

Nano FE-SEM User Manual(Eng.)

울산과학기술원 연구지원본부 기기분석실 UNIST Central Research Facilities UNIST Materials Characterization Lab

**CONTACT** 

**Ulsan National Institute of Science and Technology** 

**Address** 50 UNIST-gil, Ulju-gun, Ulsan, 44919, Korea **Tel.** +82 52 217 0114 **Web.** www.unist.ac.kr

**UCRF** 

Bldg.102 201-5

Tel. +82 52 217 4163

Web. http://ucrf.unist.ac.kr

# Index

1. L	_ab safety	03
2. l	JCRF SEM Feature	04
3. S	SEM operation	05
1	l) What to prepare for SEM imaging	05
2	2) Specimen preparation	06
3	3) Starting instrument	07
4	l) Coating	8
5	5) Loading the specimen stub	09
6	6) High voltage & Spot size	10
7	7) Beam on	11
8	B) Finding the sample	12
9	9) Focus & Stigmator	13
1	0) Link Z to FWD	14
1	11) Taking photo	15

17
18
19
20
21
22
25
32
33
34
41
46

# **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** 

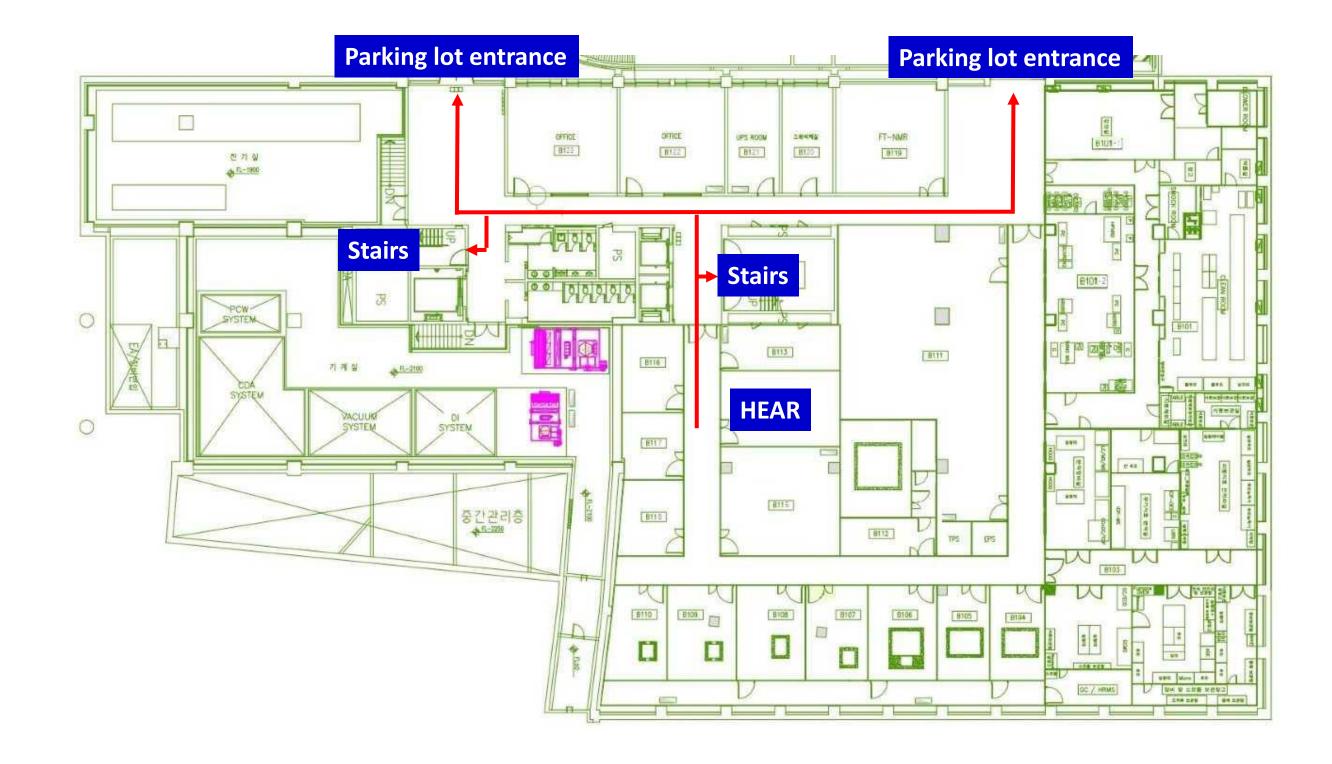
Wear Foot Protection No Open-Toe Shoes

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%)
SU8220 cold FE-SEM		0	Slight	X	X	Busy	2013	40,710won /30min
SU7000 FE-SEM	0		Serious	X	X	Busy	2021	40,710won /30min
Cold FE-SEM	0	0	Slight	X	X	Not busy	2011	31,320won /30min
Nano230 FE-SEM			Slight	X	X	Not busy	2009	26,100won /30min
Quanta200 FE-SEM	X	Δ	Slight	0	0	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

Туре	Α	В	С	D		
Equipment	SU8220 Co SU7000 Cold F	FE-SEM	Nano FE-SEM Quanta FE-SEM			
Usage	General	Cross section	General	Cross section		
Picture (Front)						
Picture (Back)						

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

SEM stub Production

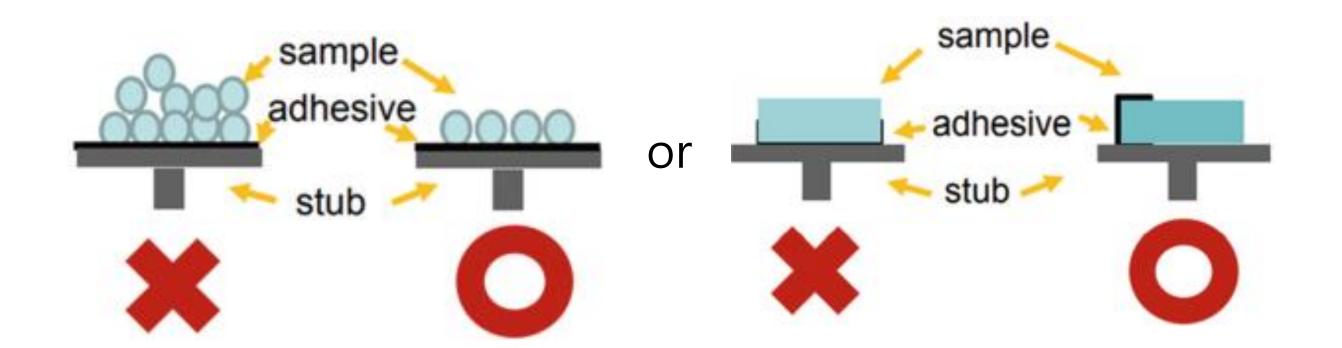
- UDMC Cha Jae-hoon, Jeong Woohyun

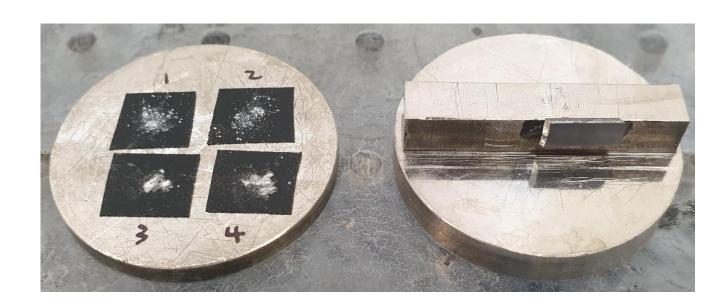
3. Conductive double-sided carbon tape for SEM





# **Specimen Preparation**

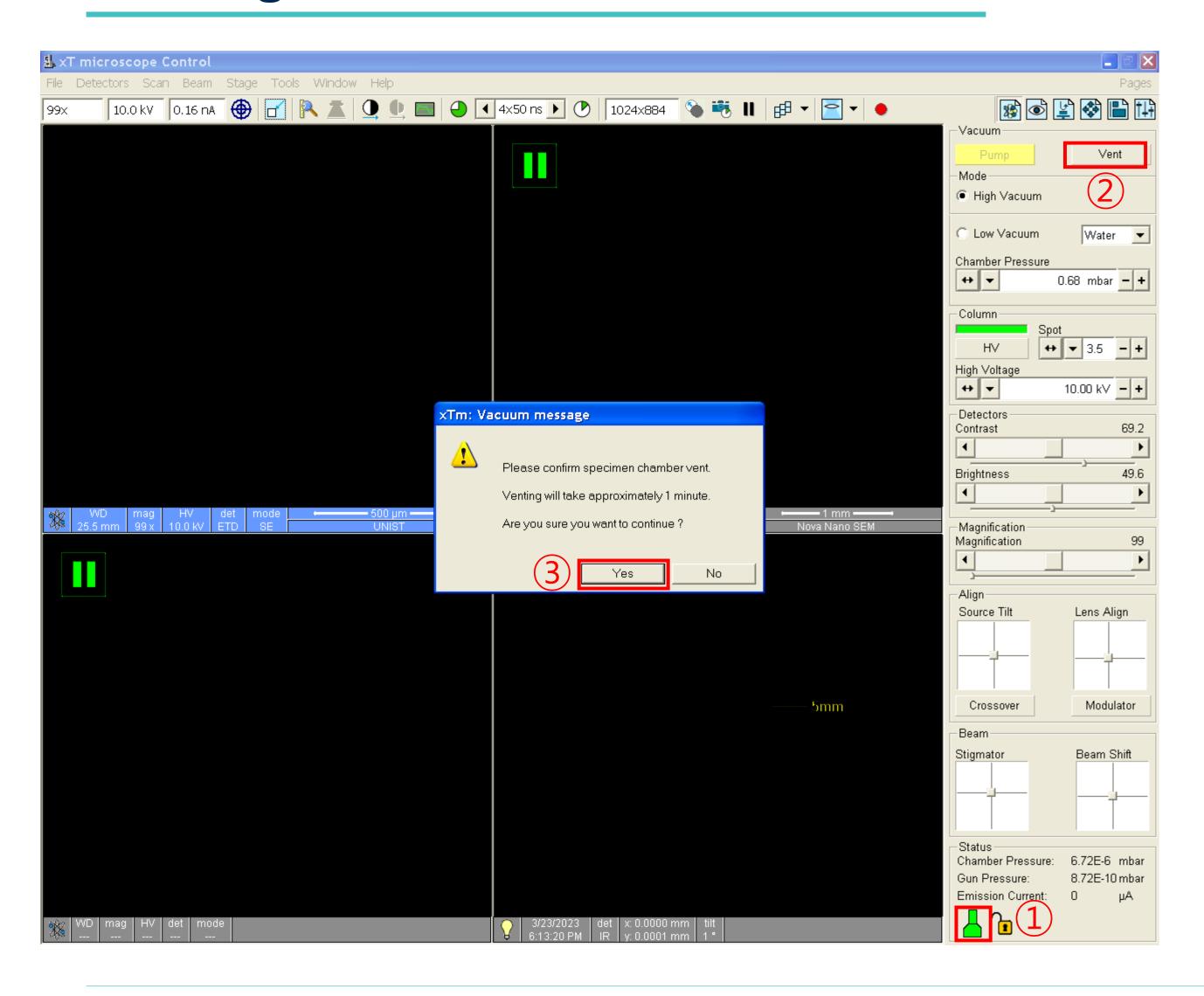




- Sample preparation should be performed by individual laboratories.
- 2. Wear appropriate safety gear.(goggles, gloves, etc.)★
- 3. Prepare completely dried specimens.
- Clean the stub with ethanol, etc.
- Attach conductive double-sided tape to the stub and secure a very small amount of specimen to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



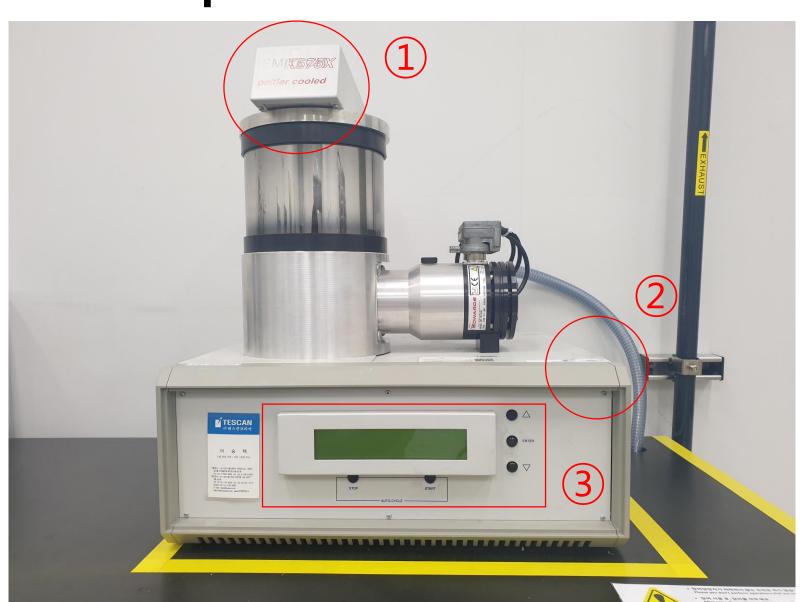
### **Starting Instrument**



- 1. Check the vacuum state.(green)
- 2. Click [Vent].
- 3. Click [Yes].
- 4. Prepare the specimen until chamber part becomes dark gray.

### Coating

### FEI Sputter









**Material: Pt** 

Sputter current: 20mA

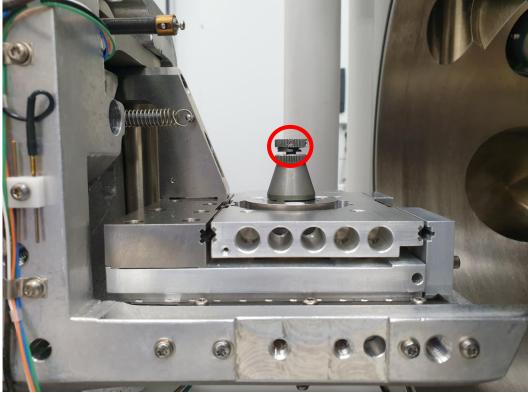
Time: 20s~120s

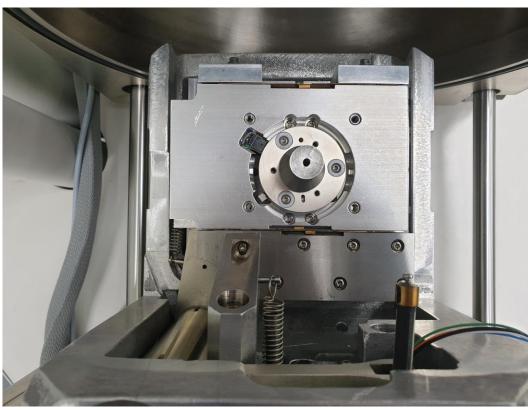
- 1. Open the lid, and turn on the instrument.
- 2. Press [ENTER] to change parameters or press [START] to run.
- 3. Press the lid until the turbo speed reaches 20%.
- 4. When the coating is finished, the first screen is displayed.
- 5. Open the lid, remove the specimen and turn off the instrument.



