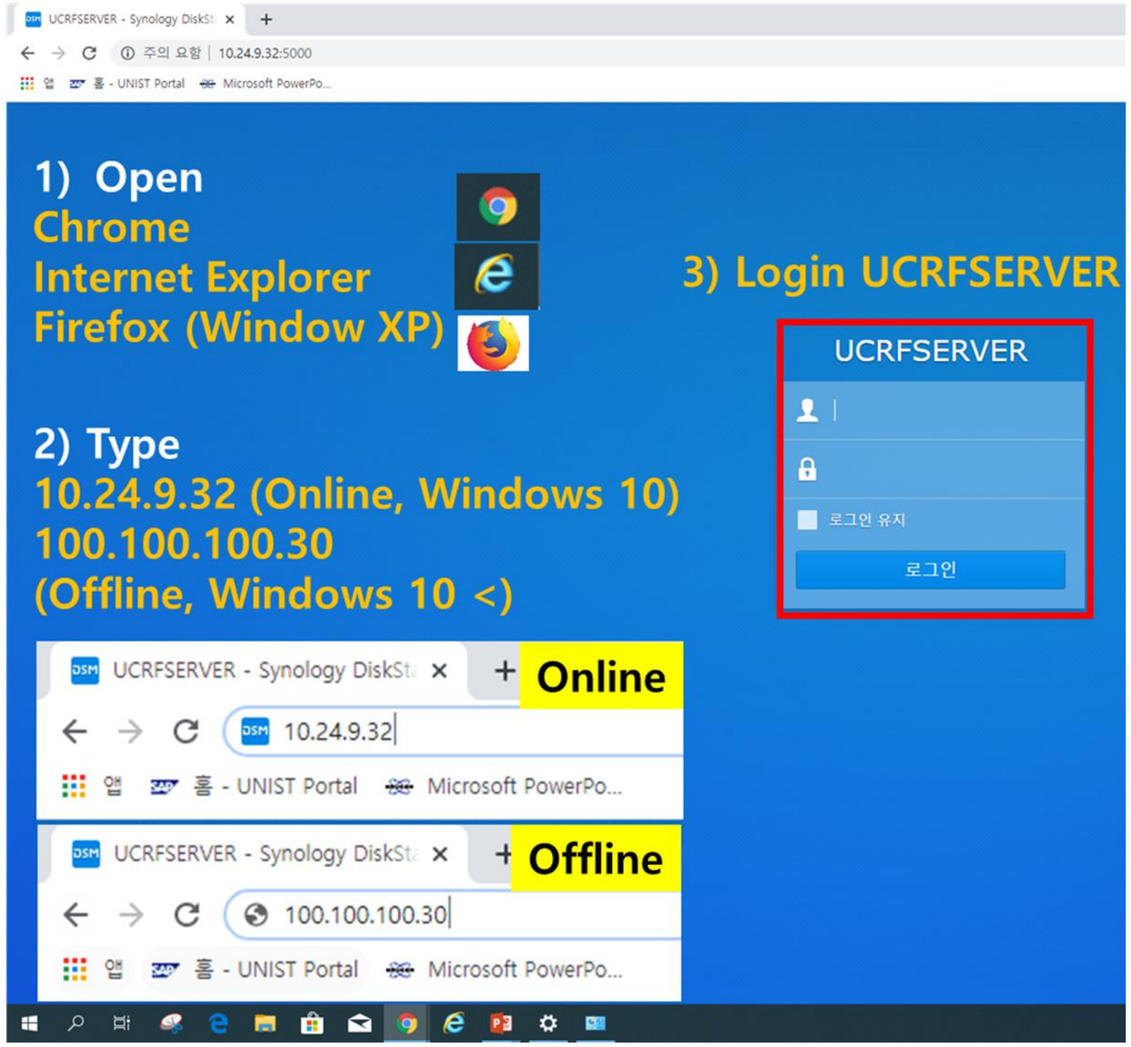
1. Click [H/L].
2. Click [Home].
3. Wait for the green light to stop flashing.
4. Click the [OFF].★
5. Click [EXC].★
6. Wait for the green light to stop flashing.
7. Click [X] to close the program.
8. Click [OK].

Retrieving the Holder

1. Press [OPEN], wait until the buzzer sounds.
2. Push the rod into the chamber.
3. Turn the specimen holder lock/unlock knob to LOCK position.
4. Pull out the rod all.★
5. Press [CLOSE], wait until the buzzer sounds.
6. Press [AIR], wait until the buzzer sounds.
7. Open the exchange chamber door.
   (Do not hold the exchange rod to open the door.)
8. Turn the knob clockwise to release.
9. Remove the specimen stage from the exchange rod.
10. Close the exchange chamber door.
11. Press [EVAC], wait until the buzzer sounds.



Transfer SEM data



1. Do not use USB when transferring SEM data.
2. Double-click the web browser.(Chrome, etc..)
3. Enter 100.100.100.30. in the address bar.
   (Enter 10.24.9.32 when downloading from Lab)
4. Enter your Lab ID and password.
5. Find your professor's folder and create your own folder.
6. Drag the SEM data into your own folder.
7. Close the window when the move is complete.

- UCRF server manager: Park Ji-hye(052-217-4035)

Daily Checklist in Lab

1. Put the specimen holder back in place.★
2. When using weekends or holidays, check the [Daily checklist in lab] and sign the checker's signature.
3. Enter the name of your advisor and submit it in the original place with your signature or stamp.★

Daily checklist in lab.

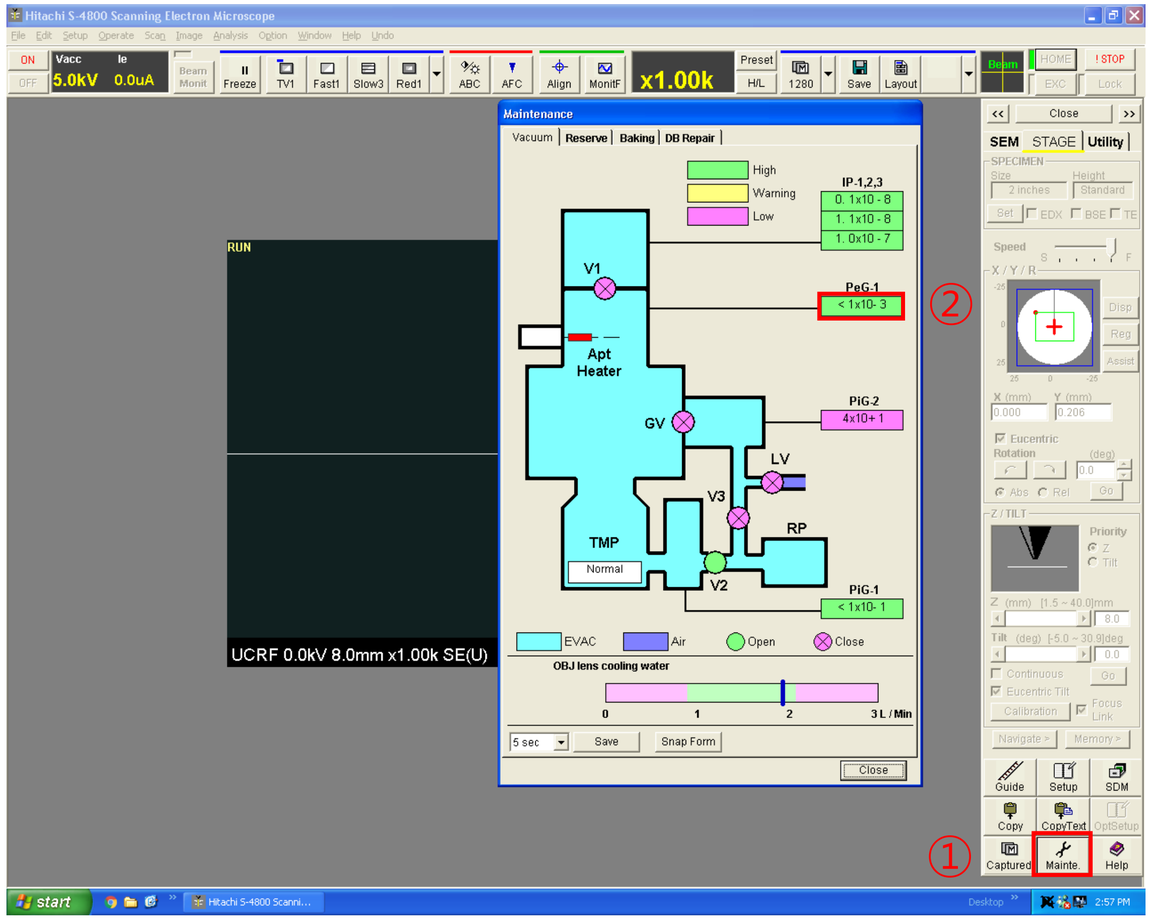
Lab title	Electron Microscopy Preparation	Bldg./NO.	102-B115	Date	