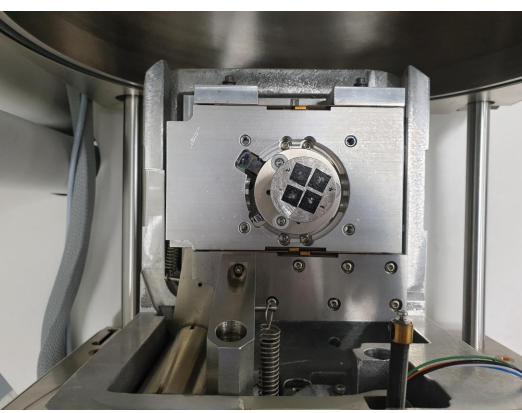
# Loading the Specimen







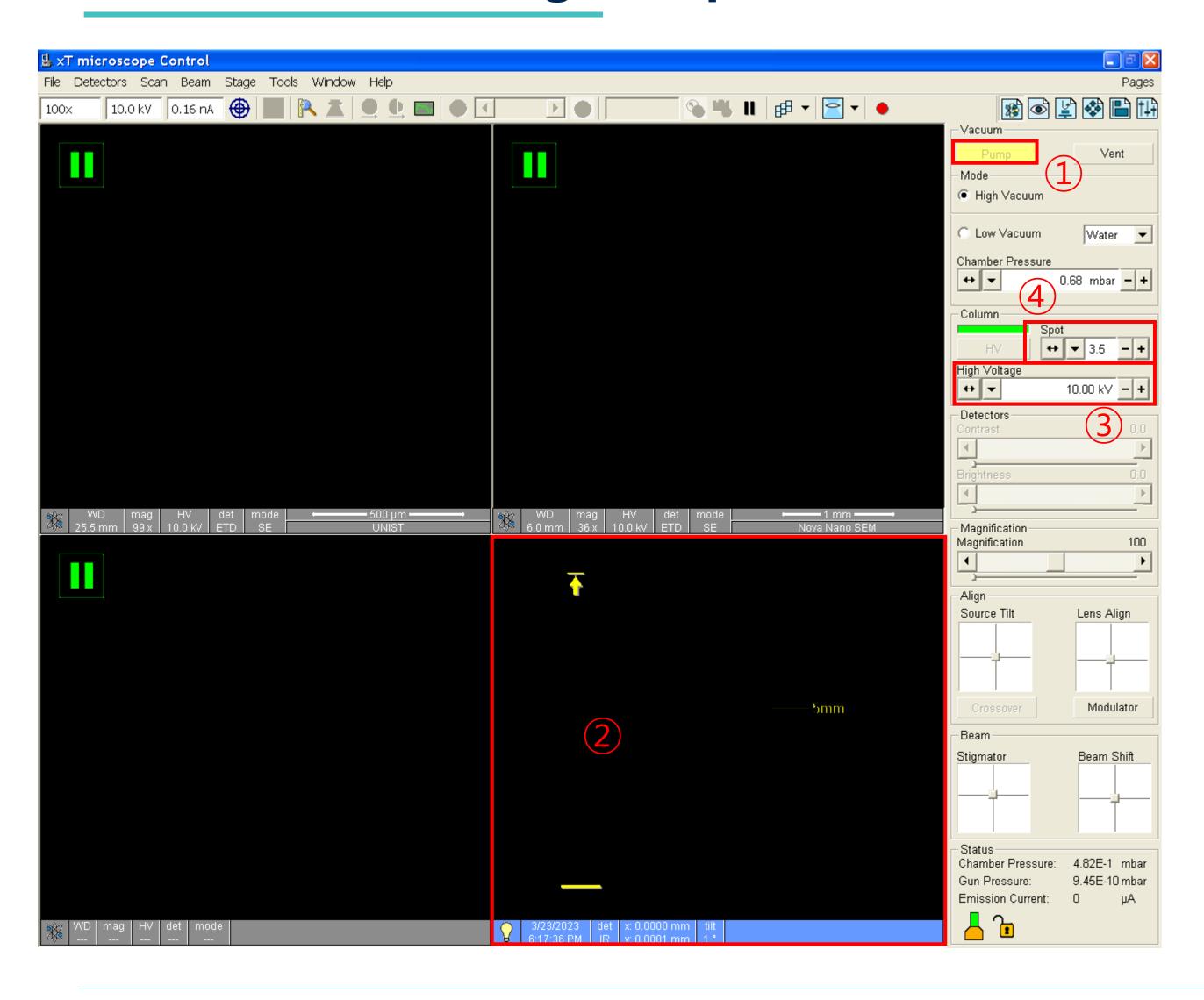




- 1. Pull the handle to open the chamber door.
- 2. Insert the stub into the holder and tighten the screw.

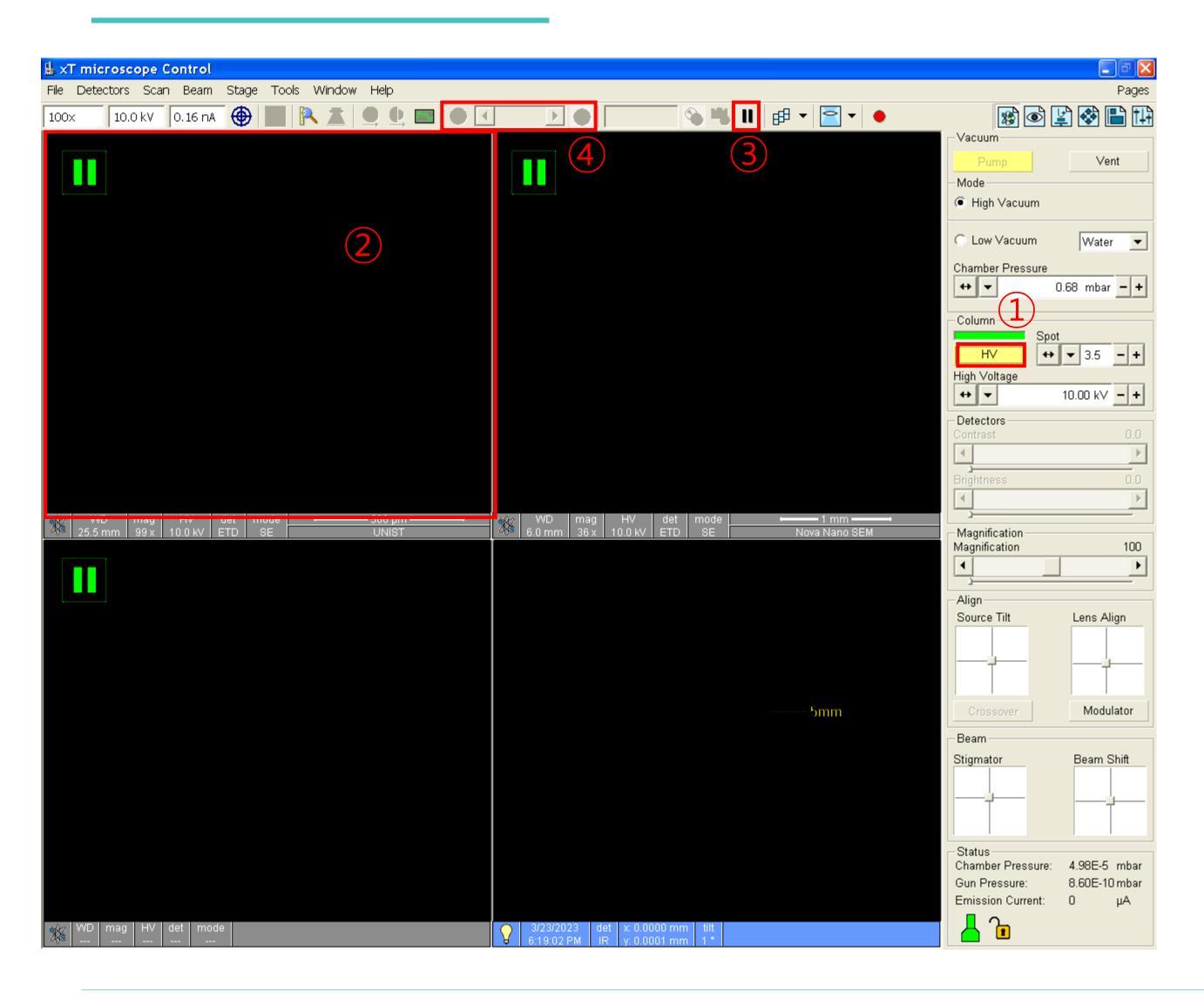


### **Acceleration Voltage & Spot Size**



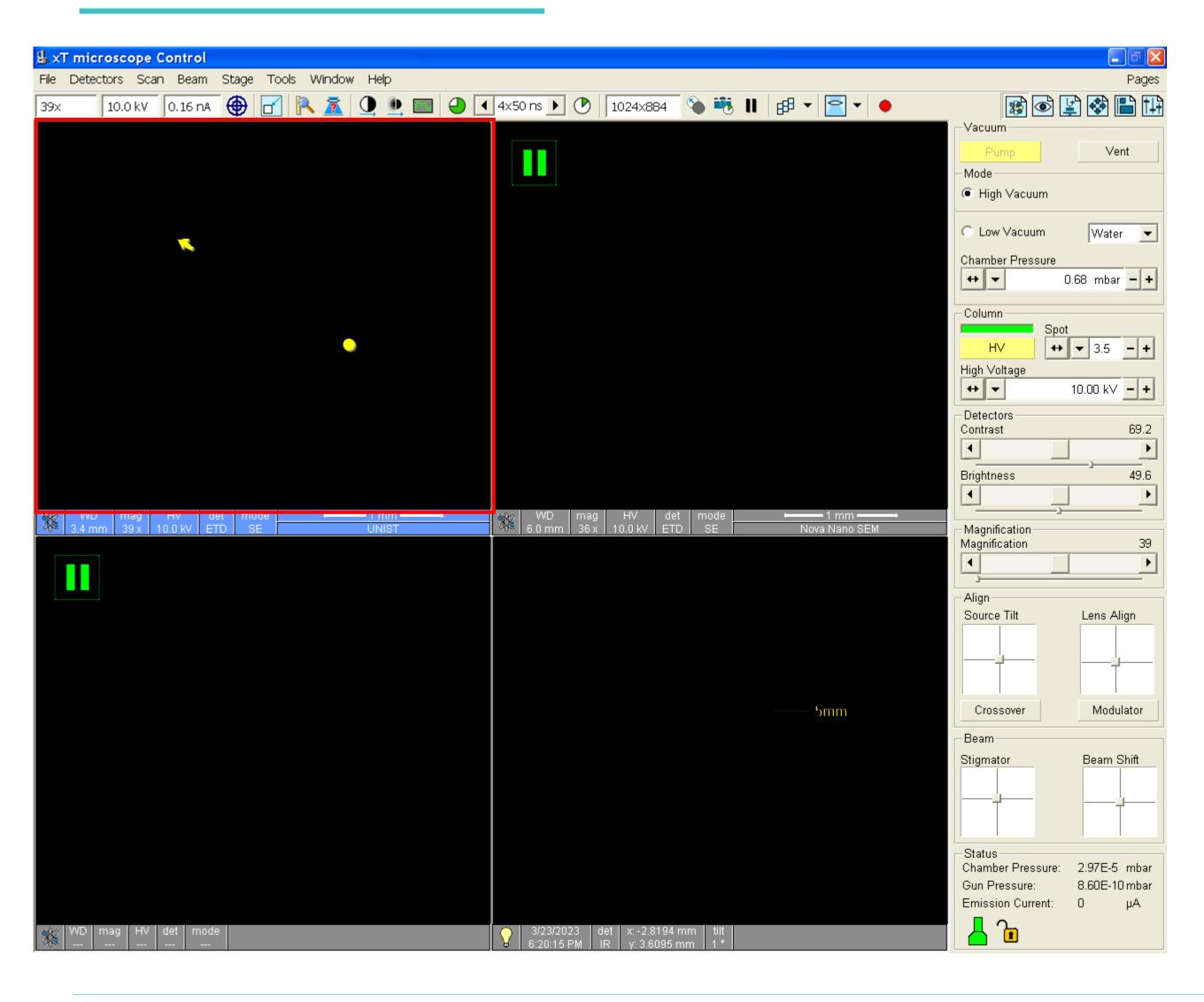
- 1. Click [Pump].
- 2. Click on the 4<sup>th</sup> screen.
- 3. Hold down the mouse scroll and drag up so that the highest part of the specimen touches the 5mm marker.(Proceed while vacuuming)
- 4. Choose [High Voltage].
- 5. Choose [Spot].(3~4)

### Beam on



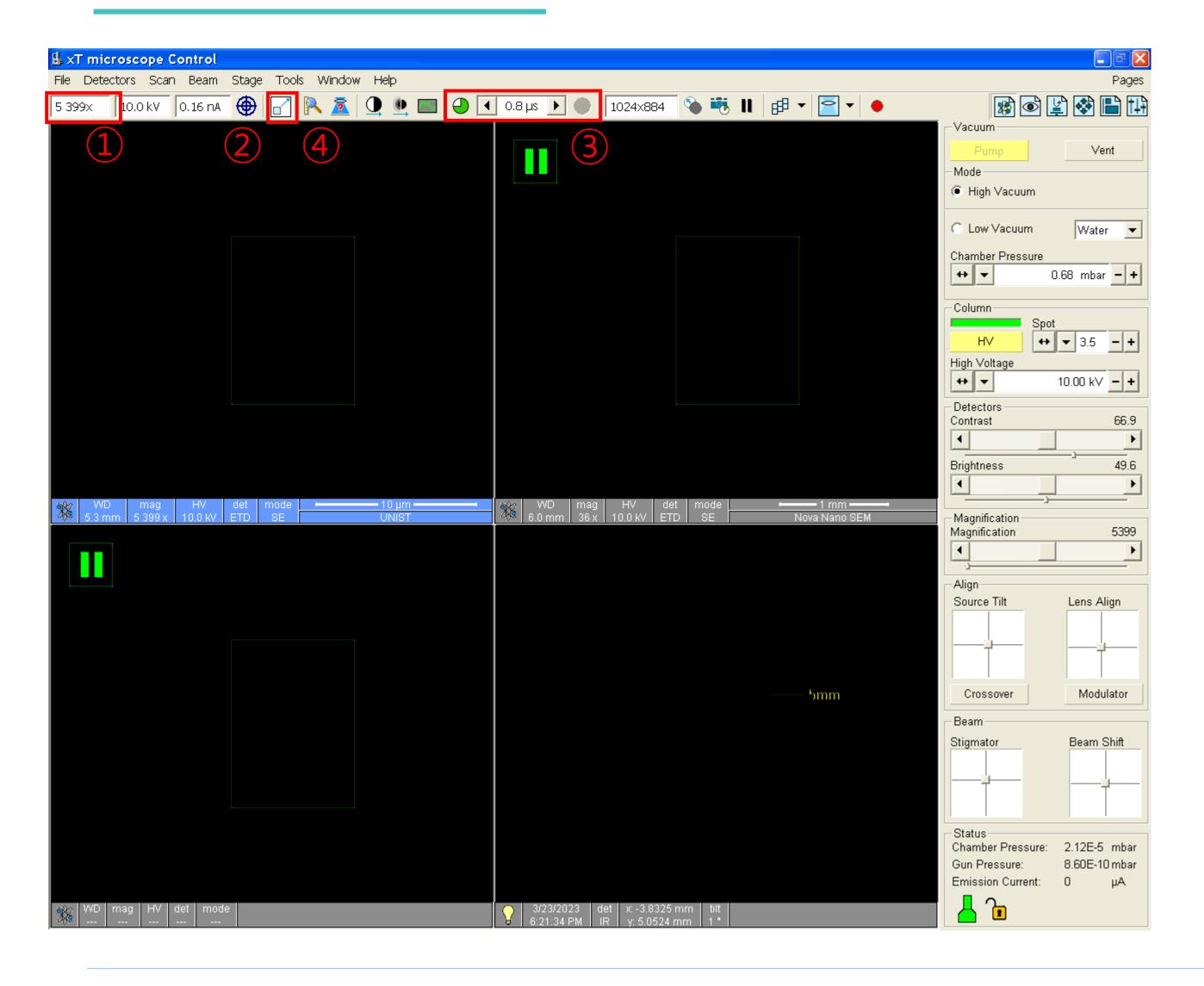
- Click [HV].(Gray → Yellow)
- 2. Click on the 1<sup>st</sup> screen.
- 3. Click [Pause].
- 4. If the screen is too dark or too bright, you can manually adjust the [Brightness] or [Contrast] knob individually. → This process can be performed at any time during image observation.
- 5. Choose the [Dwell Time].(0.8~1 µs)

# Finding the sample



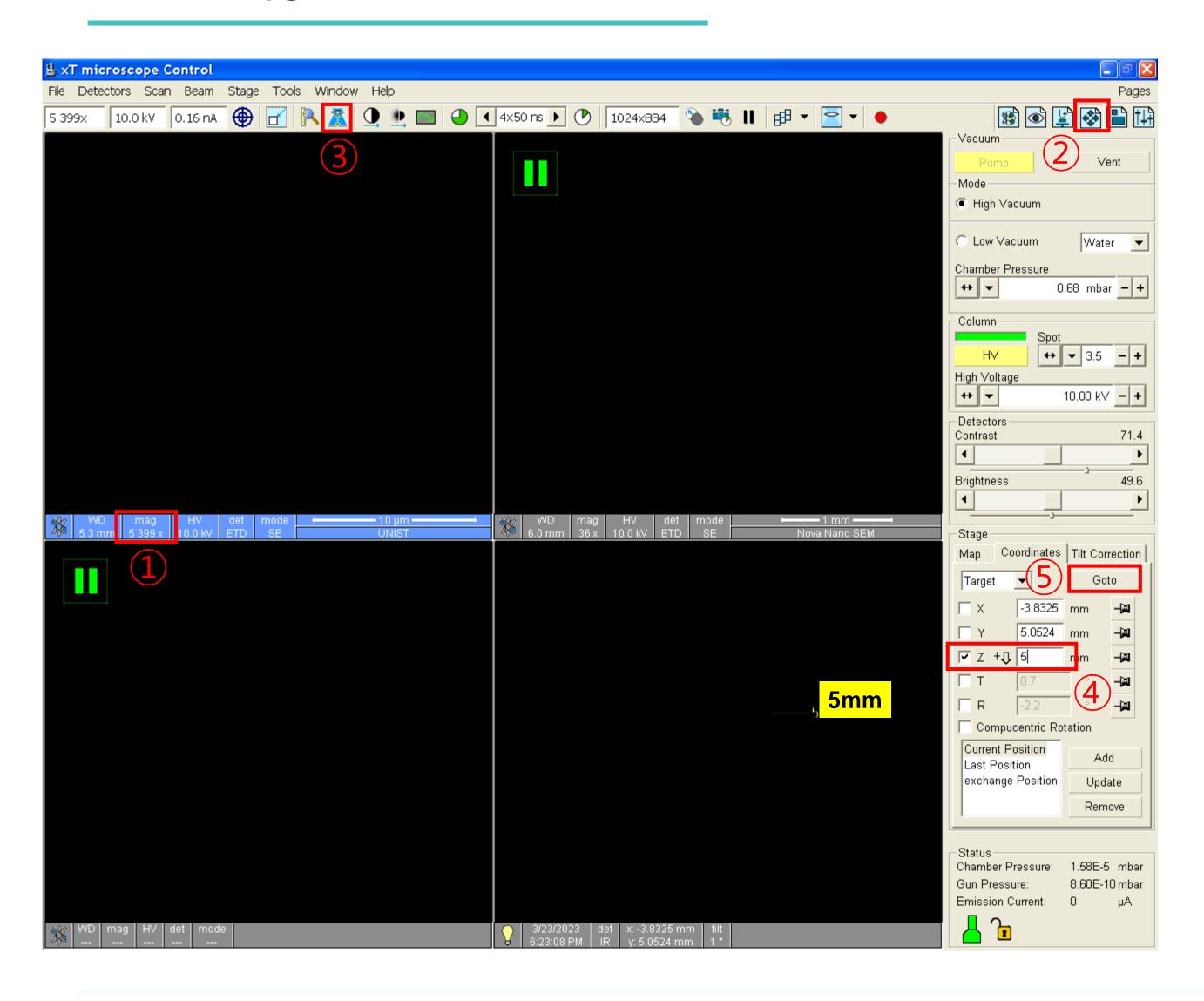
- 1. Minimize magnification and find specimens.
- 2. If the image is blurry, use the [focus] knob to adjust the focus.
- 3. Move the stage to the location you want to observe.
  - How to move the stage position
    - 1) Drag with the mouse scroll pressed
- 2) If you double-click the desired location, that part moves to the center of the screen.

# Focus & Stigmator



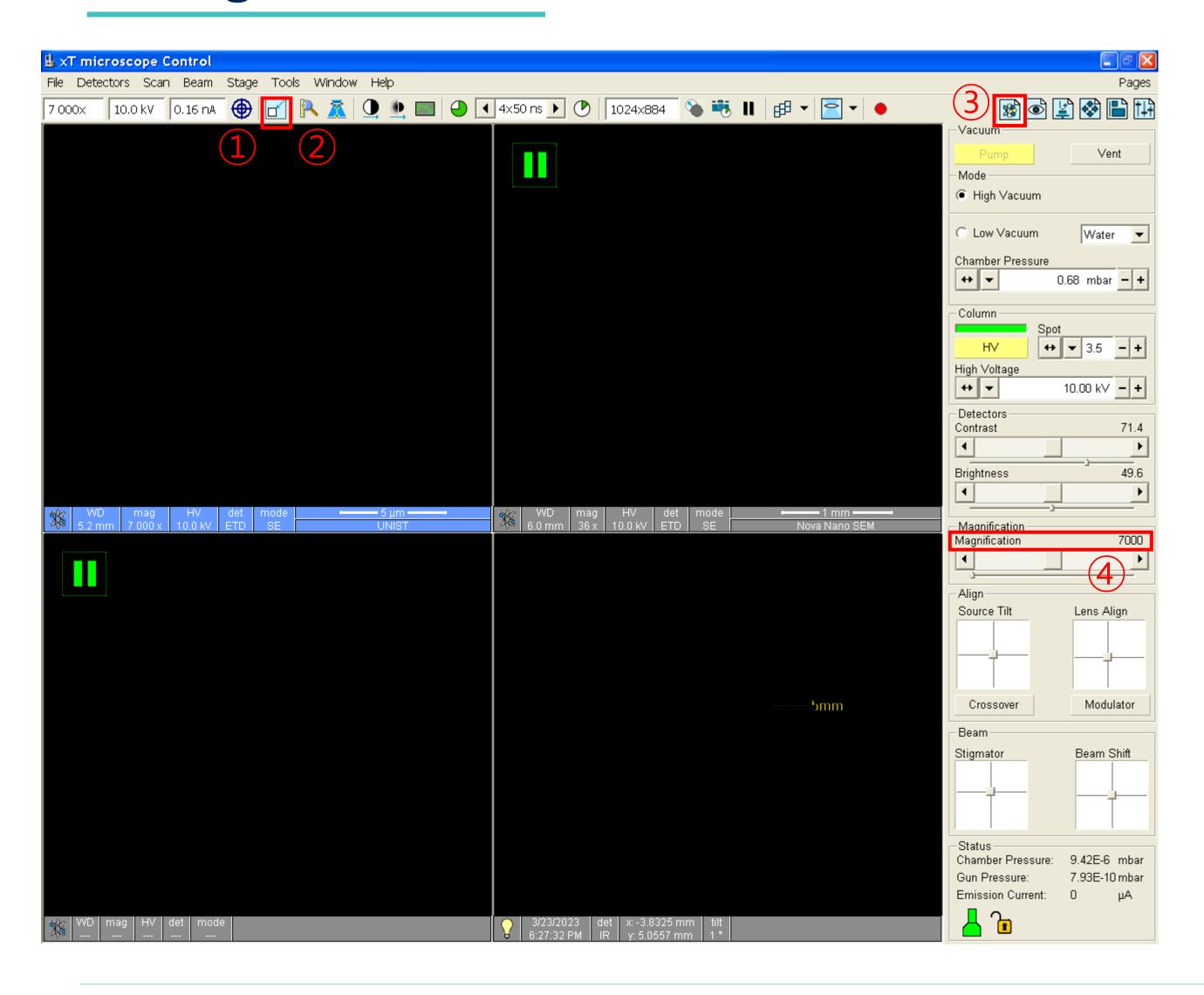
- 1. Use the magnification knob to slowly raise the magnification to 5000 times and adjust the focus in between.(Coarse: fast adjustment, Fine: fine adjustment)
- 2. Click [Reduced Area].
- 3. Check the [Dwell Time].
- 4. Adjust the focus.
- 5. Adjust stigmator X, Y knobs one by one.
- 6. Adjust the focus.
- 7. Click [Reduced Area].

### Link Z to FWD



- 1. Check the WD(5mm±1mm)★
- 2. Click [Navigation].
- 3. Click [Link Z to FWD].
- 4. Put 5mm to [Z].
- 5. Click [Goto].

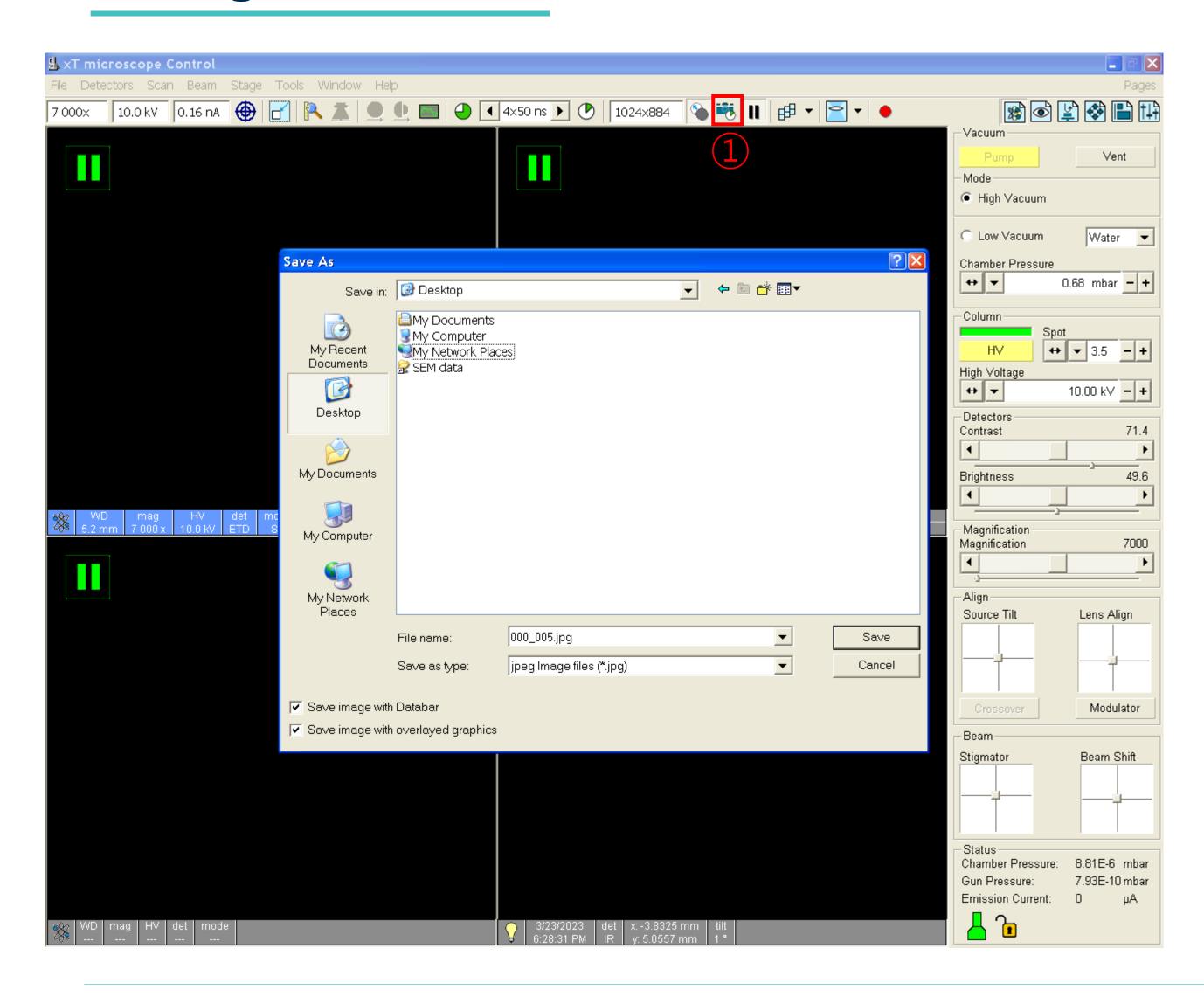
### **Taking Photo**



- 1. Move the stage to the location you want to observe.
- 2. Adjust the magnification slightly higher than the magnification you want to observe.
- 3. Click [Reduced Area].
- 4. Adjust the focus.
- 5. Adjust Stigmator X, Y knobs one by one.
- 6. Adjust the focus.
- 7. Click [Reduced Area].
- 8. Click [Beam Control].
- 9. Double-click the value of [Magnification] and enter the desired magnification.



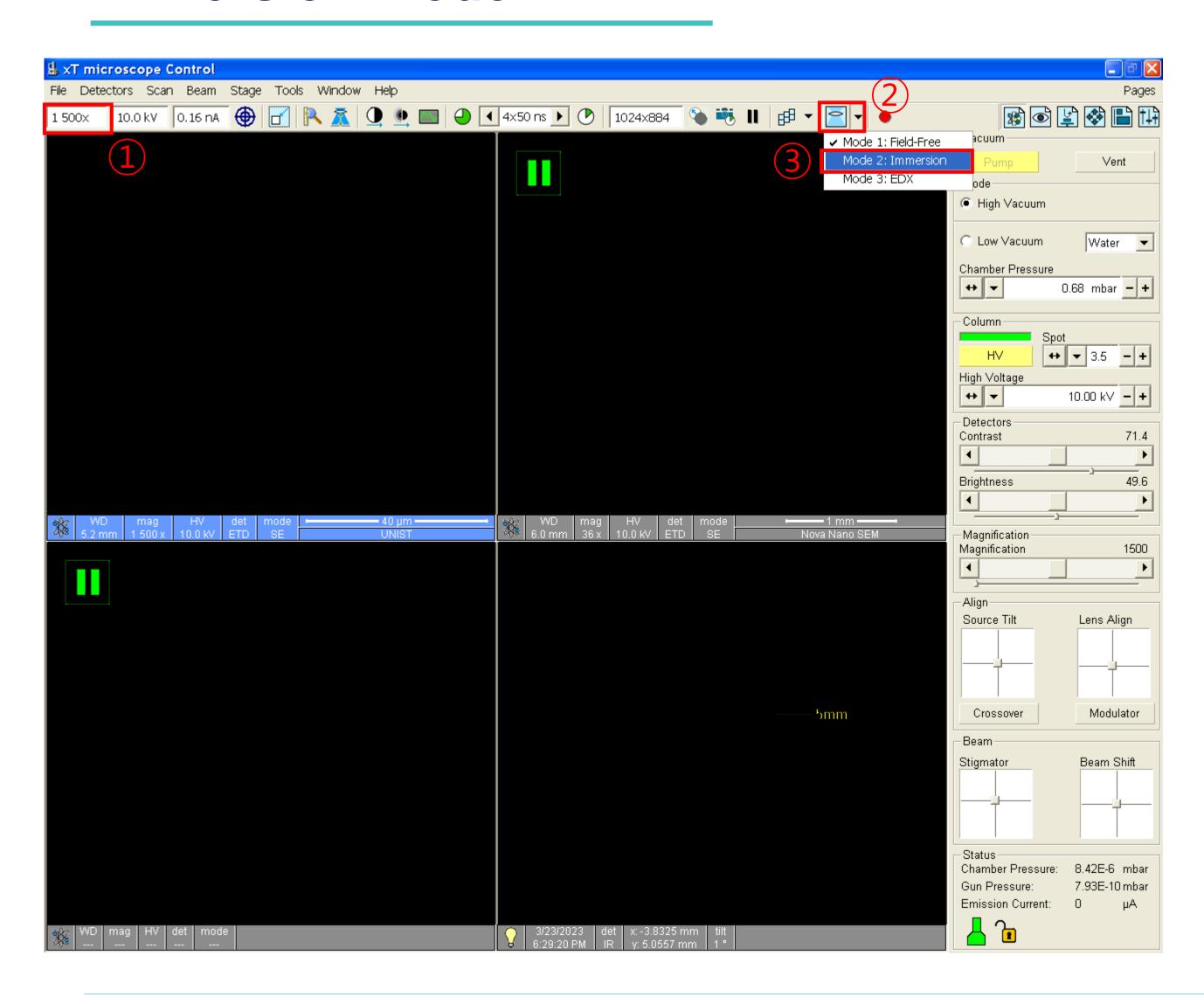
### **Taking Photo**



- 1. Click [Snapshot].
- Select the data storage location.(Desktop-SEM data-Analysis year-Professor folder-Personal folder-Date folder).
- 3. Repeat steps 1 in the previous slide to 2 in this slide.