Division	checkpoint	check
General Safety	Laboratory overall cleanliness condition.	
	Smoking or bringing food into the lab.	
	Management status of experimental equipment such as safety regulations, safety signs, personal protective equipment, first aid, etc.	
	Checking the presence of a pre-hazard risk analysis report.	
Electric	Checking the power supply status of unused electrical equipment and checking for overloaded outlets.	
	Using grounded outlets, checking damage on the insulating coating of electric wiring, electric wiring arrangement.	
	Checking ground conditions for preventing external or static disturbances of the instrument.	
	Non-load status around electric panelboards.	
Fire	Fire extinguisher sign, proper fire extinguisher and regular inspection status.	
	Emergency exits, escape routes, and any obstacle blocking the passage.	
	Storage of foreign substances around fire hydrant and fire extinguishers.	
Gas	Outdoor storage of gas containers, no risk of tipping over, and checking ventilation conditions.	
	Corrosion, deformation, nozzle lock status on the exterior of the gas containers and checking the packing time limit of gas containers.	
	Checking installation and operational status of gas leakage detection alarm, anti-backflow/anti-backfire prevention devices, neutralizing decontamination devices.	
	Attachment of pipe marks, gas facility boundaries/warning marks and operation status of regulators and valves.	
	Safe separation distance from surrounding fire hazards.	
Chemical	Keeping hazardous factors handling and management registers and MSDS.	
	Categorizing chemicals by description and storing chemical reagents in safety cabinets.	
Equipment check	SU8220 Cold FE-SEM user: Check the pressure of nitrogen gas	

Confirm	Inspector Signature	(sign)
	Lab Director Signature	(sign)

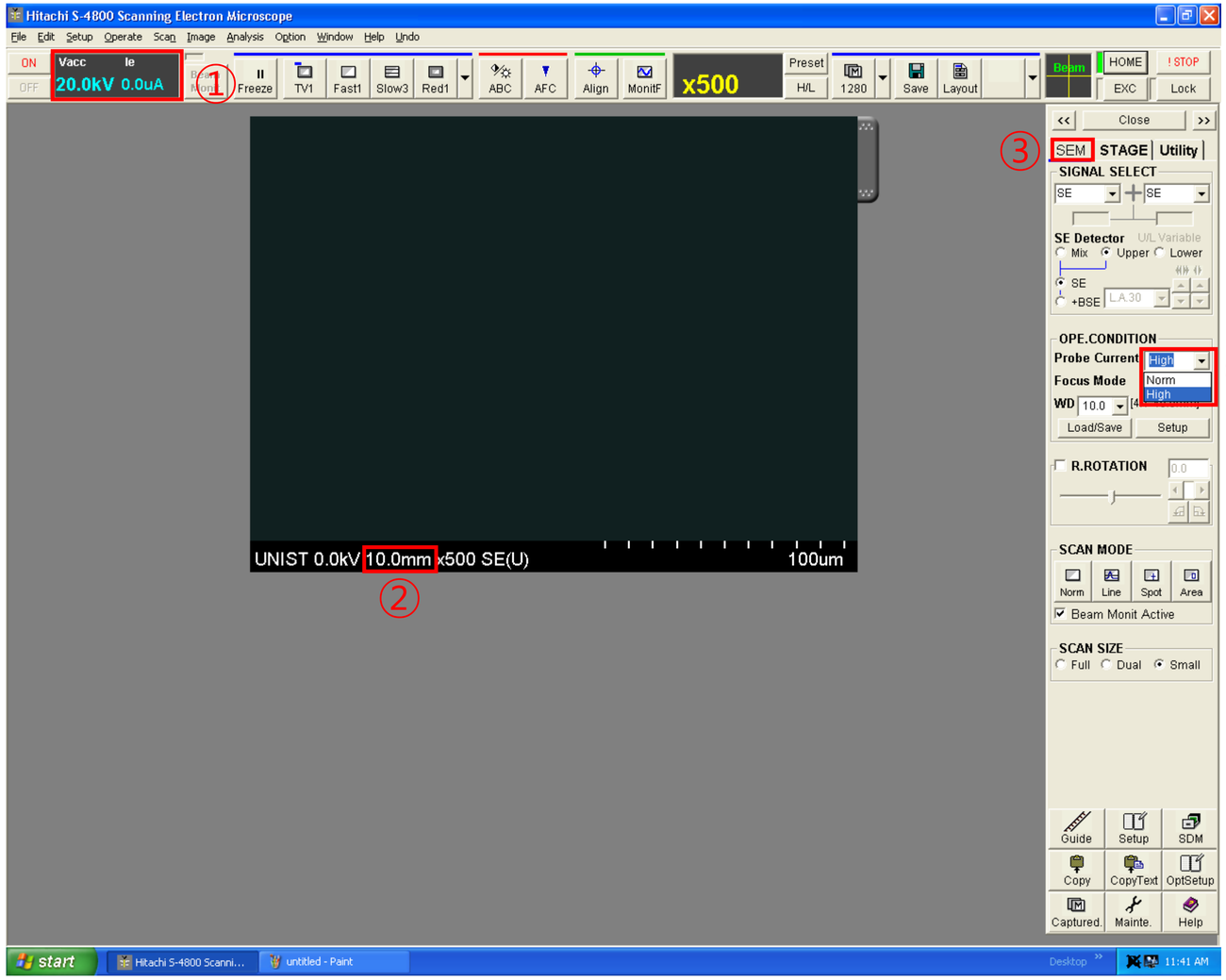
If you encounter a problem while using SEM

- When [ON] is not activated



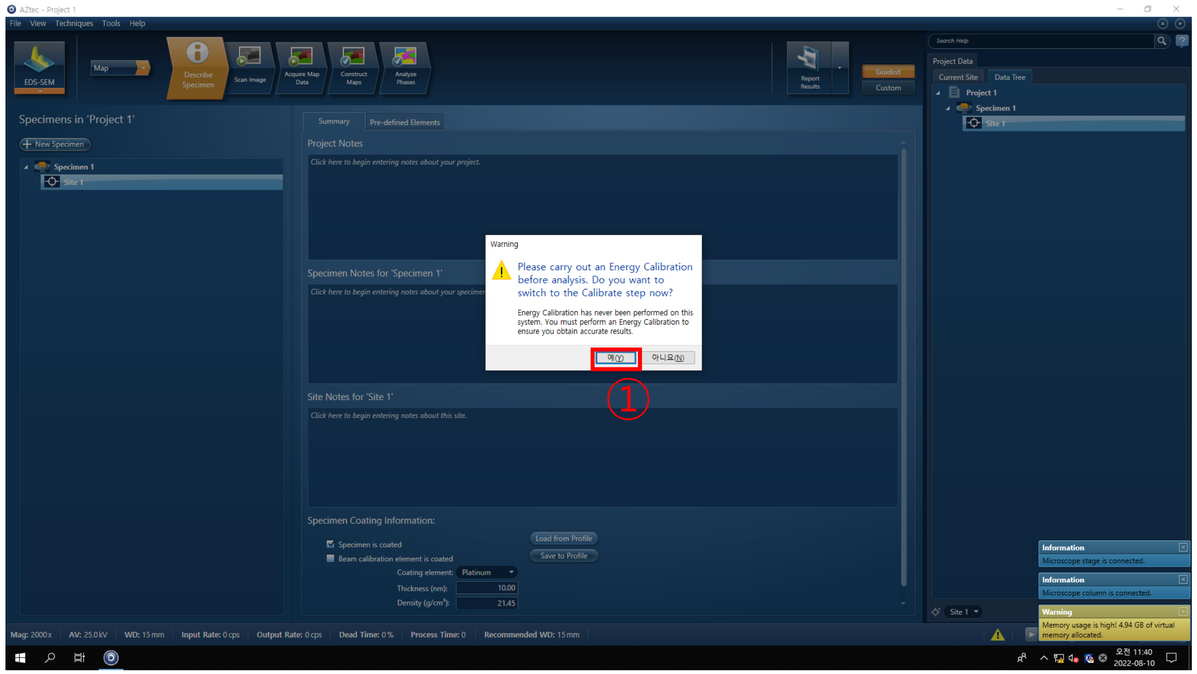
1. Clcik [Mainte.]
2. Wait until the value of PeG-1 becomes <1x10-3 (green)
3. [ON] is activated.

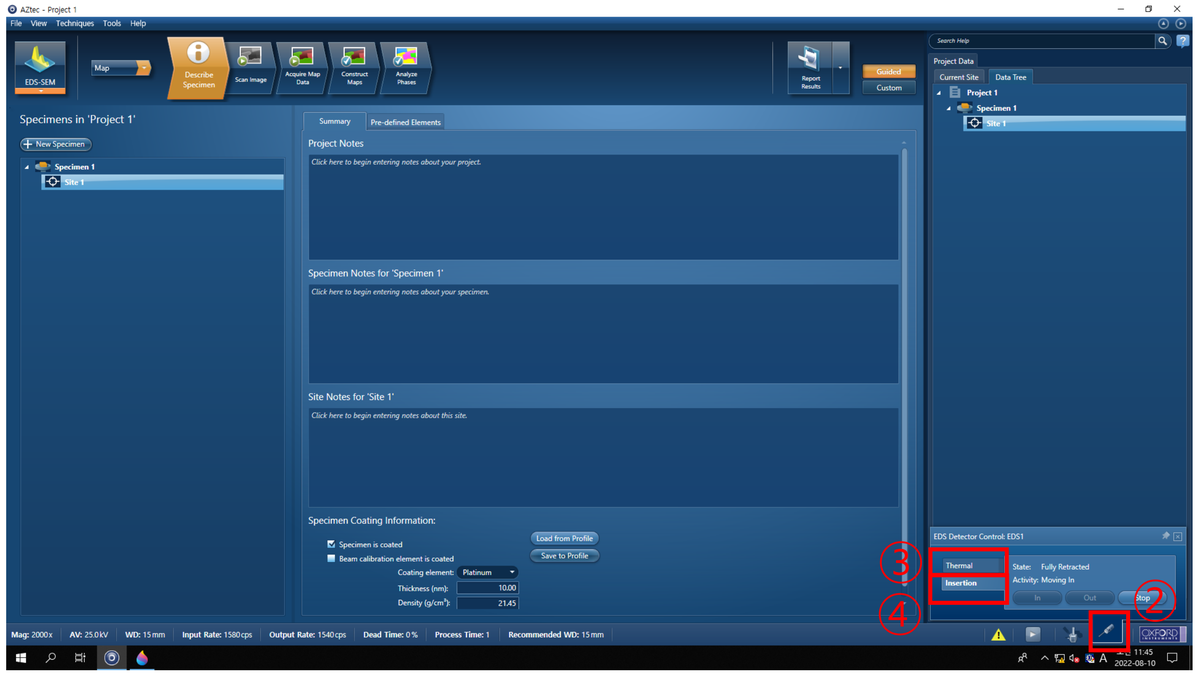
Energy Dispersive Spectroscopy



1. Click the [Electron Beam]
2. Select the acceleration voltage in [Vacc.].★
   (1.5 times kV of the x-ray value of the element to be observed or unknown specimens: 20 kV)
3. Select beam current (15~20) in [Set Ie to]
4. Click [Close]
5. Select [High] in [Probe Current].★
6. Adjust WD to 15.
7. Move to the position of the specimen to be measured and adjust the magnification.