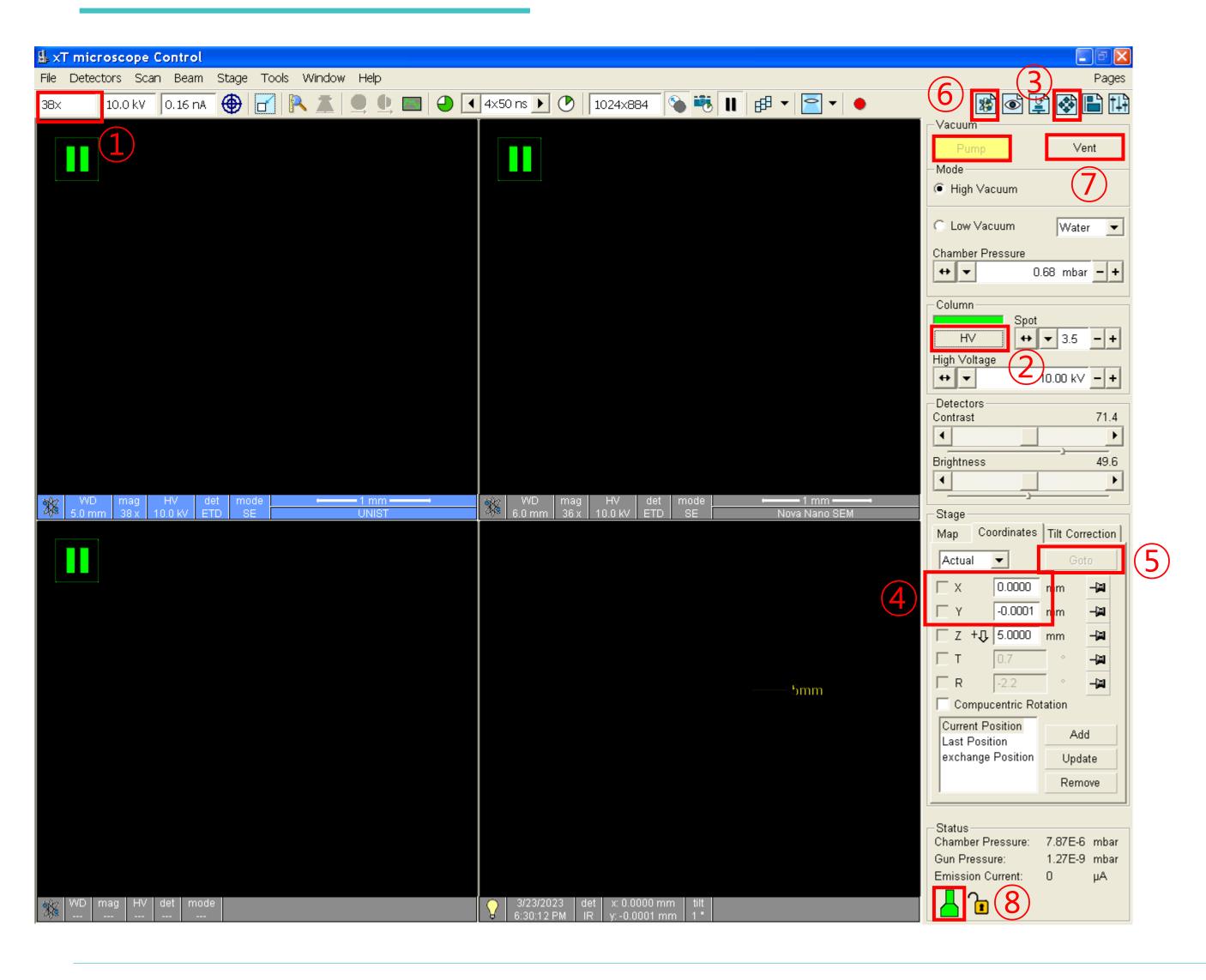


### **Immersion mode**



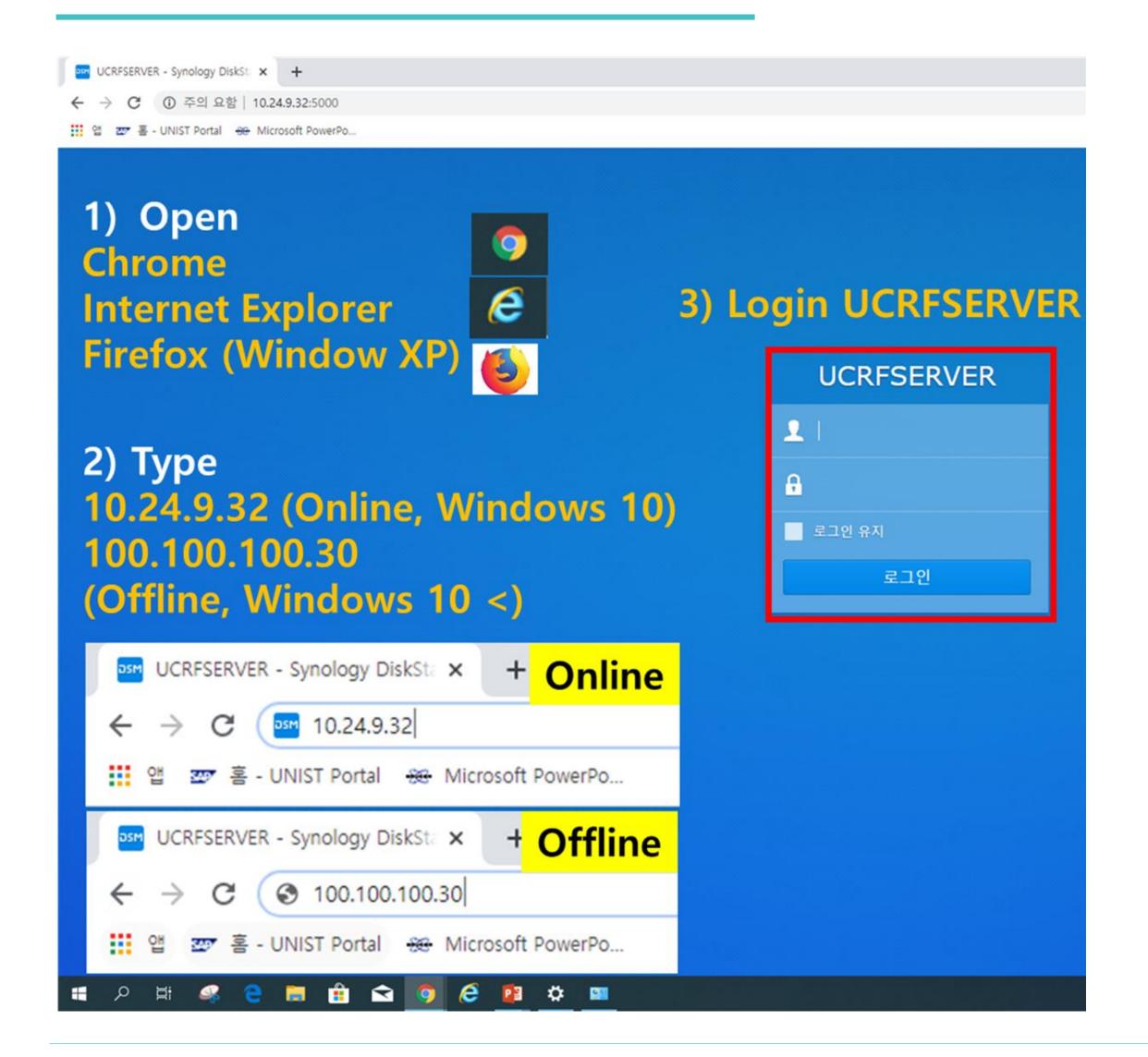
- 1. Adjust the magnification to 1500x or higher.
- 2. Click the down arrow.
- 3. Click [Mode 2: Immersion].
- 4. Properly adjust the contrast and brightness so that you can see the circular beam at the lowest magnification.
- 5. Adjust the focus.

### **Finish**



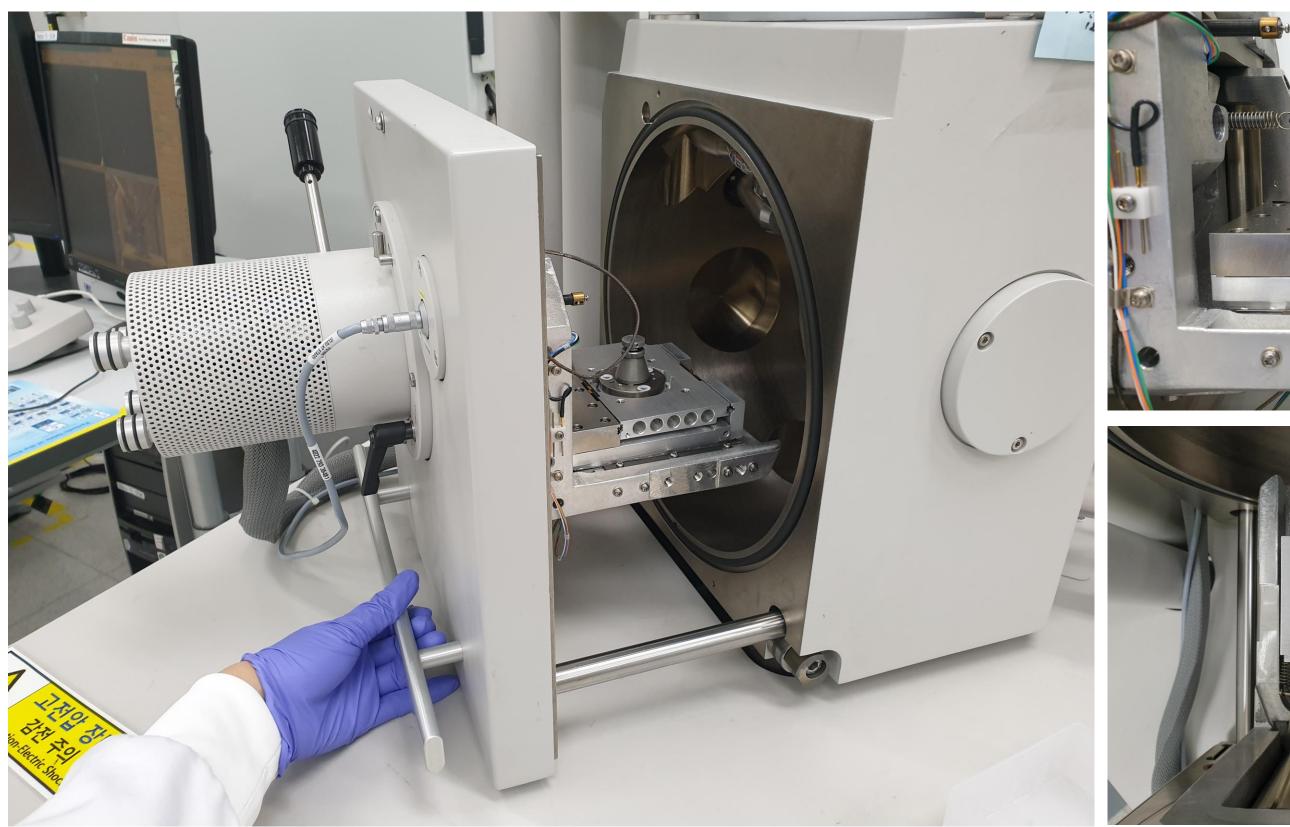
- 1. Minimum magnification at Mode2.
- 2. Minimum magnification at Mode1.
- 3. Click [HV].(Yellow → gray)★
- 4. Click [Navigator].
- 5. Put 0 in [X], [Y].★
- Click [Beam Control] [Vent].
- 7. When the Chamber icon changes from green to dark gray, open the chamber and remove the specimen.
- 8. Click [Pump].

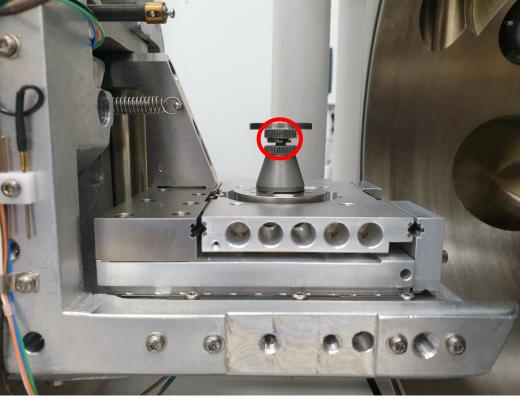
### **Transfer SEM data**

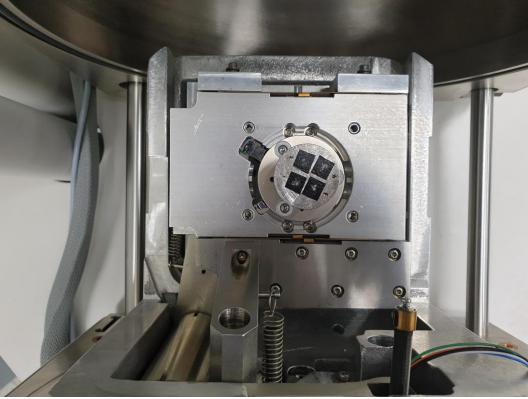


- 1. Do not use USB when transferring SEM data.
- 2. Double-click the web browser.(Chrome, etc..)
- 3. Enter 100.100.100.30. in the address bar.(Enter 10.24.9.32 when downloading from Lab)
- 4. Enter your Lab ID and password.
- 5. Find your professor's folder and create your own folder.
- 6. Drag the SEM data into your own folder.
- 7. Close the window when the move is complete.
  - UCRF server manager: Park Ji-hye(4035)

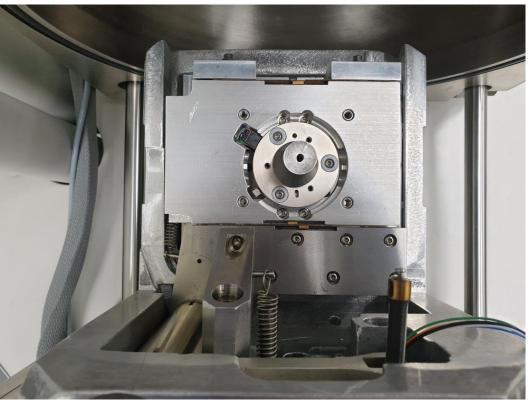
# Unloading the specimen stub











### Daily checklist in lab

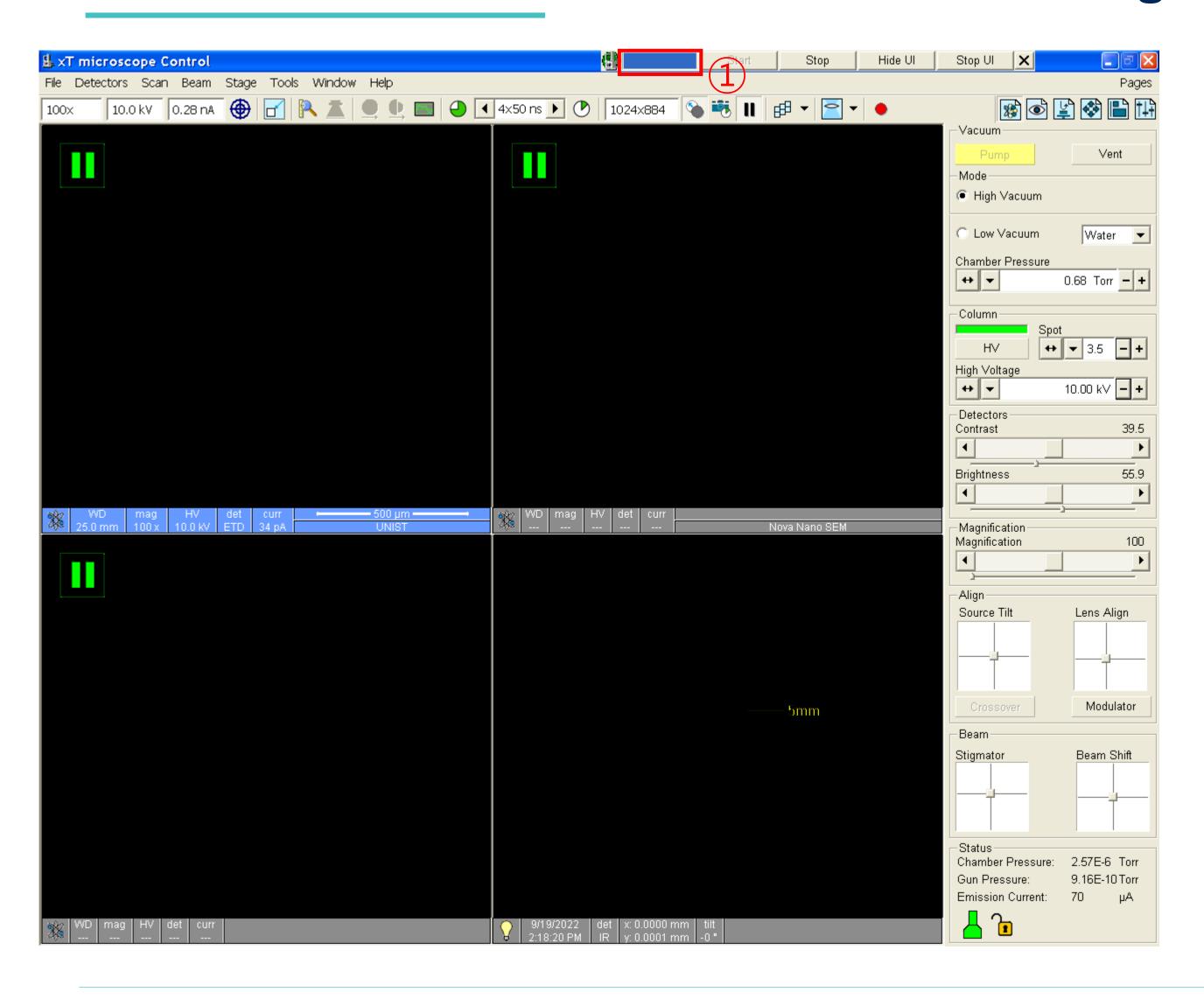
- When using weekends or holidays, check the [Daily checklist in lab] and sign the checker's signature.
- Enter the name of your advisor and submit it in the original place with your signature or stamp.★

#### Daily checklist in lab.

Lab title	Electron Microscopy Preparation	Bldg./NO.	102-B115		Date		
Division	checkpoint						check
	Laboratory overall cleanliness condition.						
	Smoking or bringing food into the lab.						
General Safety	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.						
	Checking the power supply status of unused electrical equipment and checking for overloaded outlets.						
Electric	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 statu	s around elect	tric panelbo	ards.	1		
	Fire extinguish inspection statu		er fire ex	tingu	iisher	and regular	
Fire	Emergency exits, escape routes, and any obstacle blocking the passage.						
	Storage of foreign substances around fire hydrant and fire extinguishers.						
	Outdoor storage of gas containers, no risk of tipping over, and checking ventilation conditions.						
Gas	Corrosion, deformation, nozzle lock status on the exterior of the gas containers and checking the packing time limit of gas containers.						
Gas	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.						
Ch:	Keeping hazardous factors handling and management registers and MSDS.						
Chemical	Categorizing chemicals by description and storing chemical reagents in safety cabinets.						
Equipment check	SU8220 Cold FE-SEM user: Check the pressure of nitrogen gas						
Con	nfirm	Inspector	Signature				(sign)
	Lab Director Signature						(sign)

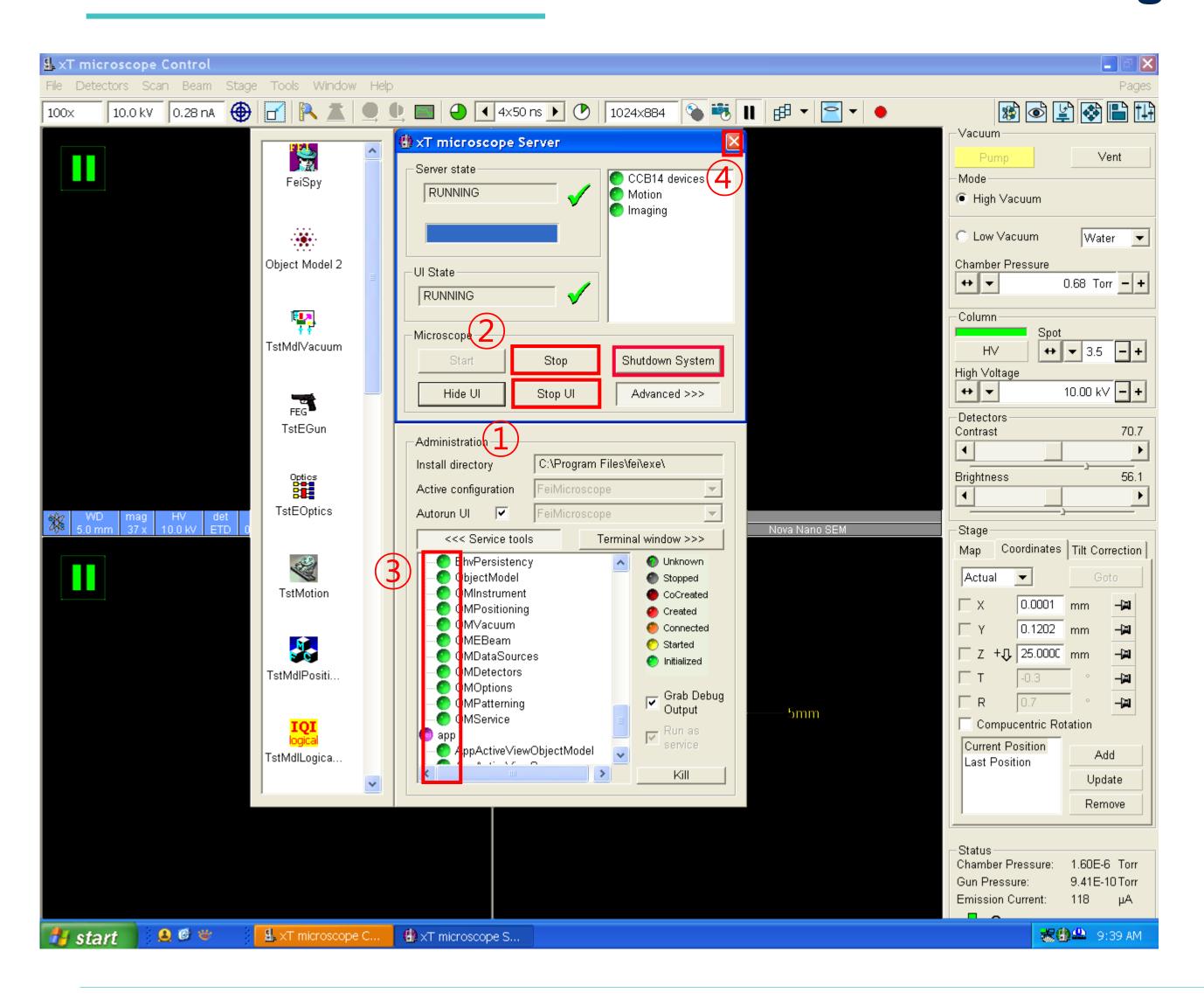


### How to reboot the device when SEM PC lag



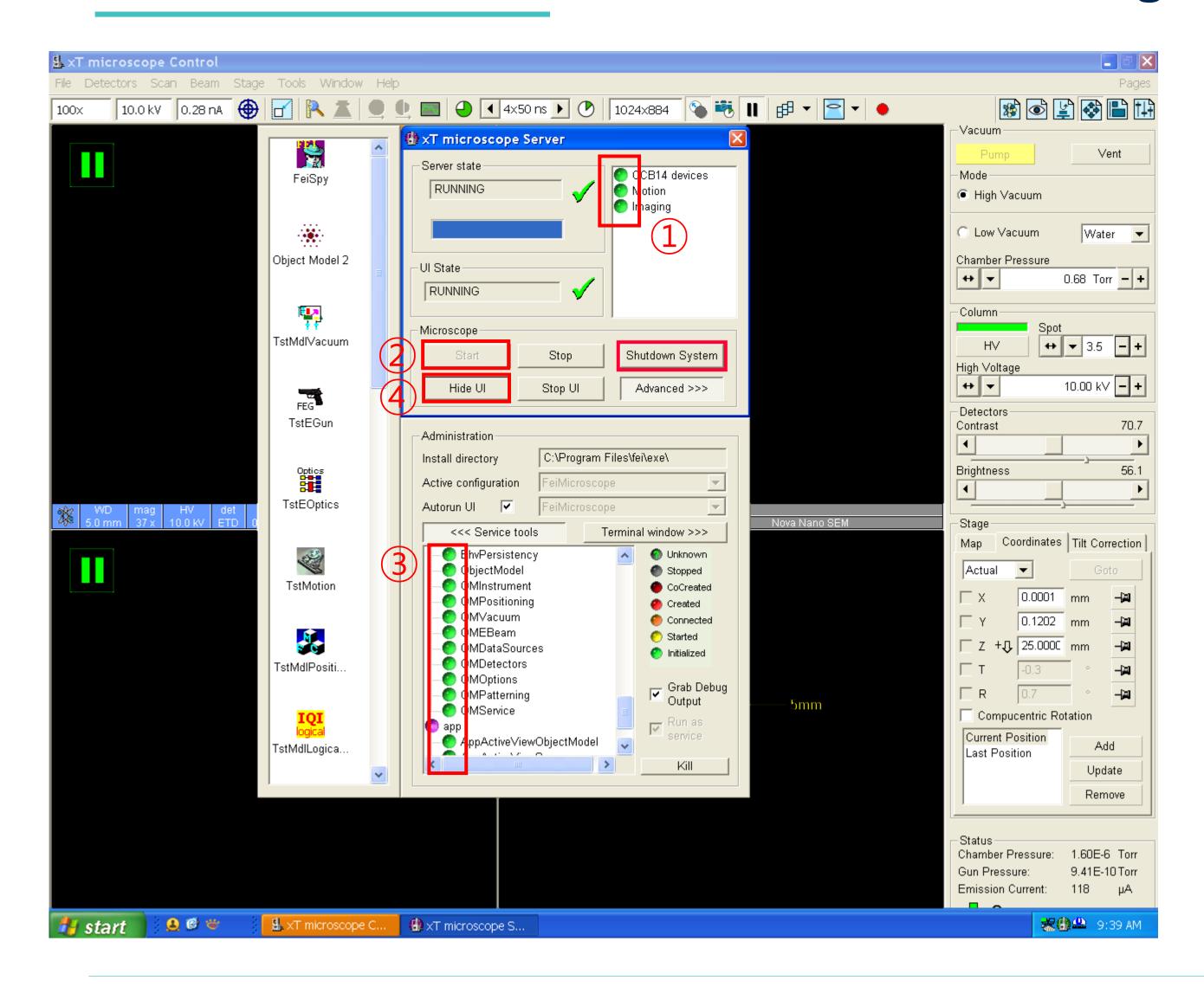
- Beam off
   (If click is not possible, this step is pass.)
- Click the blue part on the small bar (If there is no blue small bar, click [xT microscope Server] on your desktop.)

### How to reboot the device when SEM PC lag



- 1. Click [Stop UI]
- 2. Click [Stop]
- Check the small circles below change from green to dark grey
- 4. Close the window
- 5. Restart SEM PC
- 6. SEM PC ID: support PW: feico95-001
- 7. Click [xT microscope Server]

### How to reboot the device when SEM PC lag

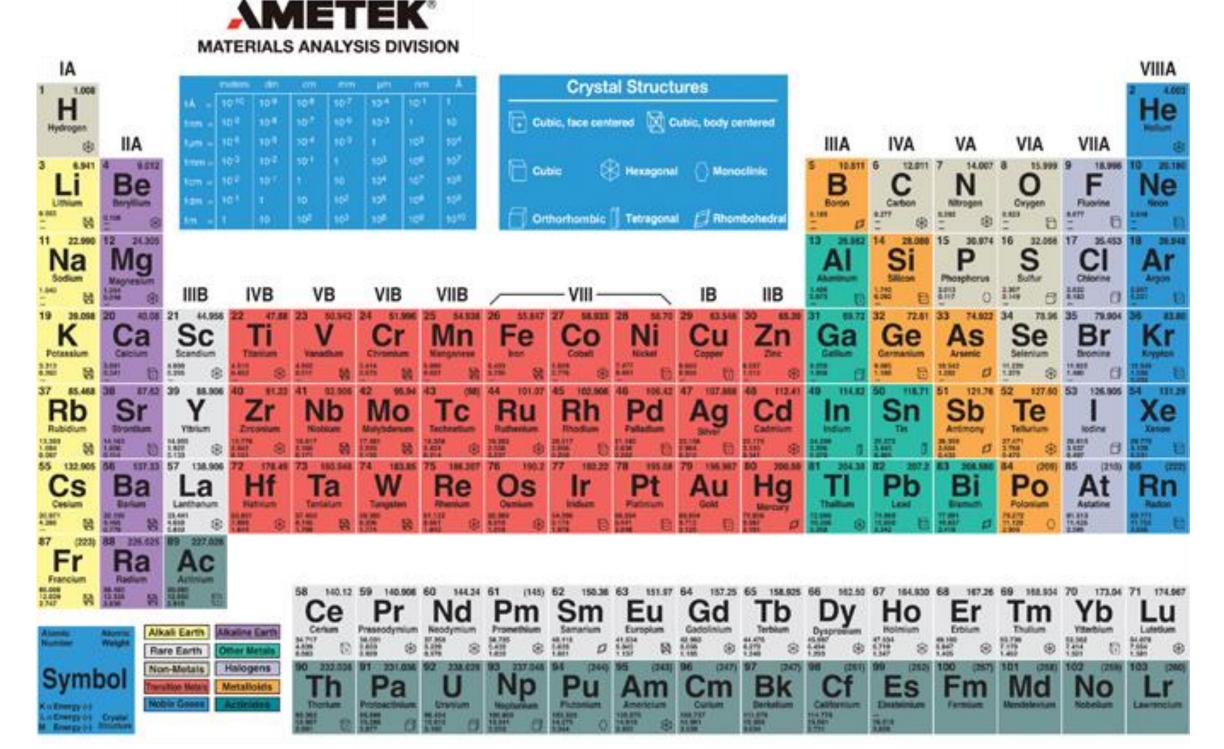


- Make sure three circles in the upper right corner are green
- 2. Click [Start]
- Check the small circles below change from dark grey to green
- 4. Click [Start UI]
- 5. UI ID: supervisor PW: supervisor
- 6. Click [Yes]

### **Energy Dispersive X-ray Spectroscopy**

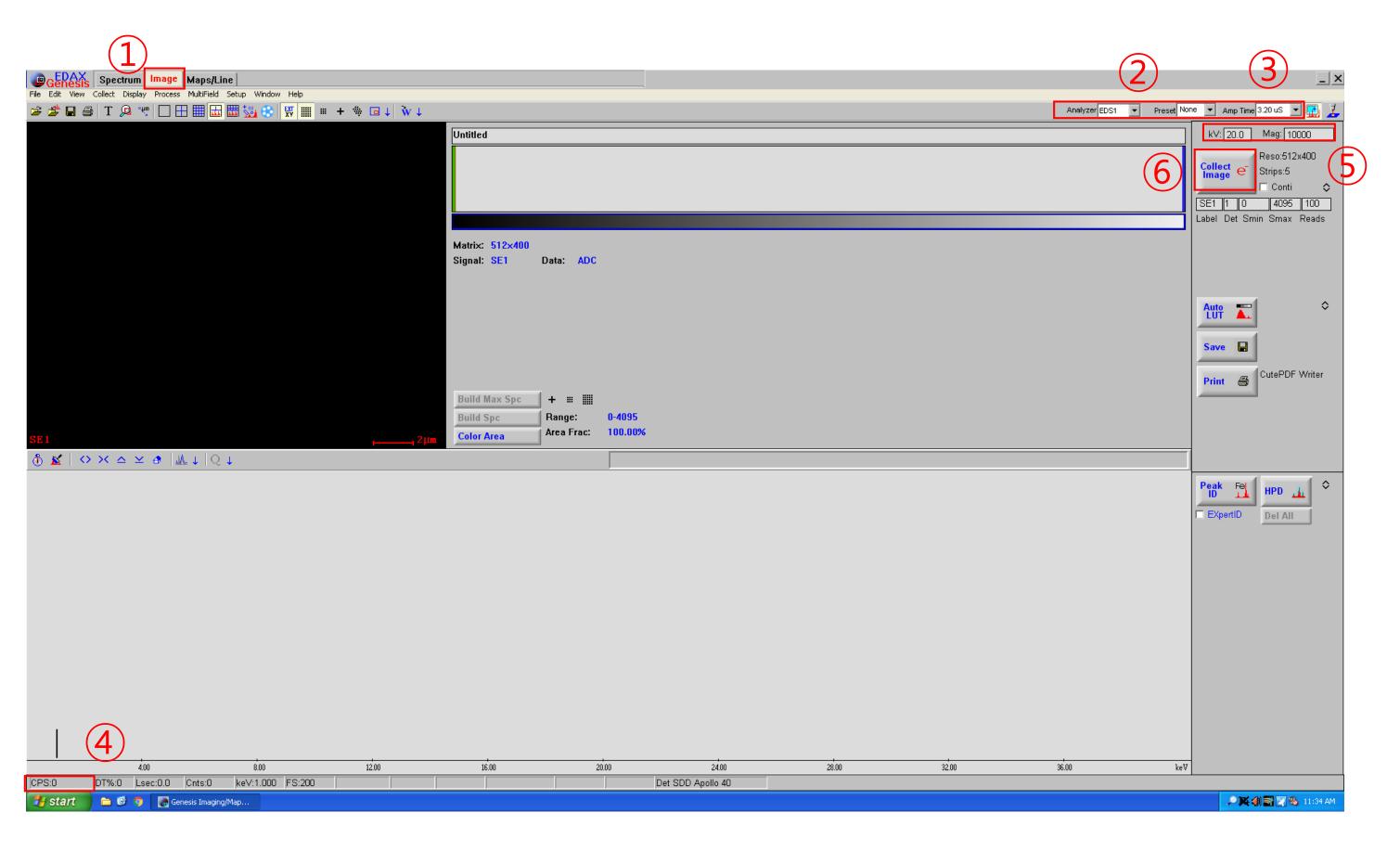


#### www.edax.com



- 1. Press the switch on the back of the Edax controller.(Check the 2 green lights on the front)
- Select an appropriate accelerating voltage.★
   (1.5 times kV of the x-ray value of the element to be observed or unknown specimens 20 kV)
- 3. Select a [Spot size].(4~5).
- 4. Move to the position of the specimen to be measured and adjust the magnification.
- On the fourth screen, press [Pause] to stop the screen.★
- 6. Click [Scan]-[External] on the first screen.
- 7. Double-click [Genesis] in Edax PC.