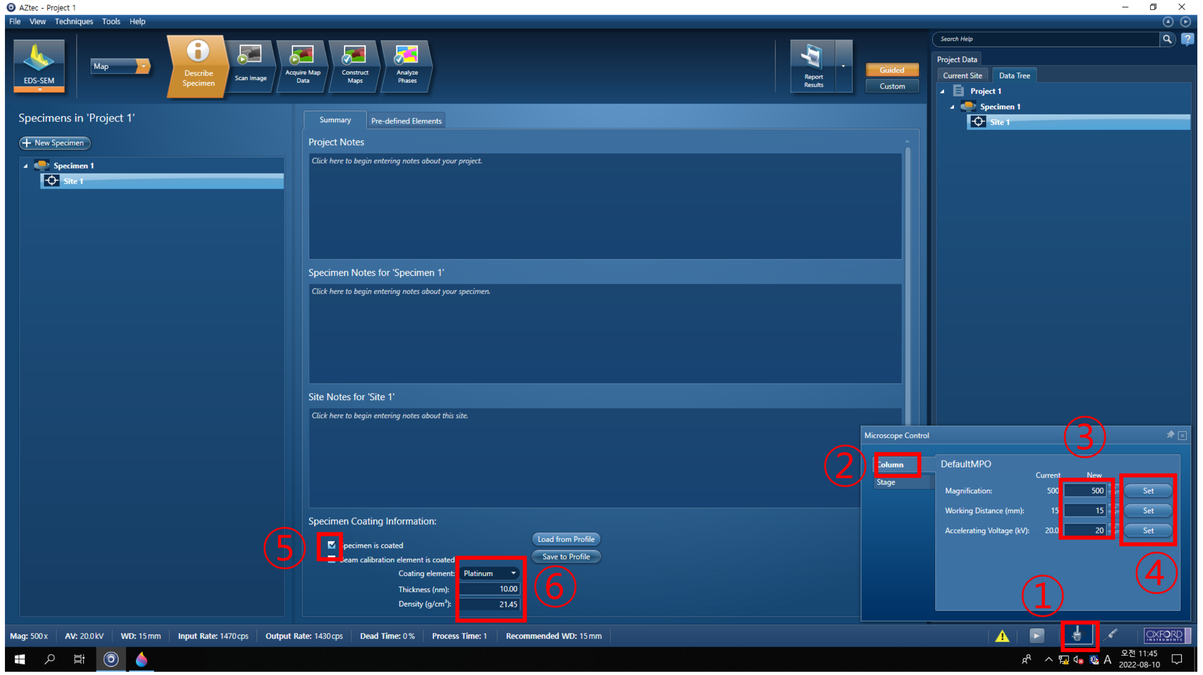
Energy Dispersive Spectroscopy

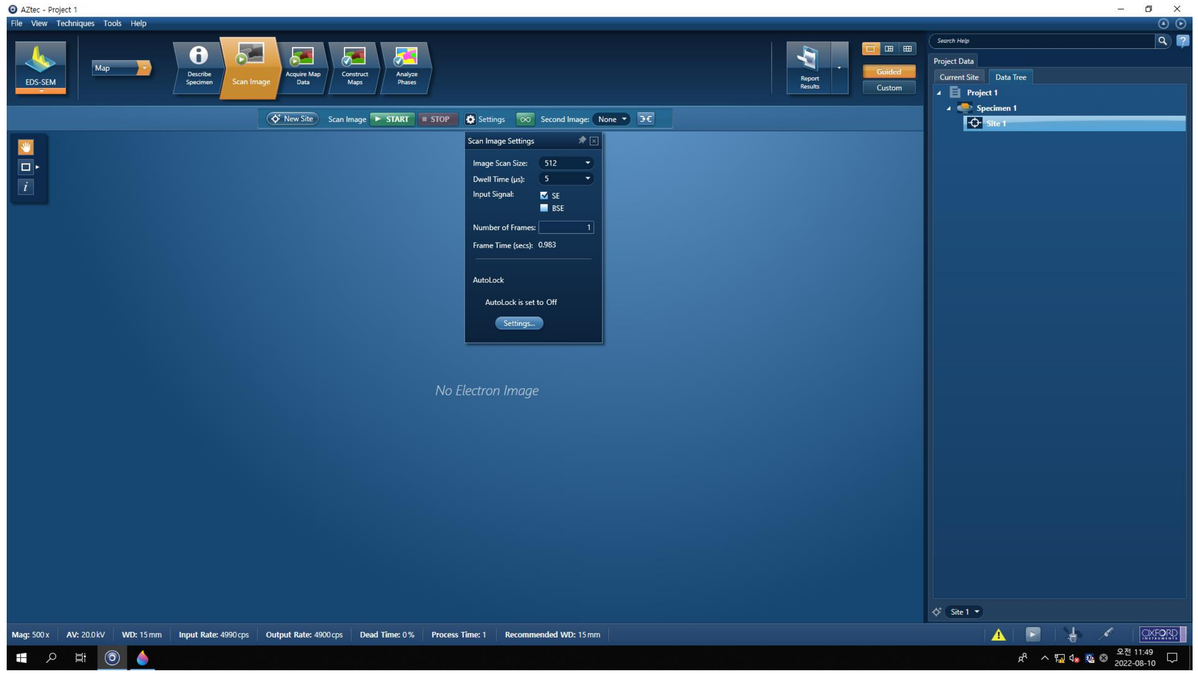




1. Double click [Aztec]
2. Click [예(Y)]
3. Click [EDS Detector Control] – [Thermal] – [Operate]
4. Click [Insertion] and click [In]