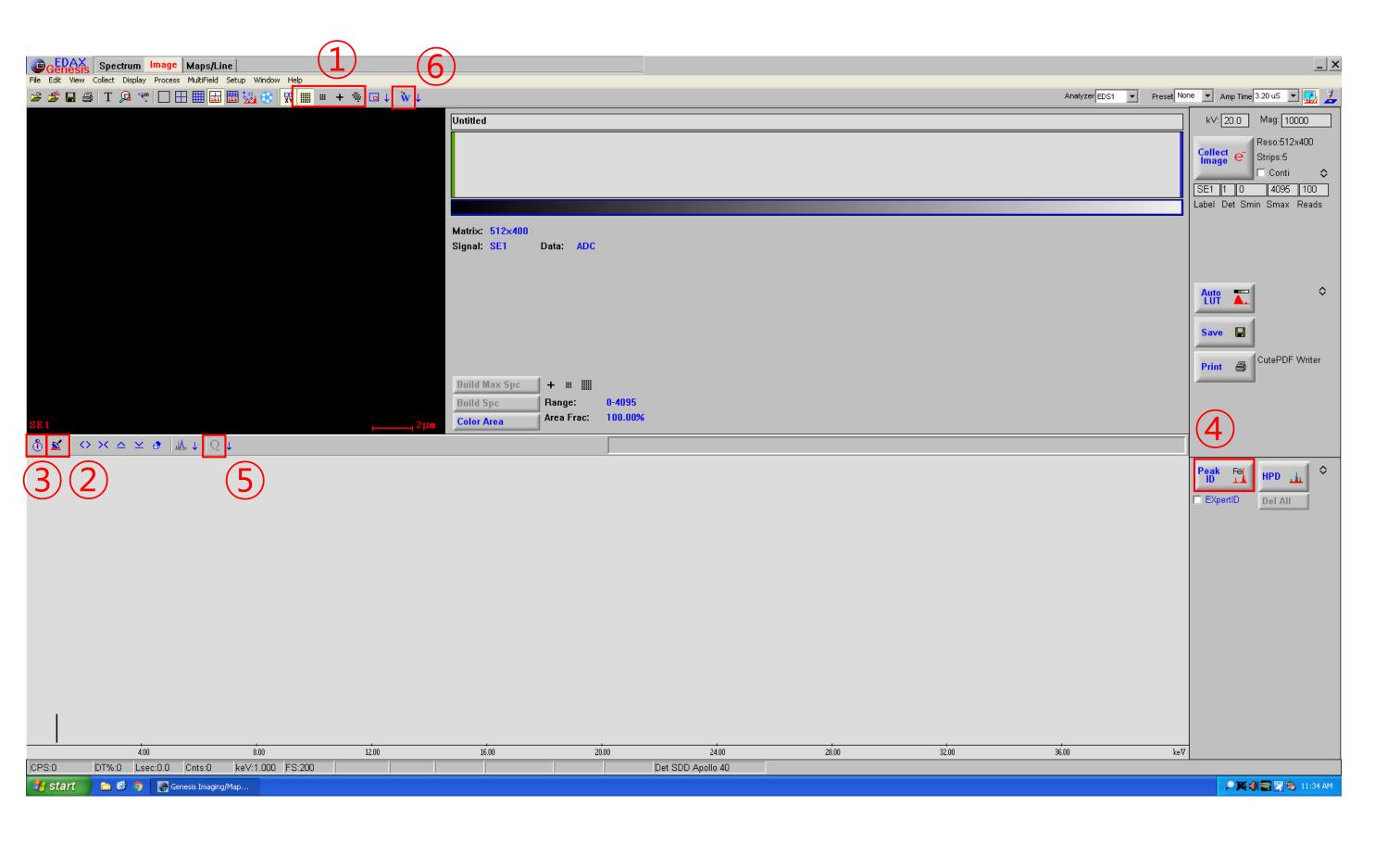
# **EDS Spectrum Analysis**



- 1. Click [Image]
- 2. [Analyzer] select EDS1.
- 3. Select the time you want to measure in [Preset]. (None continues to be analyzed until [Collect] is pressed again.)
- 4. Adjust [Amp Time] to 3-6.
- 5. Check if the CPS value is properly displayed.
- 6. Input the current acceleration voltage and magnification.
- 7. Click [Collect e]

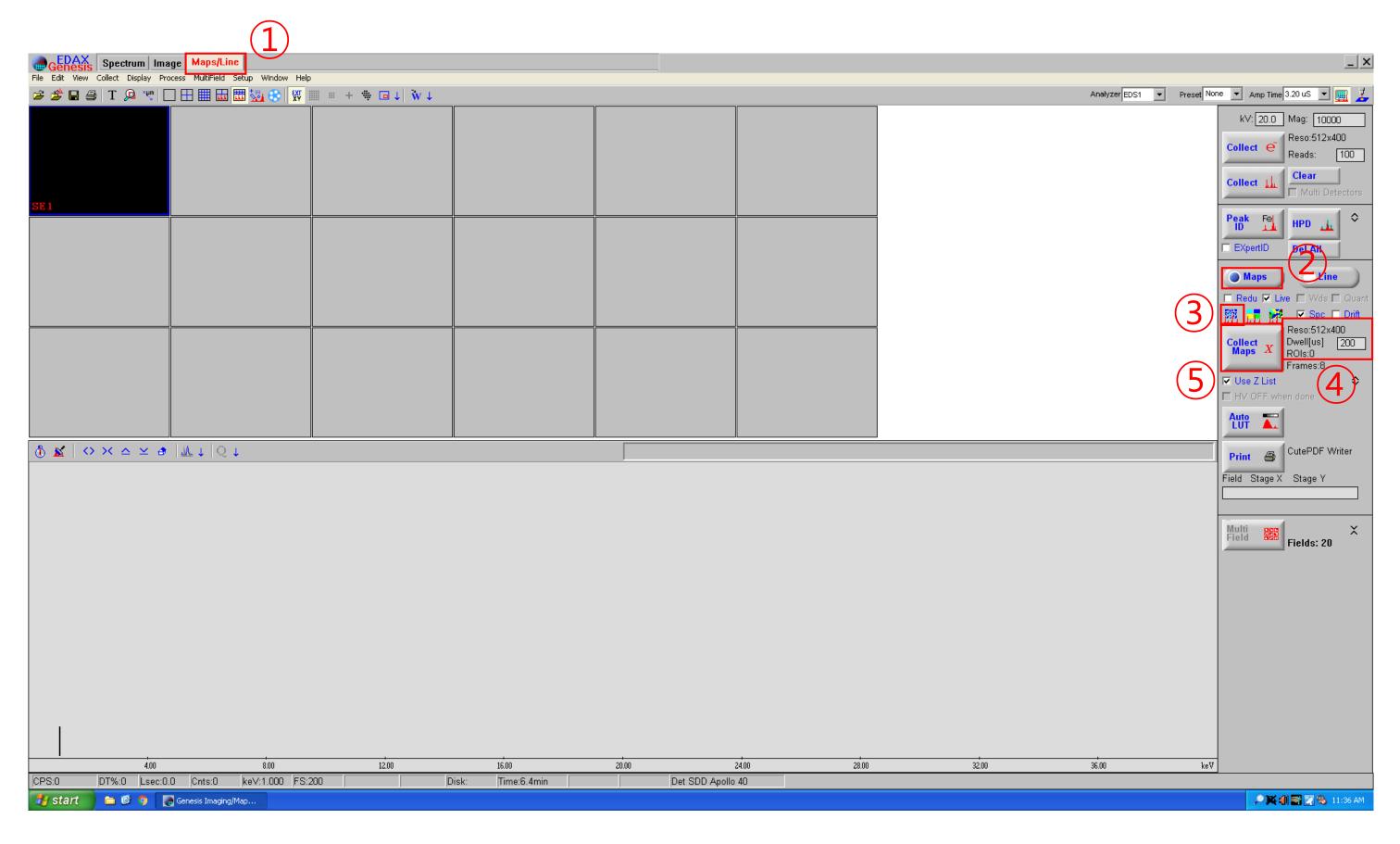


# **EDS Spectrum Analysis**



- 1. Select the area you want to measure.
- 2. Clear the existing spectrum.
- 3. Click [Collect]
- 4. Click [Peak ID]
- 5. Click [Q] [W] [W].

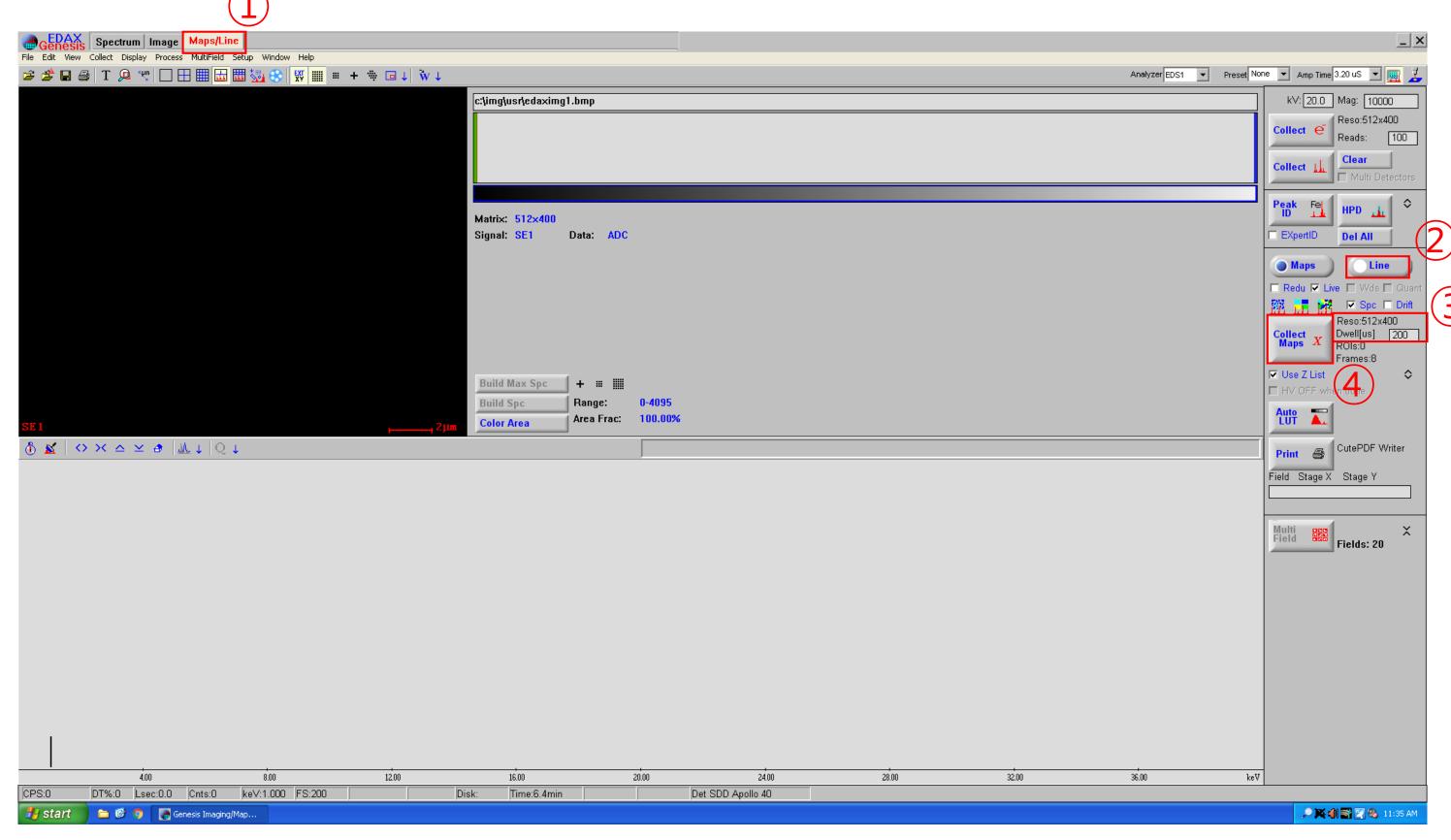
# **EDS Mapping Analysis**



- 1. Click [Maps/Lines]
- 2. Repeat the spectrum analysis if you want to check which elements are present in the specimen.
- 3. Click [Maps]
- 4. Select the map type
- 5. Click right select a matrix size, dwell and the data type
- 6. Click [Collect Maps X]
- 7. A dialogue box will open to select a file name and location to save the maps [Desktop]-[EDS Data]-[Professor folder]-

[Individual folder]-[Date folder]

# **EDS Line Analysis**



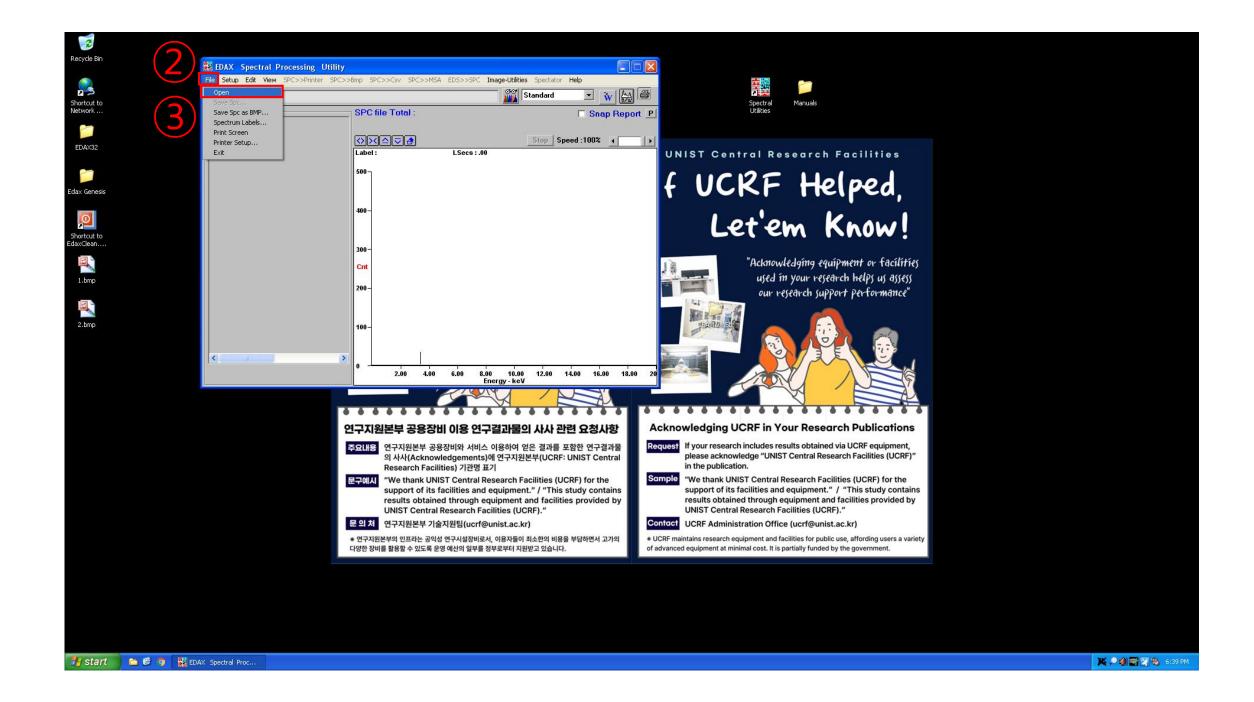
- 1. Click [Maps/Lines]
- 2. Repeat the spectrum analysis if you want to check which elements are present in the specimen.
- 33. Click [Line]
  - 4. Select the line type
- 5. Click right select a matrix size, dwell and the data type
- 6. Click [Collect Maps X]
- A dialogue box will open to select a file name and location to save the maps [Desktop]-[EDS Data]-[Professor folder]-

[Individual folder]-[Date folder]

### **Extracting EDS Data**



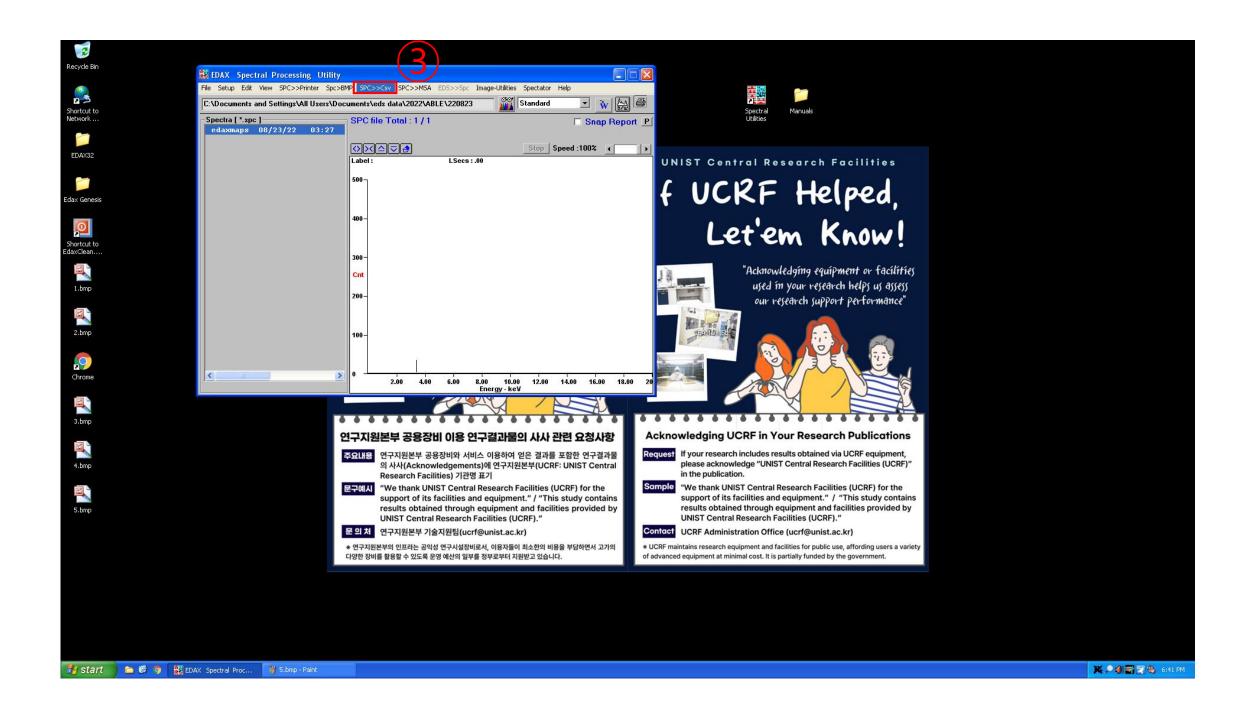
- 1. Double click [Spectral Utilities]
- 2. Click [File]-[Open]



### **Extracting EDS Data**



- 1. Find your spc data
- 2. Click [Open]3. Click [Spc>Csv]



# **Energy Dispersive X-ray Spectroscopy**

- EDS error
  - 이미지가 안 불러와질 경우
    - 1. 책상 위 매뉴얼에 있는 순서대로 진행
    - 2. SEM, EDS 컴퓨터 재부팅
  - Spectrum 이상
    - 1. CCD 화면 일시 정지인지 확인
    - 2. External 상태인지 확인
  - Collection problem
    - 1. Please proceed in the order in the manual on the table \* (Image collection problem)
    - 2. Please reboot both computers(SEM, EDS)
  - Spectrum problem
    - 1. Please verify that the CCD screen is paused.
    - 2. Please verify 'scan-external' or not.

X If the problem cannot be resolved, please contact the person Select and RENAME: Genesis.ini to Genesis.old in charge.

Image Collection problem Subject:

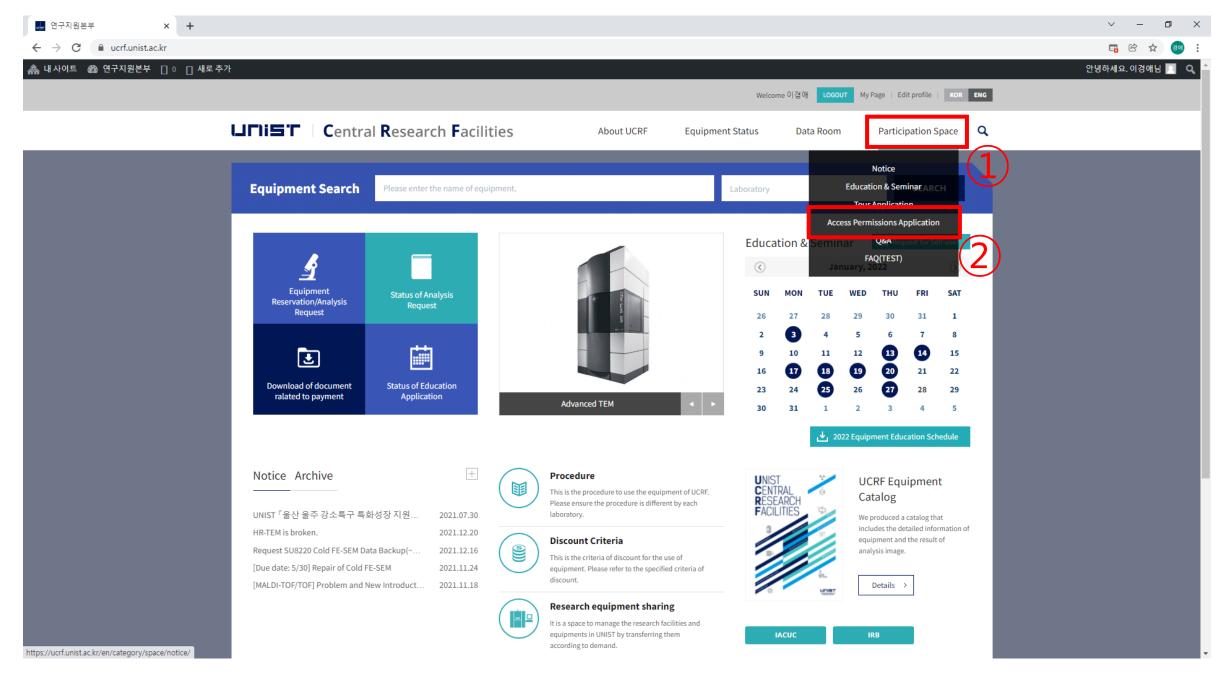
Problem: No image collected (Black, Gray or White) from the microscope in Genesis. Image collection is okay in the EDAM or SG Shell.

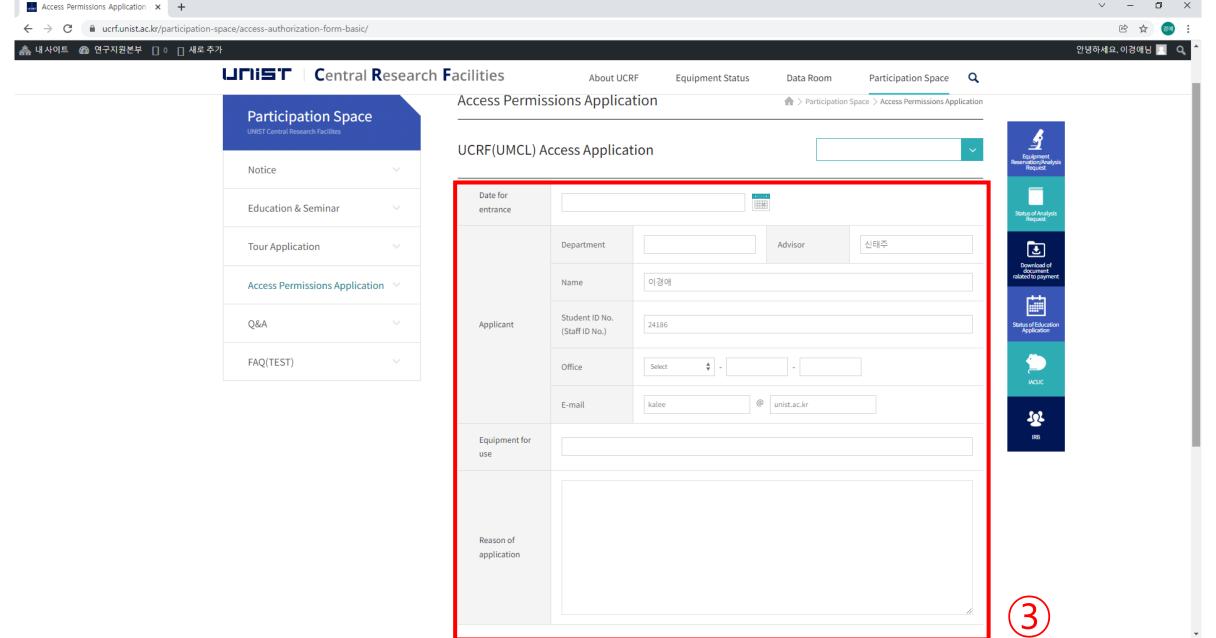
If the image collection in the SG or EDAM Shell is okay, but the image collection in TEAM or Genesis is not okay, it may be due to a corrupted configuration file.

- Close all EDAX software
- Go to the My Documents folder and delete all gen\*.ini files such as geneds.ini, genimg.ini, genpart.ini, etc.
- Check if image collection in Genesis works.
- If the above does not work do the next steps:
- Close all EDAX software
- Use Windows Explorer to browse the c:\ drive
- Go to c:\edax32\sys folder
- Locate and then double click on edaxclean.exe
- Go to c:\edax32\img folder
- Delete file edi32coll.cfg.
- Delete the file gpuscan.cfg
- Reboot the pc.
- Check the image collection in Genesis
- If the above does not work do the next steps:
- Close all EDAX software
- Use Windows Explorer to browse the c:\ drive
- Go to c:\edax32\Genesis folder
- Select and RENAME: Preference.ini to Preference.old
- Check the image collection in Genesis

### **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(4168)



### Request for Self-user

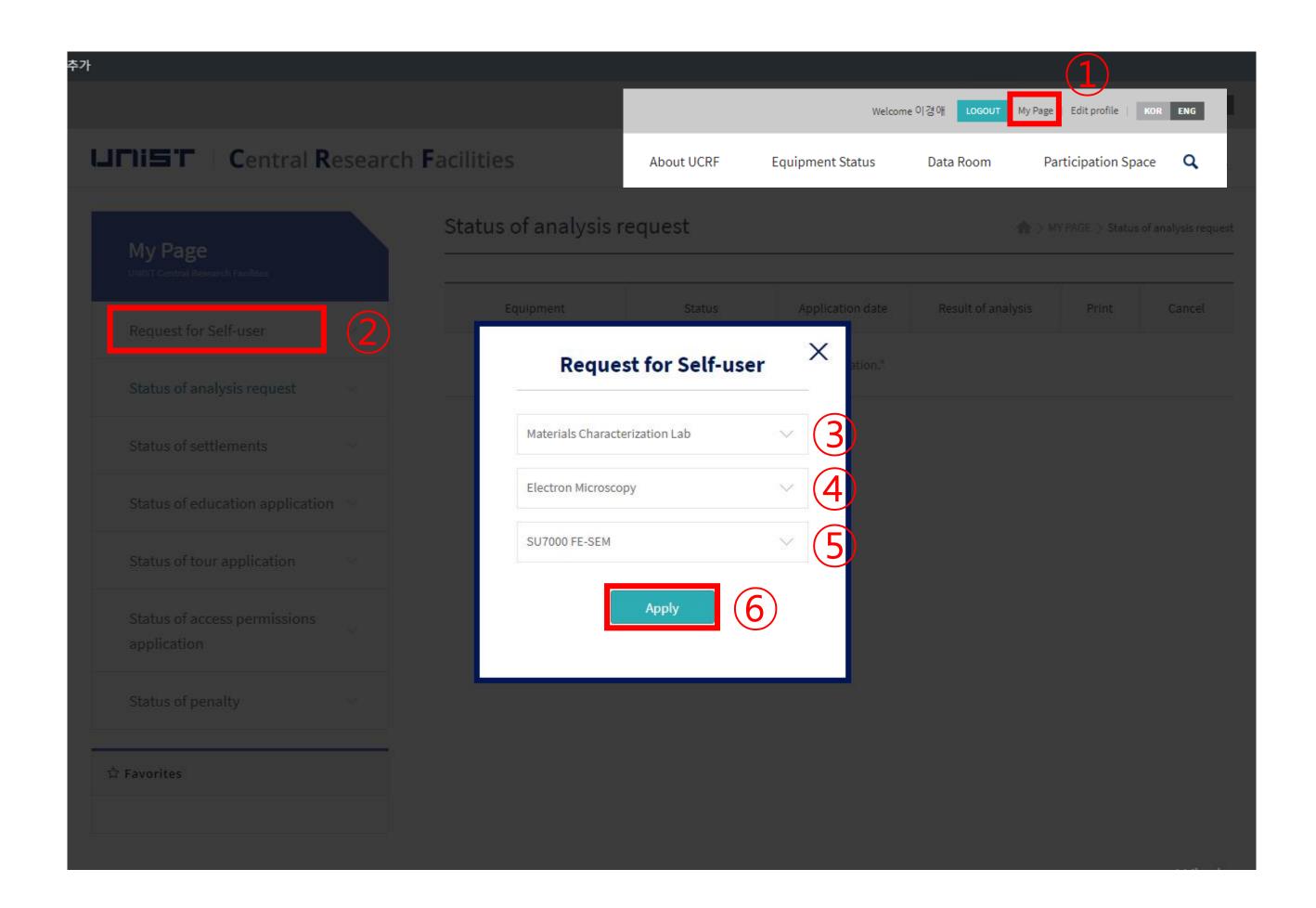
Create Account



- 1. Click [Sign up]
- 2. Click [UNIST Member]
- Input [Portal ID/PW] → Click [Confirm]
   Please check your information
- 4. Input professor name in [Principal Investigation]
  - → Click [Professor search] → Click professor name
- 5. Click [Create Account]