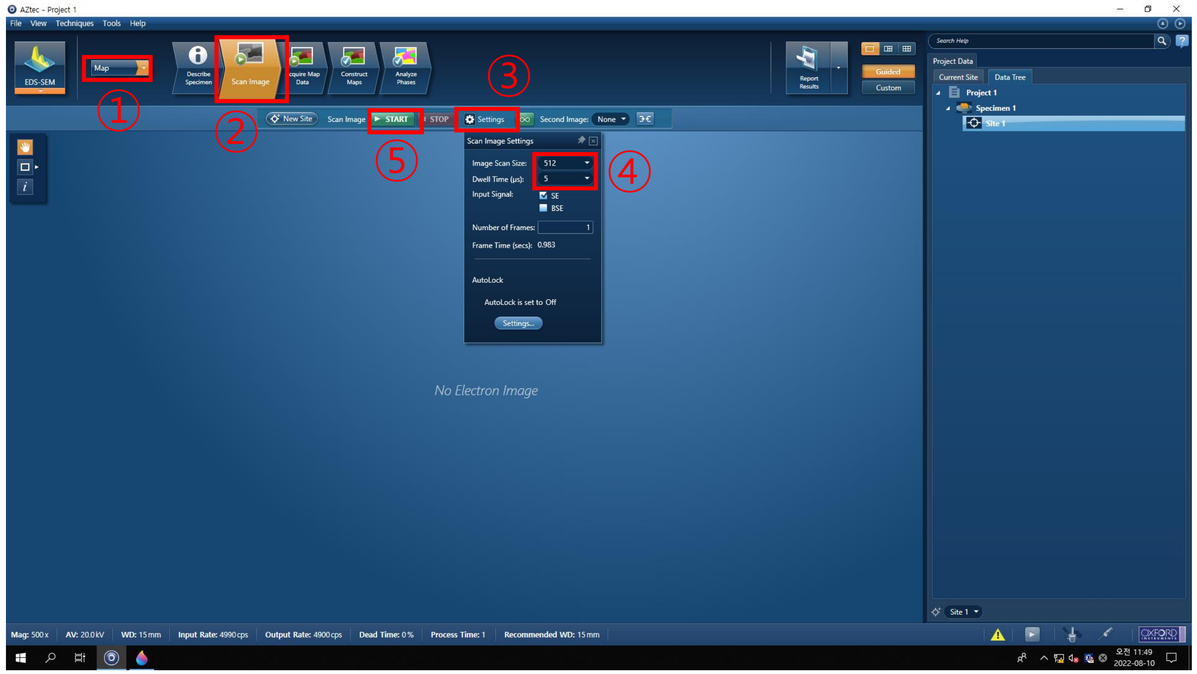
Energy Dispersive Spectroscopy

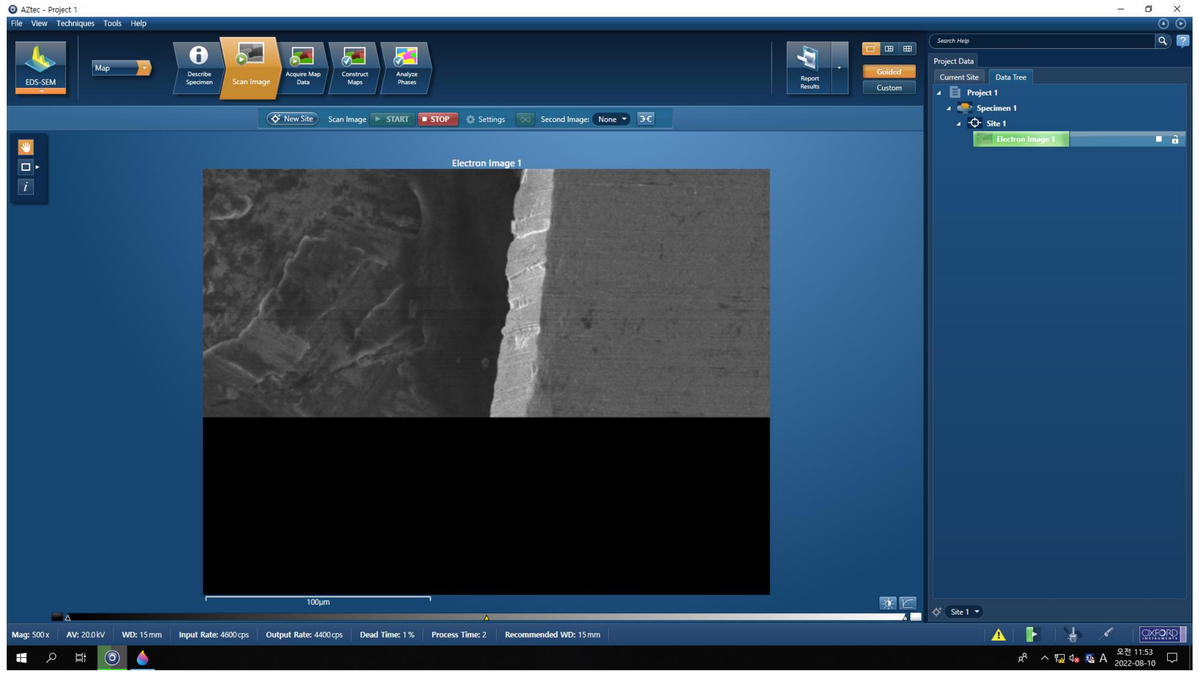




1. Click [Microscope Control]
2. Click [Column]
3. Enter the magnification, WD, and acceleration voltage and click [Set]
4. If the specimen is coated, check and select coating material.

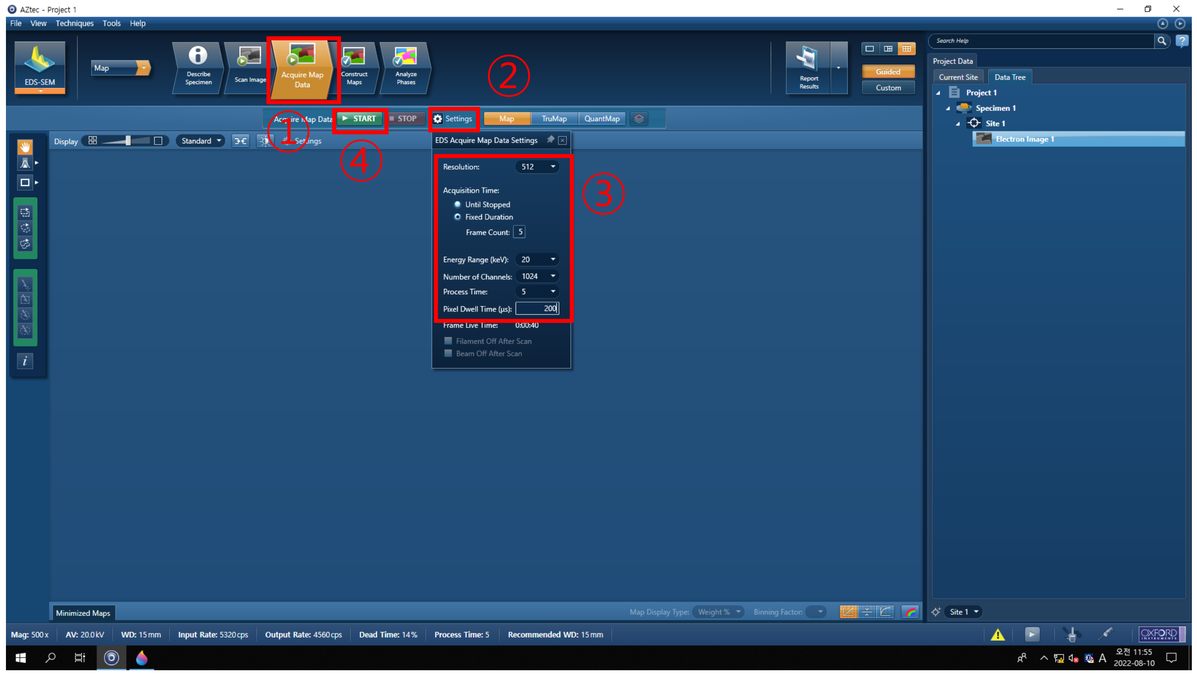
Map





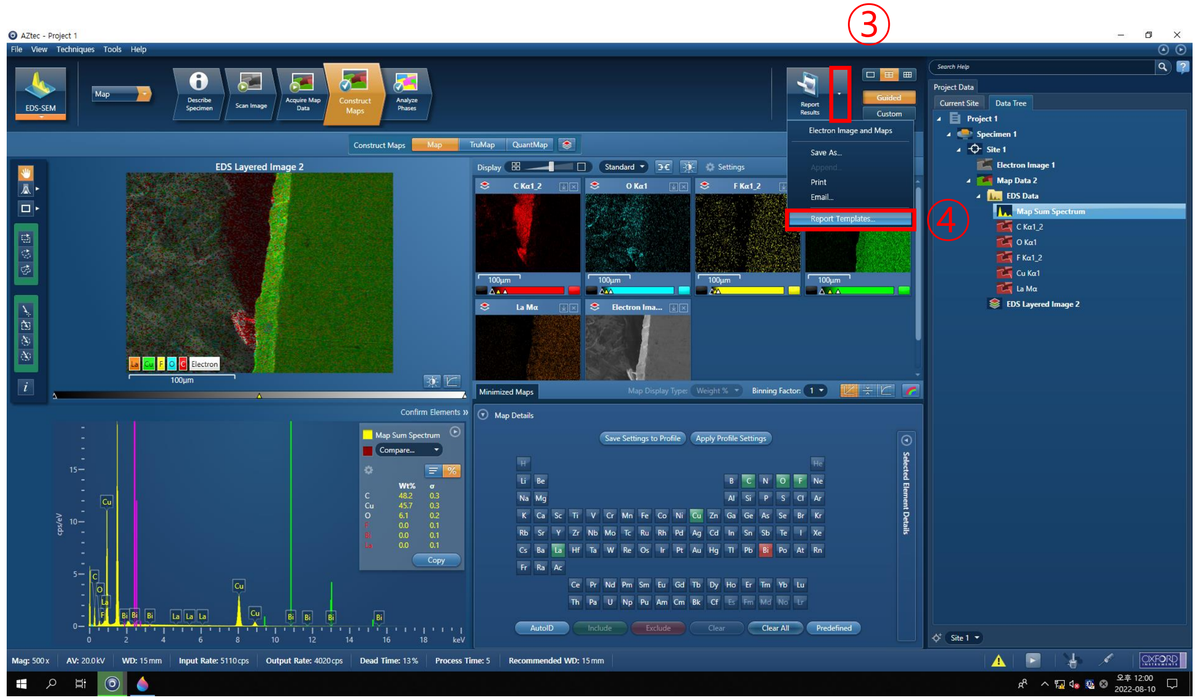
1. Select [Map]
2. Click [Scan Image]
3. Click [Setting]
4. Click [START]