# Request for Self-user

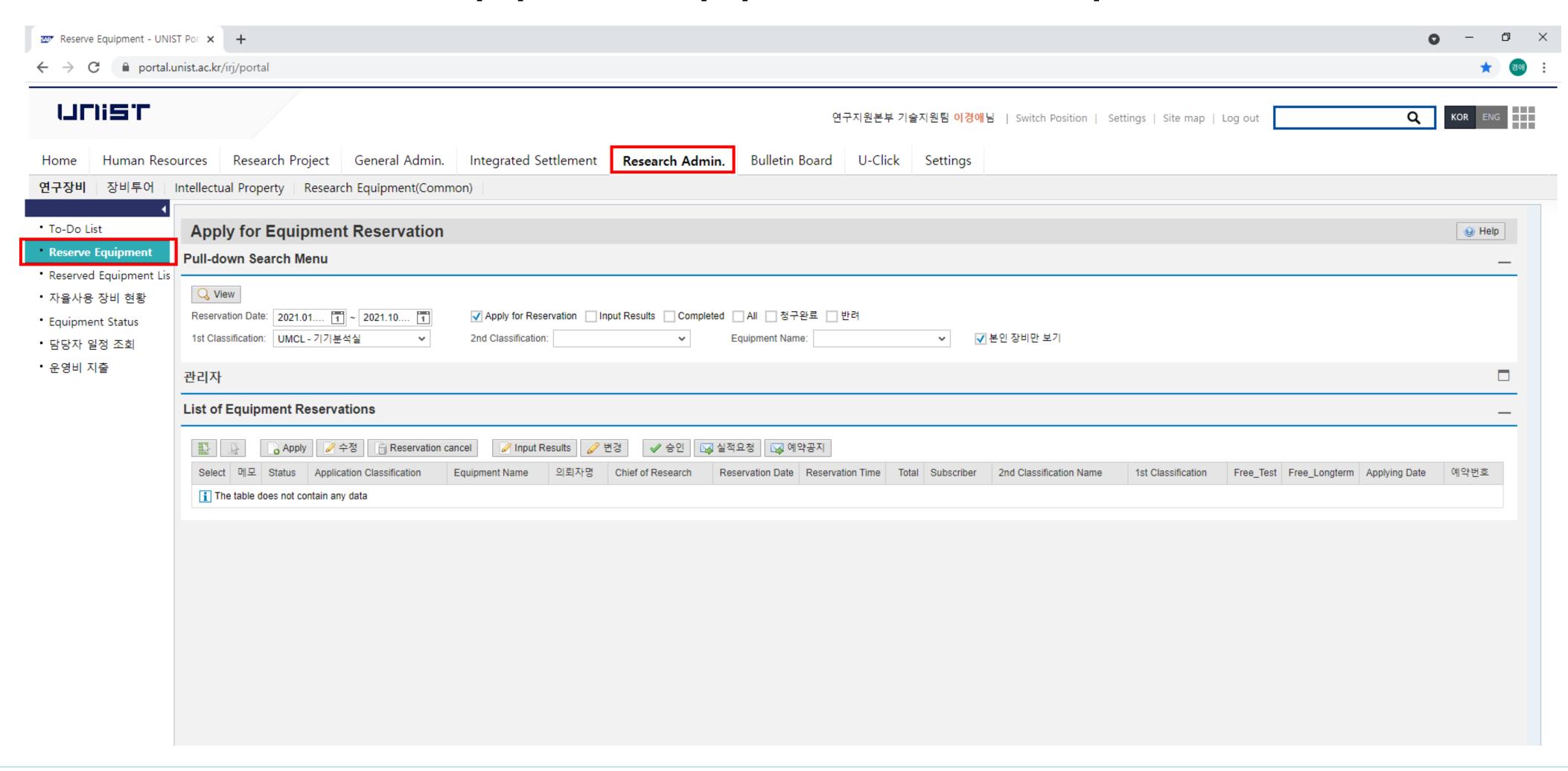


### After pass the test,

- 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 [Nano230 FE-SEM]
- 7. Click [Apply]

### Reservation

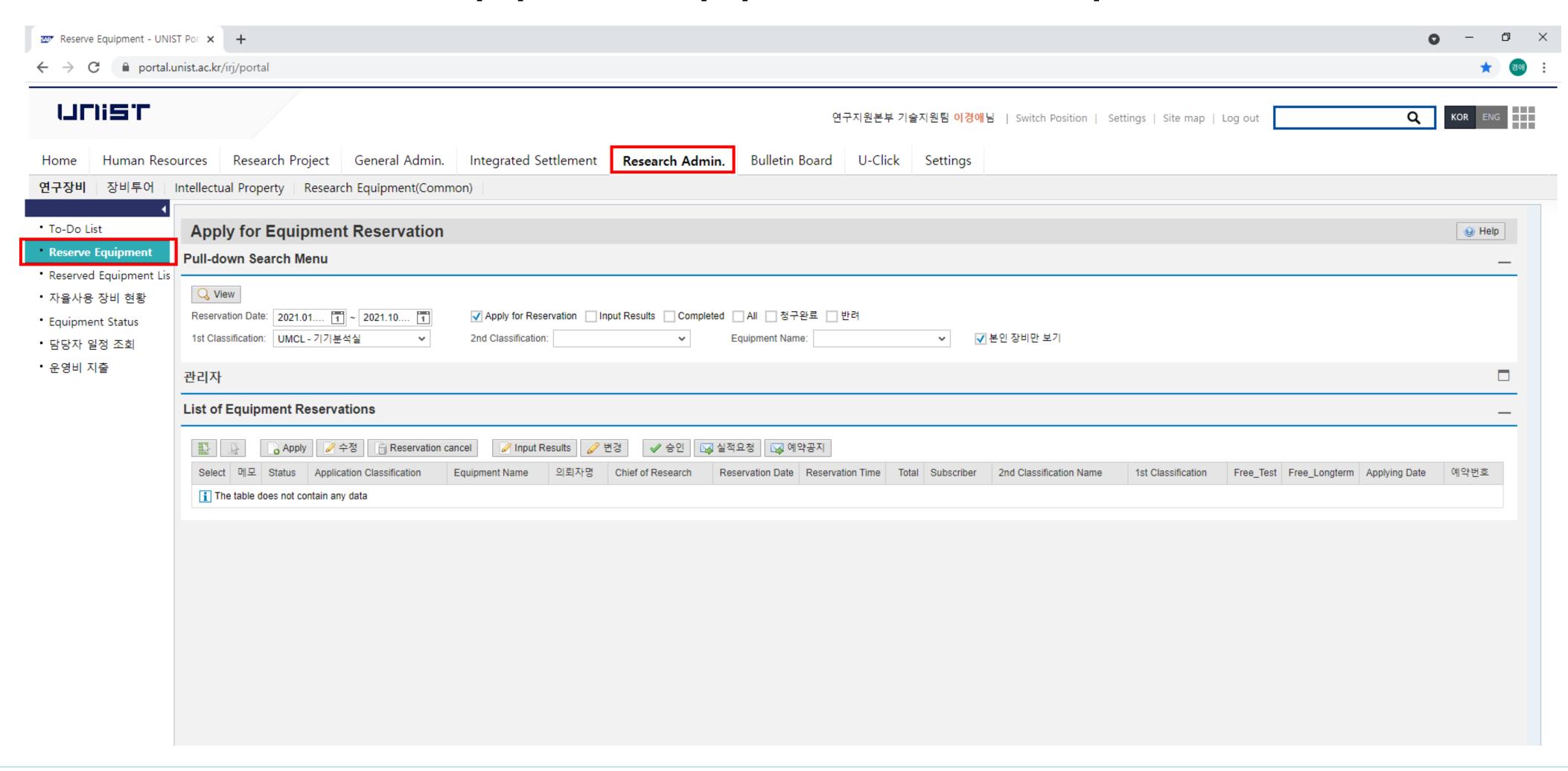
### portal.unist.ac.kr - Research Equipment - Equipment reservation/input result





#### Reservation

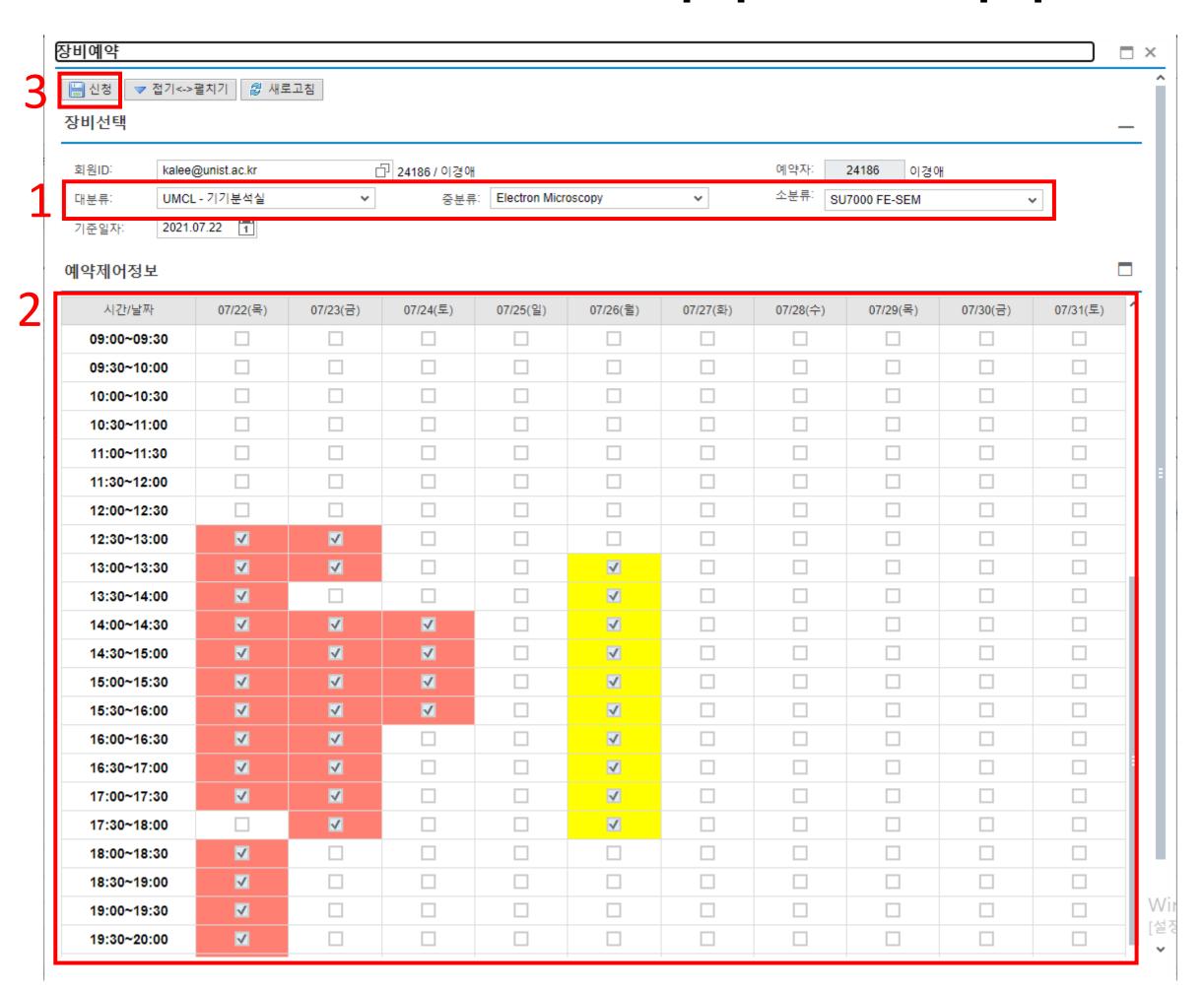
### portal.unist.ac.kr - Research Equipment - Equipment reservation/input result





#### Reservation

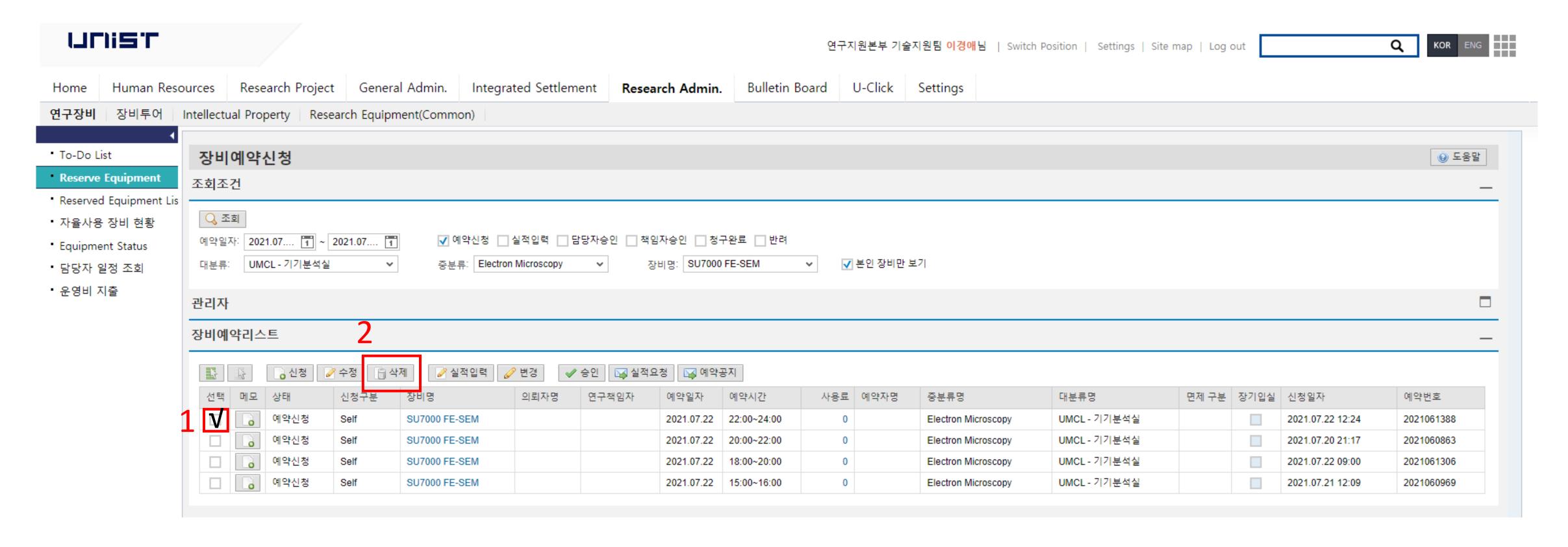
#### portal.unist.ac.kr - Research Equipment - Equipment reservation/input result



- 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**

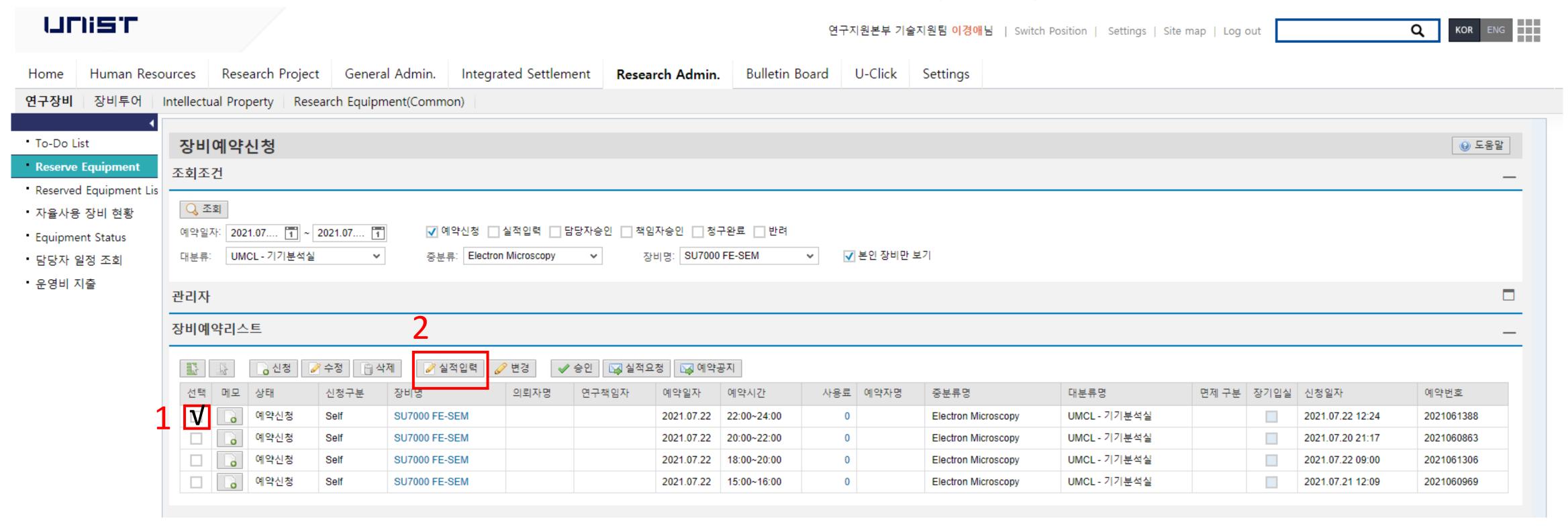
### portal.unist.ac.kr - Research Equipment - Equipment reservation/input result





### 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]



#### **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  $1 \sim 4$  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).

- 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.

- 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.
- ® 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.

#### 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.



- 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		
4	An abnormality or malfunction discovered before or after using the equipment did not immediately reported to the manager		
5	Damage, malfunction, and loss of equipment due to user negligence (* The user is responsible for all costs incurred due to the negligence. *)		
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		
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	5 points or more	<ul> <li>The user is prohibited from using the equipment for 1 month.</li> <li>When the prohibition period is finished, the user must complete the training and assessment again.</li> </ul>
to 1 user	8 points or more	<ul> <li>The user is prohibited from using the equipment for 3 months.</li> <li>When the prohibition period is finished, the user must complete the training and assessment again.</li> </ul>
Total penalty points awarded to 1 laboratory for 1	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."
equipment	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)





# THANK YOU