Map



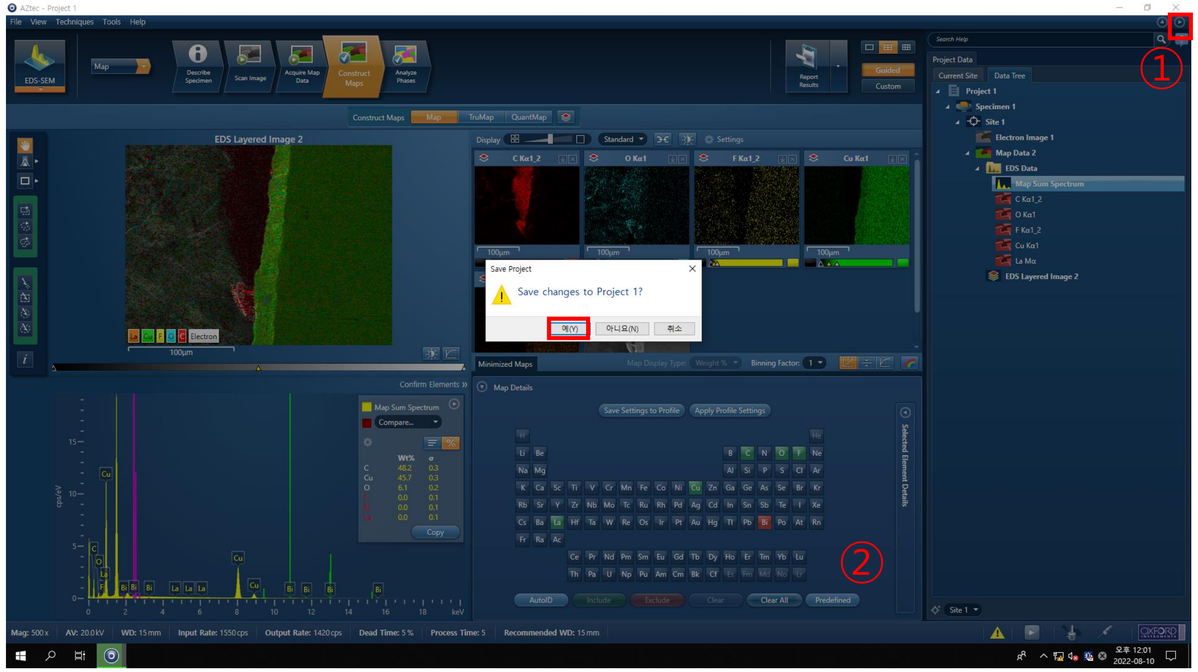
1. Click [Acquire Map Data]
2. Click [Setting]
3. Choose your analysis time
4. Click [START]

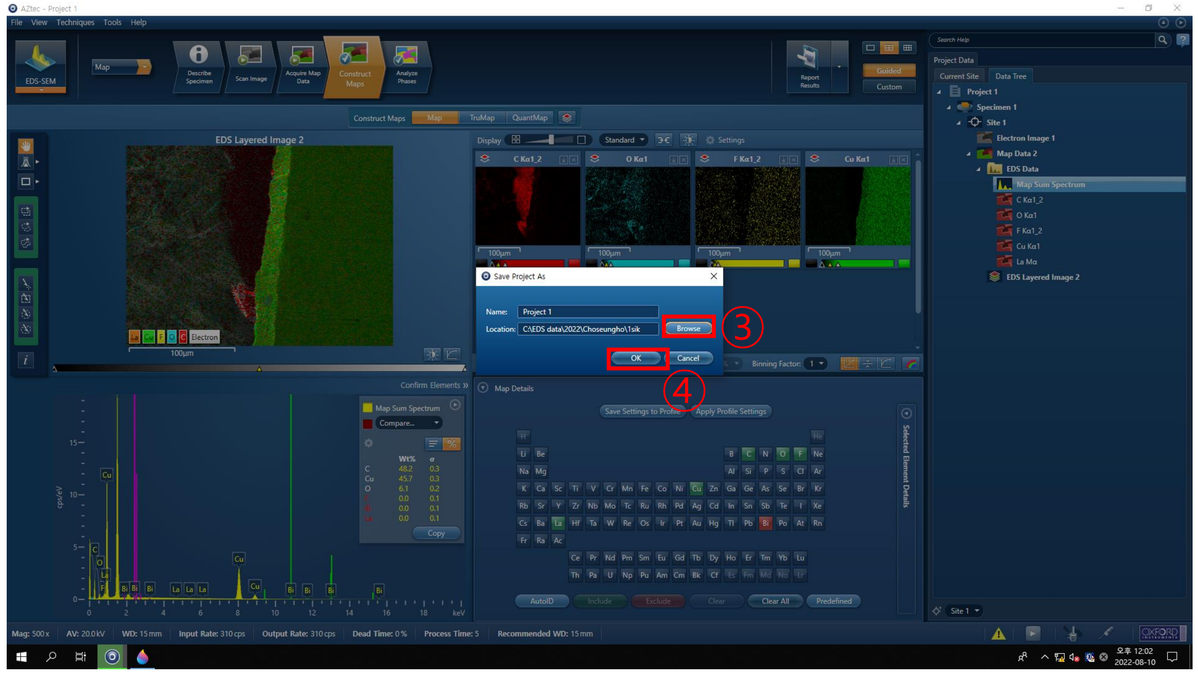
Map



1. Click [Construct Maps]
2. Click on the desired element to make it green
3. Click the arrow next to [Report Results]
4. Click [Report Templates]

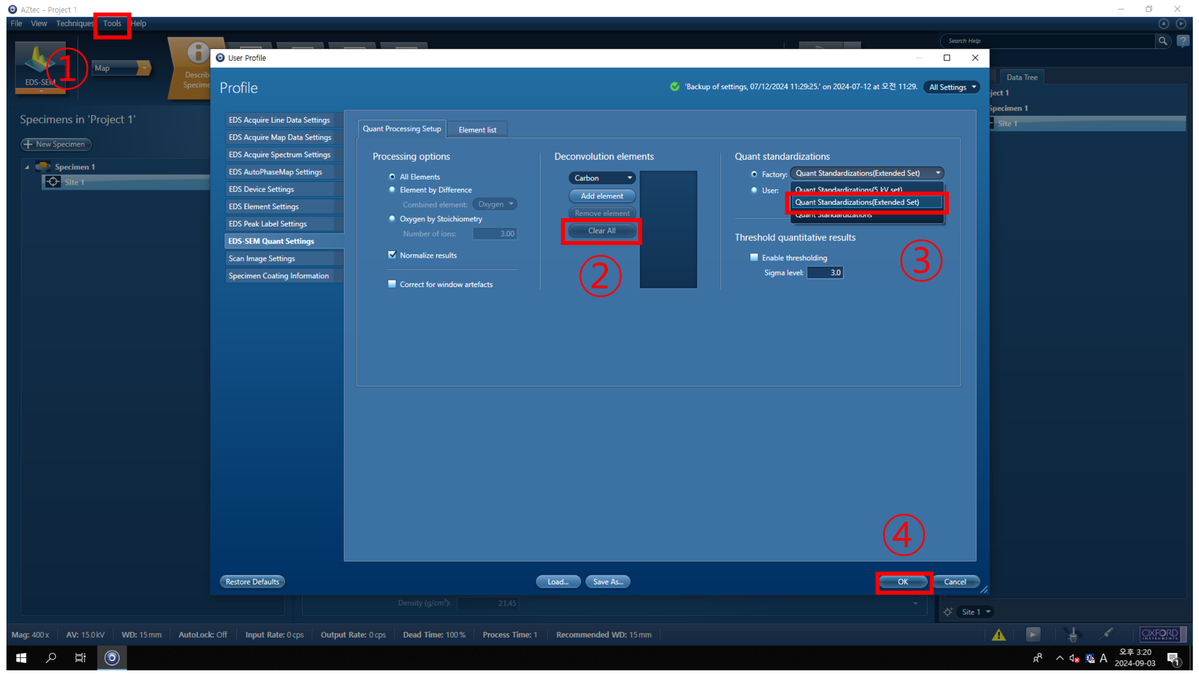
Map





1. Click [EDS Detector Control] - [Insertion] - [Out] – [Thermal] – [Standby]
2. Click [X] and click [예(Y)]
3. Click [Browse] and create a folder
4. Click [OK]

If you encounter a problem while using EDS



1. EDS connection problem

   1) Shut down the EDS PC.

   2) 2) Turn off the switch on the back of the controller on the EDS PC, and turn it back on after 10 seconds.

   3) 3) Use after booting the EDS PC.

   PC PW: ucrfumcl1!

2. When only some elements are analyzed

   1) Click [Tools] - [User profiles]

   2) Click [Deconvolution elements] - [Clear All]

   3) Click [Quant stadardizations] - [Factory] - [Quant Stadardizations[Extended Set]] - [OK]

Create Account

www.ucrf.unist.ac.kr

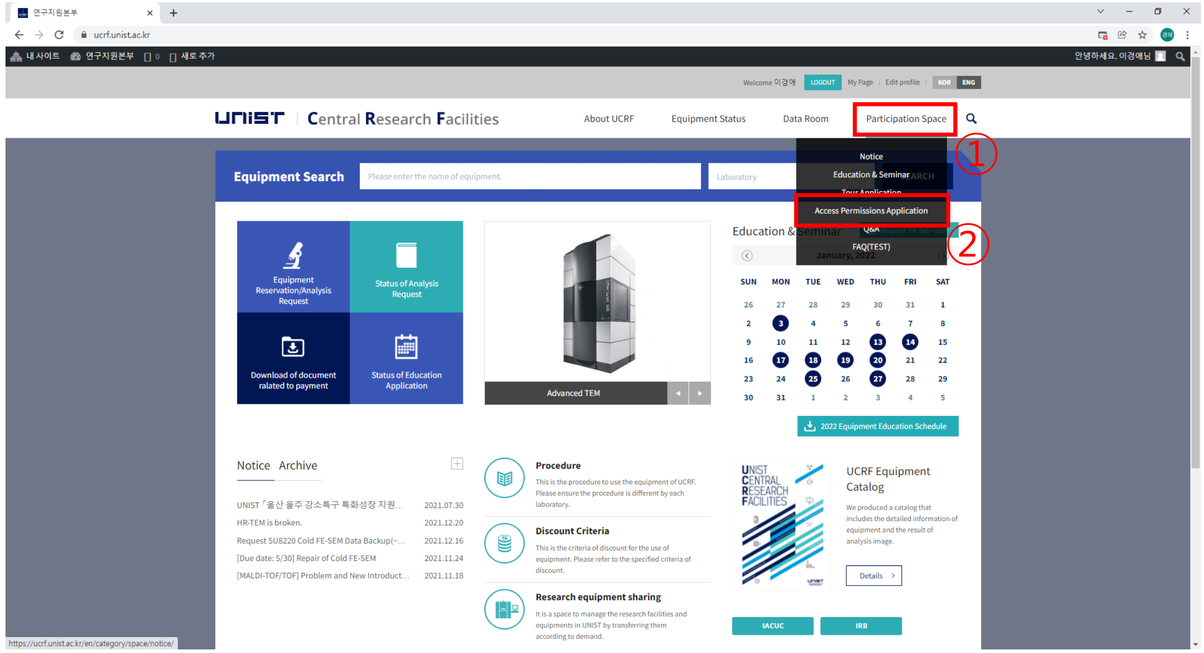
1. Click [Sign up]
2. Click [UNIST Member]
3. Input [Portal ID/PW] → Click [Confirm]

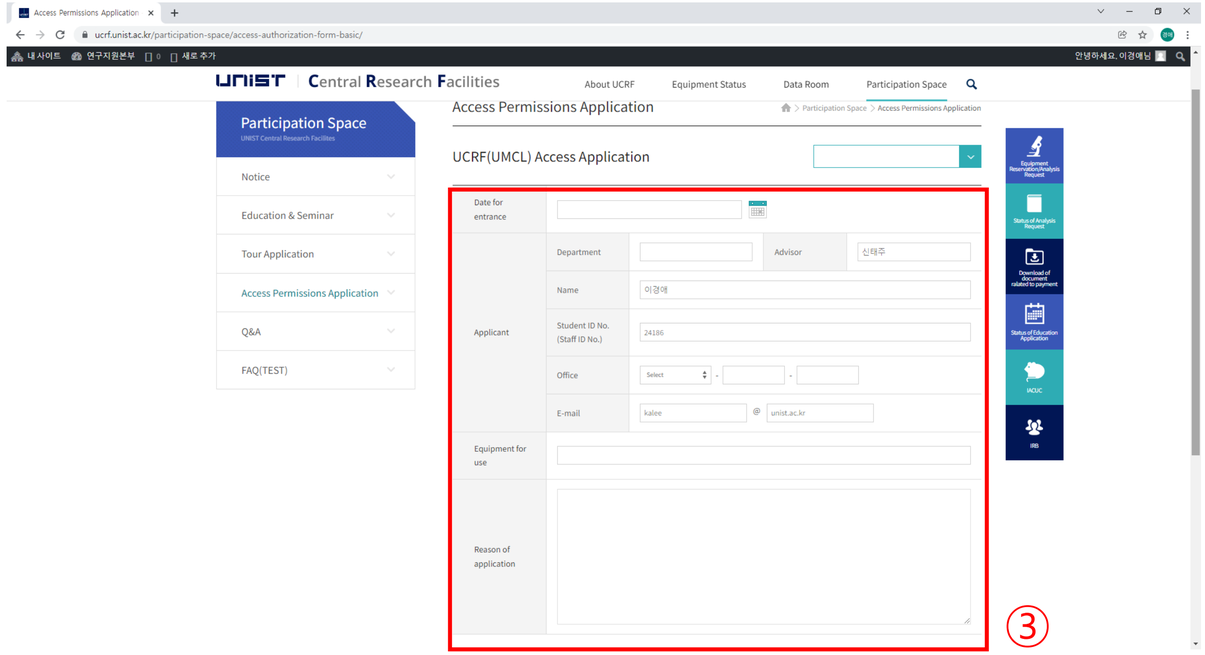
   Please check your information
4. Input professor name in [Principal Investigation]

   → [Professor search] → professor name
5. Click [Create Account]

Access Permissions Application

www.ucrf.unist.ac.kr

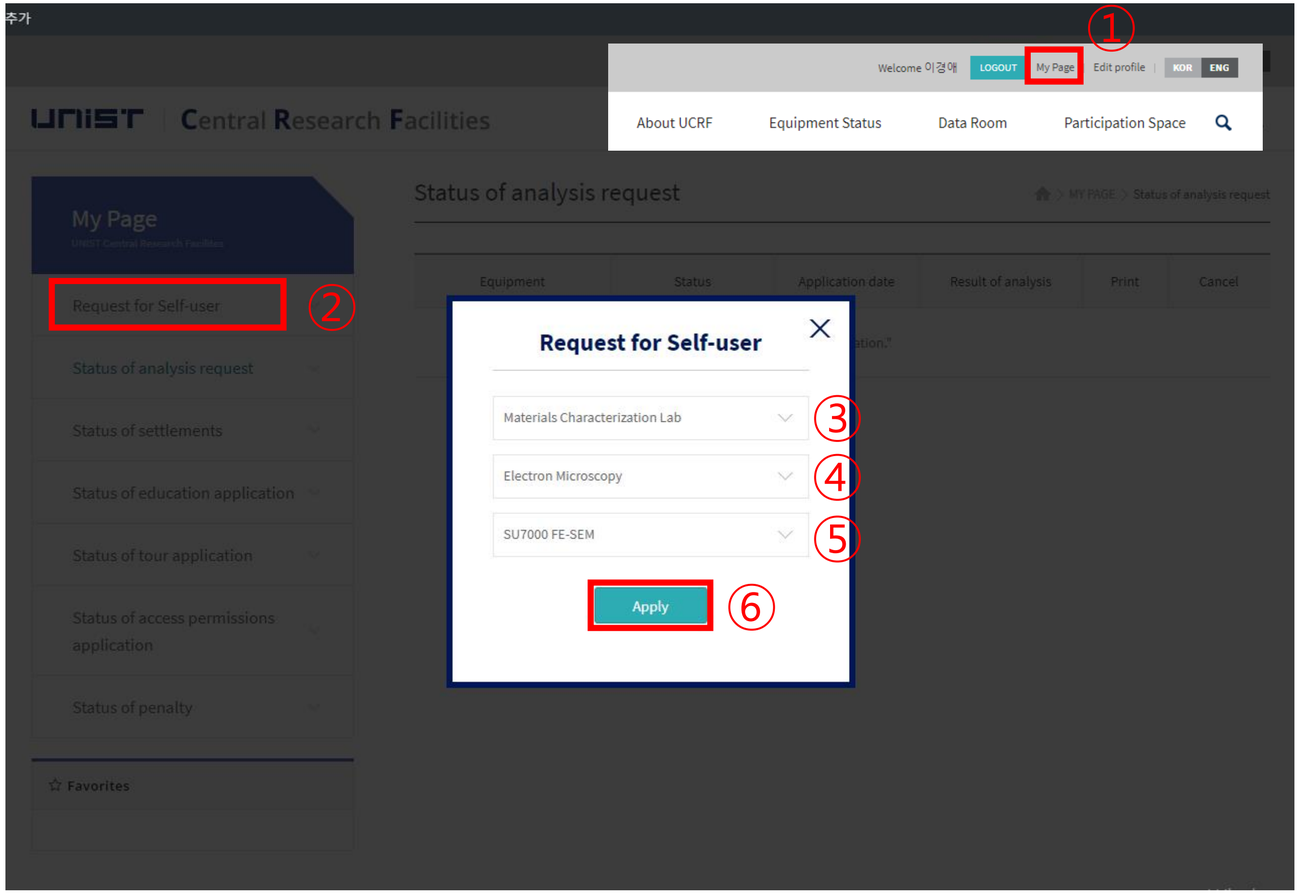




1. Click [Participation Space]
2. Click [Access permissions Application]
3. Fill out the UCRF(UMCL) Access Application form
4. Click [Apply]

- When reissuing the ID card, you must apply for access again on UCRF website.
- Access authorization officer:
  Kang Yeong-bi(052-217-4168)

Request for Self-user

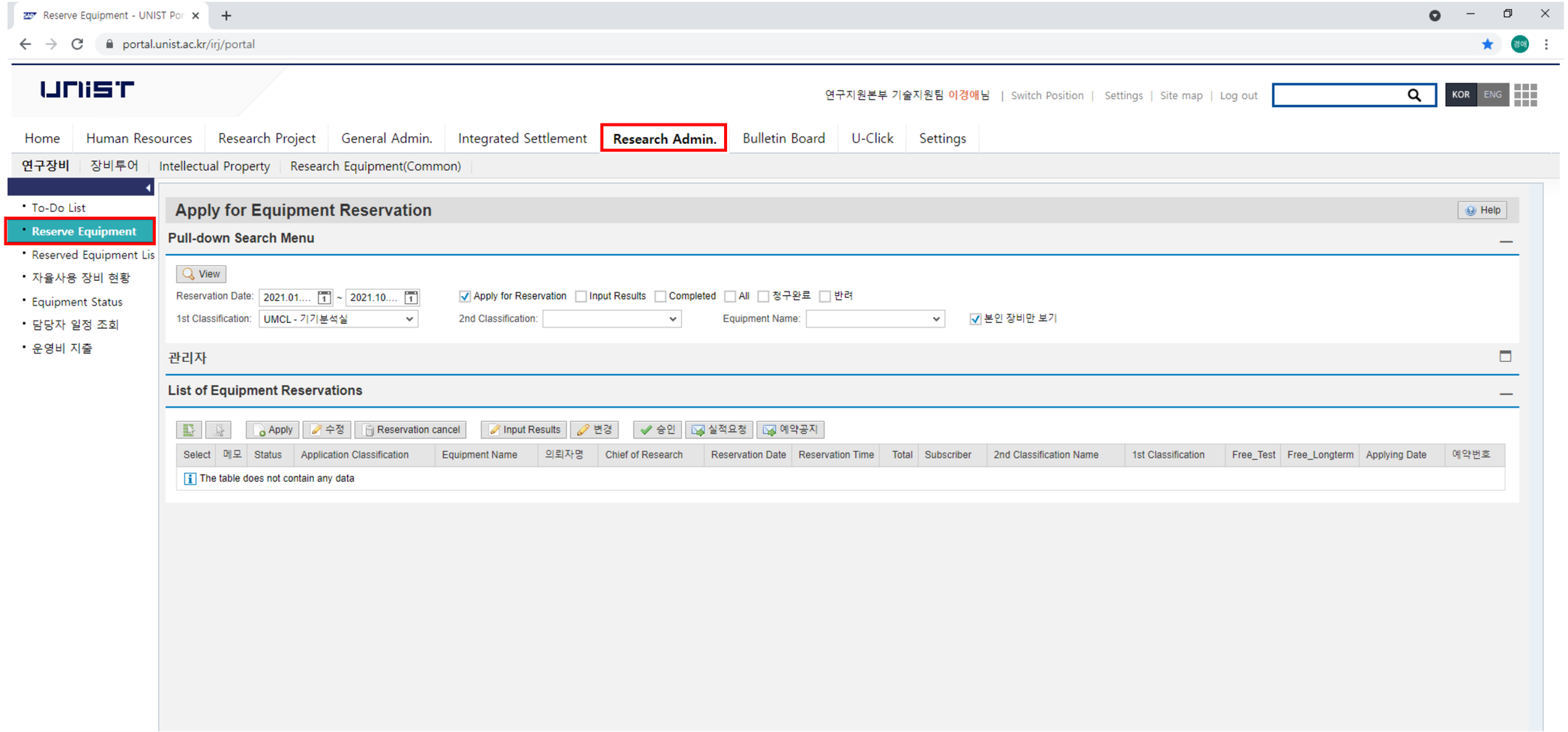


After pass the test,

1. Login UCRF website (**www.ucrf.unist.ac.kr**)
2. Click [My Page]
3. Click [Request for Self-user].
4. Select [Materials Characterization Lab]
5. Select [Electron Microscopy]
6. Select [Cold FE-SEM]
7. Click [Apply]

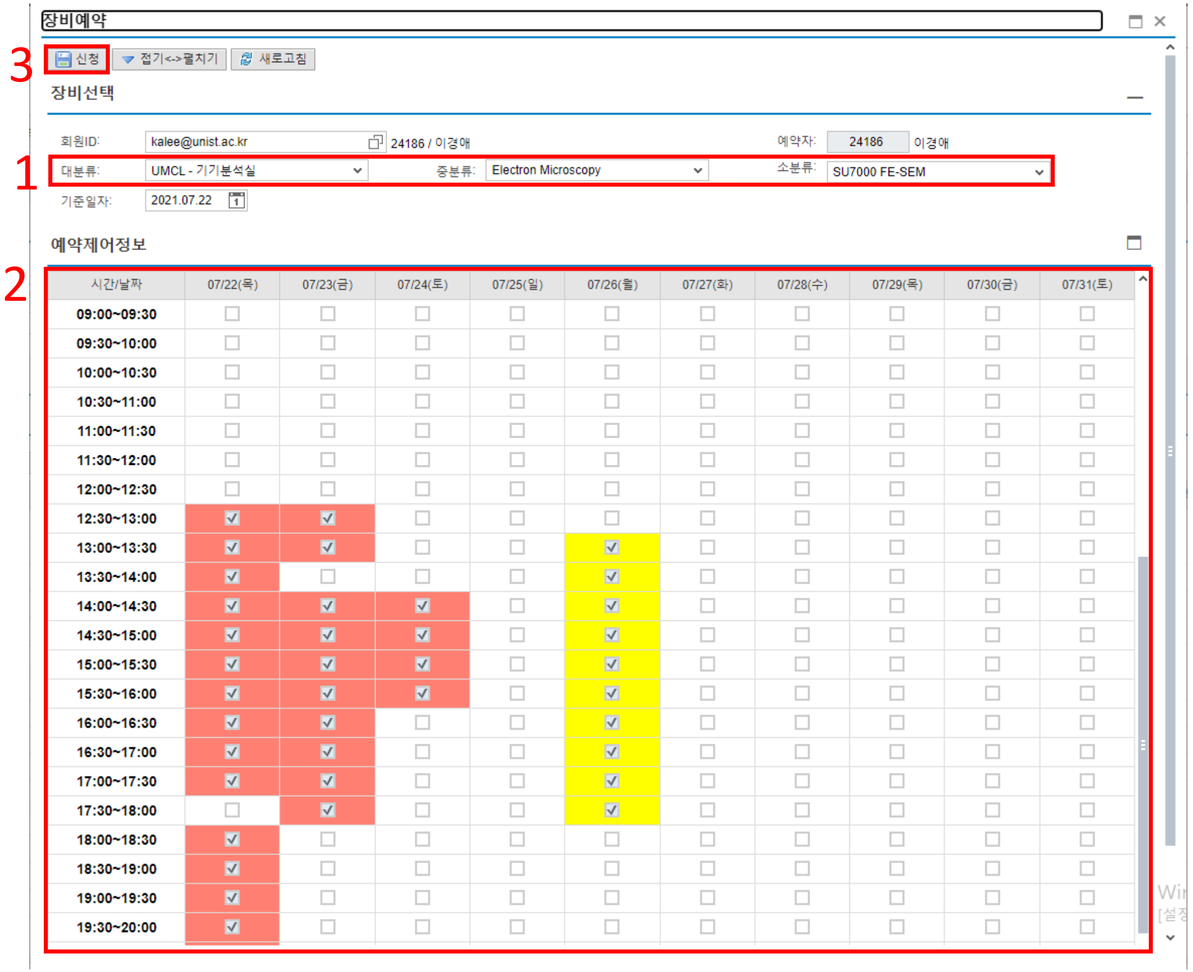
Reservation

portal.unist.ac.kr – Research Equipment – Equipment reservation/input result



Reservation

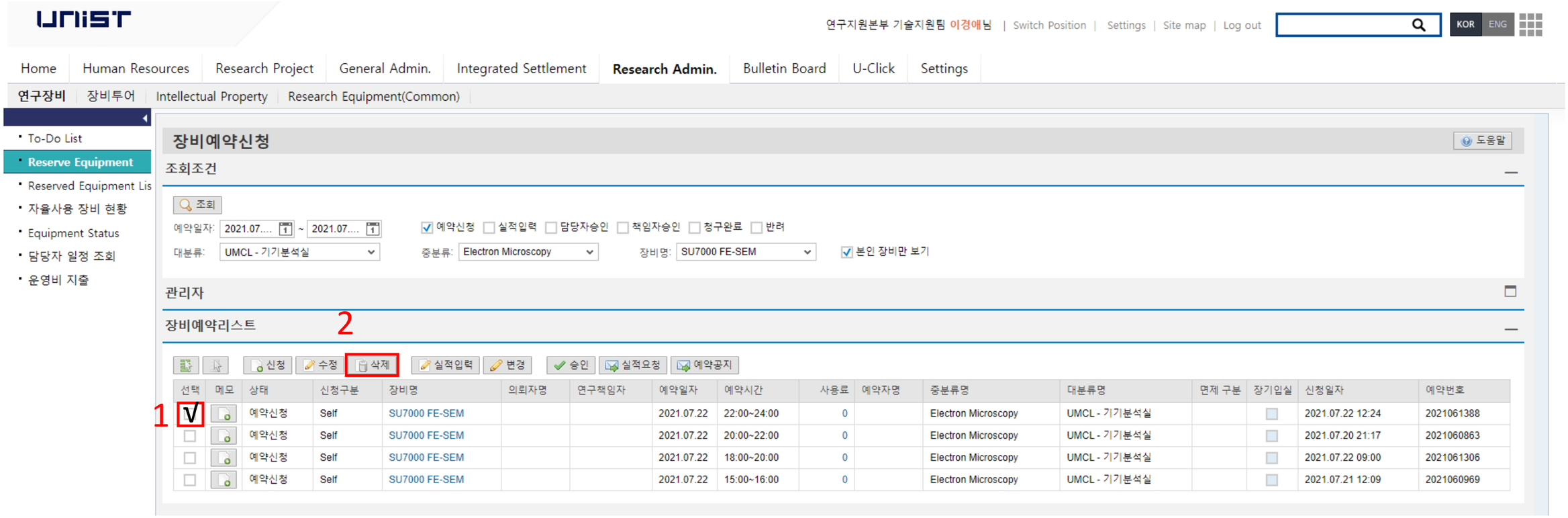
portal.unist.ac.kr – Research Equipment – Equipment reservation/input result



1. Select the classification and equipment
2. Select the time you want on white box
   - Yellow box : my reservation
   - Red box : others reservation
3. Click [Application]

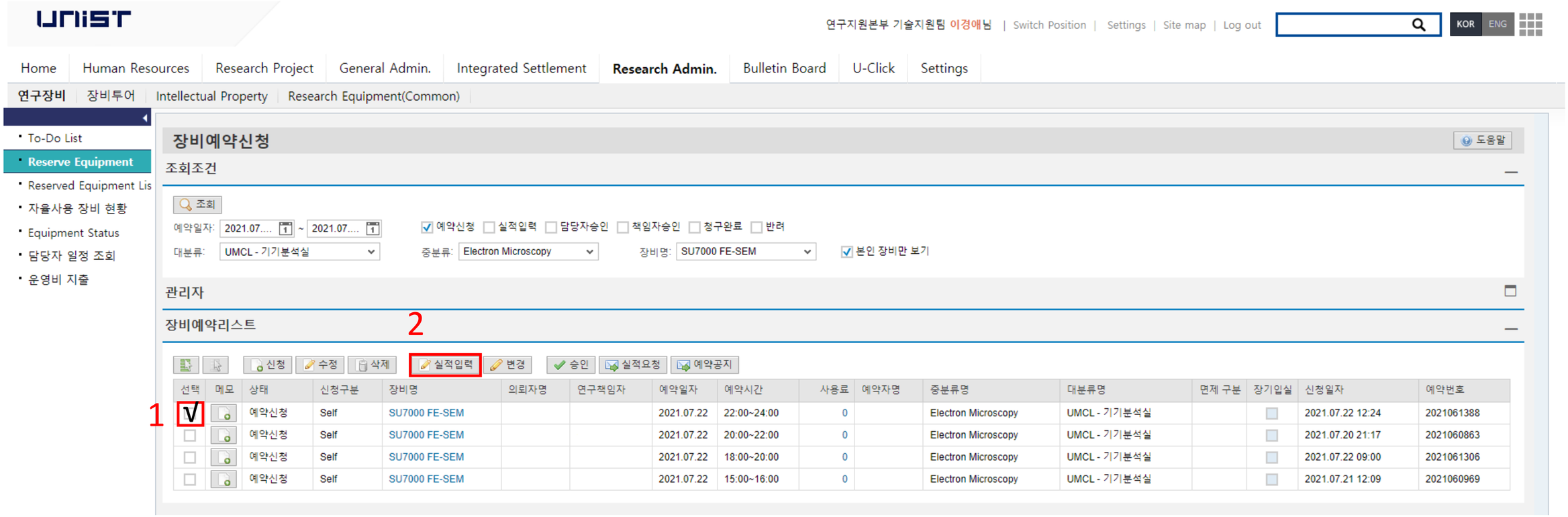
Reservation cancel

portal.unist.ac.kr – Research Equipment – Equipment reservation/input result



Input result

After measurement, you have to input result instead of filling in log sheet



1. Select the reservation
2. Click the [Input result]
3. Check the information and click [Save]

Guideline of the operation of the UCRF UMCL

Article 1. Access

① A person who wishes to be authorized access to UMCL must obtain approval from the manager in charge after submitting an application form to “Access Permissions Application” of the “Participation Space” menu on the UCRF homepage (http://ucrf.unist.ac.kr).

② A person who wishes to enter UMCL without access authority shall be accompanied by the equipment manager or shall obtain approval for access from the equipment manager.

③ Each user must enter (exit) individually using the pass card with their own identity, and it is prohibited to enter using another person’s pass or to enter together without permission.

④ A person who uses UMCL at night (PM18-AM09 on the next day) or on holiday must enter after establishing personal safety and protection rules (accompanying 2 or more people, preparing for an emergency contact, etc.) for lab accidents.

⑤ A person who violates above paragraph ①~④ regarding access to UMCL shall be obligated to compensate for all safety and property damage caused by the violation.

Article 2. Use of laboratory space

① A UMCL user must understand and follow the common safety rules for the laboratory (Attachment 1).

Guideline of the operation of the UCRF UMCL

② A UMCL user on weekends or holidays must fill out the laboratory daily checklist (Attachment 2) provided in each laboratory room, and must submit it to equipment manager, with the signature of the professor(or supervisor) within 3 days from the date of use.
③ A UMCL user should clean up the area after using the equipment, and must promptly notify the equipment manager if there is any problem with the equipment or environment. Violation of the notification obligation may result in restrictions on the use of the equipment.

Article 3. Use of Equipment
① A person who wishes to use the equipment of UMCL must make a reservation and use the equipment after completing the training of the equipment manager, assessment test, and acquiring self-user qualification. (Analysis or process request is irrelevant to equipment training and qualification evaluation.)
② A user who completes the regular or occasional training (including practice) by the equipment manager can receive practical training from the senior student of his or her laboratory to improve proficiency before the assessment test. The qualification of the senior must be at least 1 year of experience (more than 5 times in the previous 6 months) in using the equipment. The laboratory is responsible for all safety and property issues arising from the practical training conducted by the senior student.

Guideline of the operation of the UCRF UMCL

③ If there is no record of equipment used in the last 90 days, the qualification for self-use ends. A person who wants to reacquire the self-user qualification must receive equipment training and pass the assessment test conducted by the equipment manager. The upgrading for self-user can be made on the website of UCRF. (http://ucrf.unist.ac.kr)
④ Equipment reservations or requests can be made on the UNIST Portal System and the website of UCRF, and the reservation time should not be unnecessarily occupied for a long time so it does not affect the opportunity for others.
⑤ A user should be familiar with the safety rules for each equipment before using, and use the equipment after wearing appropriate safety gear.
⑥ Bringing chemicals and other items for personal use should be consulted with the equipment manager.
⑦ A user must check that there is no problem before using equipment, chemicals, and etc. If there is a problem before or after use, a user should promptly report to the equipment manager.
⑧ A user must thoroughly fill out the “Performance Records” after using the equipment.
⑨ A user must take safety measures by posting the contents of the experiment to deliver accurate information to others if he inevitably leaves his seat during the experiment.
⑩ The laboratory(in case of outsiders, affiliated institution) of self-user shall be obligated to compensate for all safety and property damage such as accidents (equipment damage, breakdowns, losses, etc.) caused by the negligence of the self-user.

Guideline of the operation of the UCRF UMCL

Article 4. Cancellation after equipment reservation

① A self-user can cancel equipment reservation by himself up to 2 hours before equipment reservation time. However, TEM (HR-TEM, FE-TEM, Normal TEM, Bio-TEM) can be canceled up to 4 hours before. (※ Cancellation is not possible after the cancellation deadline has passed.)

② If a user has reserved an analysis request but wants to cancel it, he must notify the equipment manager at least 24 hours in advance. (However, in the case of NMR, notification would be allowed up to 1 hour left.) A user who does not appear at the reservation time without prior notice will be charged a usage fee for the reservation time.

Article 5. Laboratory safety and user management

① A person who harms the safety of the laboratory or violates the rules of using the UMCL and damages the equipment use of another person, can be subject to penalties according to [Attached Table 1] and be taken appropriate action.

② If the violation of the rules is deliberately determined, the sanctions may be strengthened. If a user voluntarily declares after violating the rules, the sanctions may be eased.

③ If it is judged that the sanctions are difficult to be properly implemented due to the status of the violator (graduation or resignation), the user may be blocked from accessing UMCL.

Guideline of the operation of the UCRF UMCL

④ A person who damages the property and facilities of UMCL by violating the rules shall be held liable to compensate for the damage.
⑤ If a violator does not follow the sanctions, the advisor of the violator will be held jointly responsible. (In the case of an outsider, the supervisor in charge of the affiliated institution)

Self-user Penalty

1. Criteria for penalty points

① A user shall be fined the penalty points in the event that any inappropriate behavior falls under the following. Each penalty item may be counted multiple times. (The expiration of penalty points is 1 year from the date of imposition.)

No.	Case description	Penalty points
1	Equipment is used by an unpermitted user who does not have a qualification of self-user.	5
2	**Equipment is used without reservation**(Including excessive use of equipment without additional reservation	**3**
3	A user operates an unpermitted function of the equipment	3
4	An abnormality or malfunction discovered before or after using the equipment did not immediately reported to the manager	3
5	Damage, malfunction, and loss of equipment due to user negligence (* The user is responsible for all costs incurred due to the negligence. *)	5
6	A user does not provide the accurate information of the sample to the equipment manager, which is essential for protecting the safety, equipment or facilities	3
7	A person causes the leakage of a harmful substance, gas, or any risk of fire	5
8	**A person uses or occupies the public items and other person's items without prior consent**	**1**
9	A person leaves the laboratory without switching off the lights, locking the doors, **tidying up the area**, etc. after using equipment	**1**
10	A person does not follow the common lab safety rules(including **clothing**, prohibition of eating, etc.)	**1**

Self-user Penalty

2. Actions taken after subjecting penalty points

① If the cumulative penalty points exceed a certain criterion, the action corresponding with the table below will be taken.

② When a user violates the rule, an official letter from UCRF is sent to the department or institution(in case of the user outside) of the violator.

③ Even if the following actions have been taken due to the sum of the penalty points, the penalty points within the validity period(1 year) are effective.

Type	Penalty points	Action
Total penalty points awarded to 1 user	5 points or more	- The user is prohibited from using the equipment for 1 month. - When the prohibition period is finished, the user must complete the training and assessment again.
	8 points or more	- The user is prohibited from using the equipment for 3 months. - When the prohibition period is finished, the user must complete the training and assessment again.
Total penalty points awarded to 1 laboratory for 1 equipment	12 points or more	- Notify the user and the advisor(supervisor) by e-mail that "If the penalty score is more than 15 points, the members of your laboratory are prohibited from using the equipment for 1 month."
	15 points or more	- The members of the laboratory are prohibited from using the equipment for 1 month - Send an official letter of action to the affiliated department(institution)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2009

THANK YOU