ULSAN NATIONAL INSTITUTE OF SCIENCE AND TECHNOLOGY

Transmission Electron Microscopy

Normal-TEM Operation Training Course

UCRF TEMs Specification

Advanced TEM (Titan³ G2 60-300)	CS-STEM (JEM-ARM300F)	HR-TEM (JEM-2100F)	Normal TEM (JEM-2100)
			
B115, Bldg.102	B102, Bldg.108	B106, Bldg.102	B104, Bldg.102
Collaboration	Request	Request/Self	Request/Self
Prof. Hu Young Jeong Jong Hoon Lee	Jong Hoon Lee	Jeong Hwan Lim	Jihyun Park
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UCRF TEMs Specification

Normal-TEM

HR-TEM

모델명 (제작사)	JEM-2100 (JEOL)	JEM-2100F/Cs (JEOL)
Accelerating voltage(kV)	200	200
Lattice resolution(nm)	0.23	0.102
HR-TEM image	O	O
Diffraction pattern image	O	O
Probe-CS corrector	X	O
Image-CS corrector	X	X
Atomic STEM image(nm)	X	0.096
EDS	X	O
EELS	X	X
분석료(원/시간)	104,400	208,800
비고 및 특징	Lab_6, 자율사용장비, 시편상태확인, 일반적인 TEM 분석	FEG, HR-STEM, 원자 수준의 이미지, 의뢰 가능 장비, 자율 사용 장비 예정
담당자(연락처)	박지현(4177)	임정환(4175)

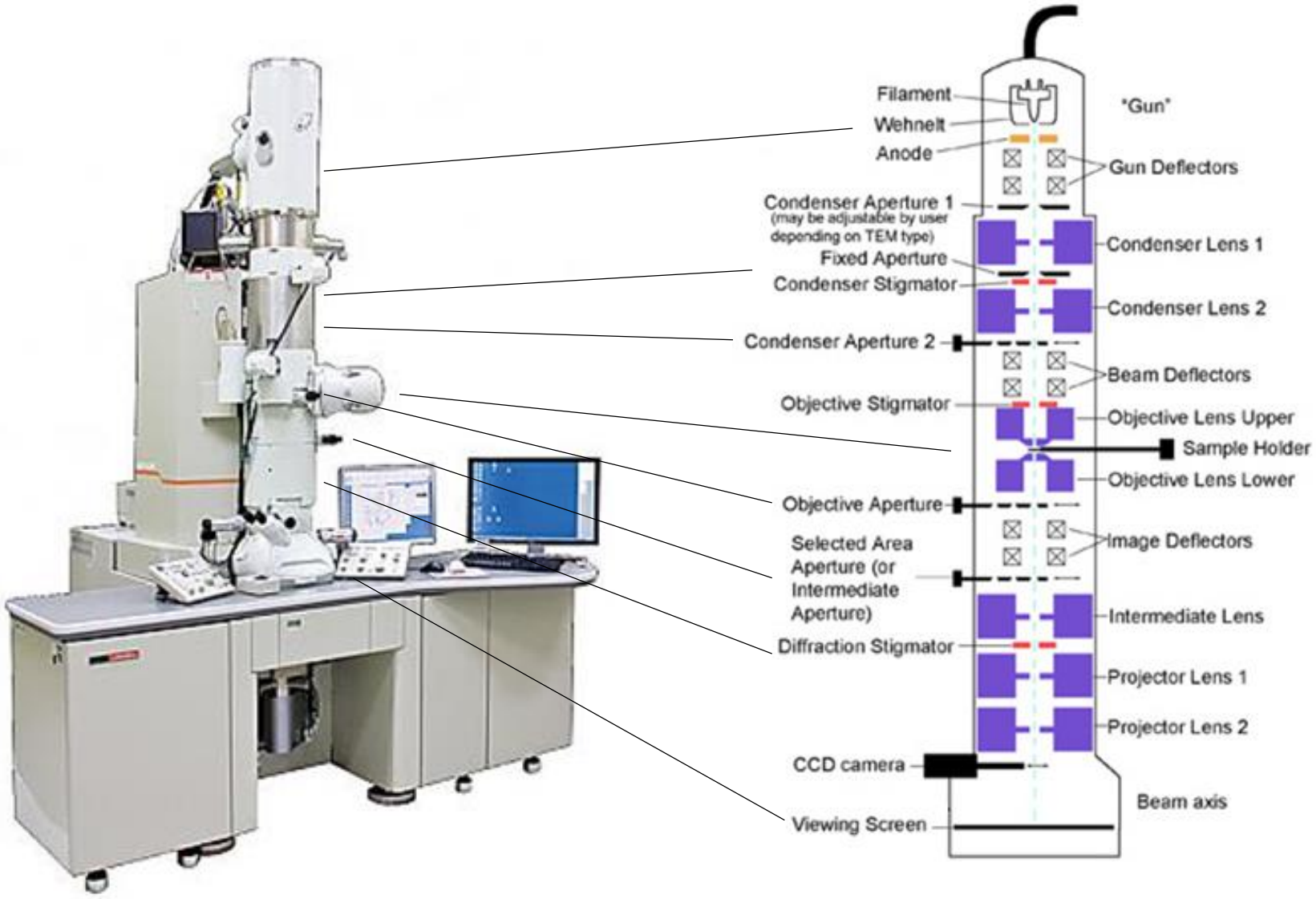
UCRF TEMs Specification

CS-STEM

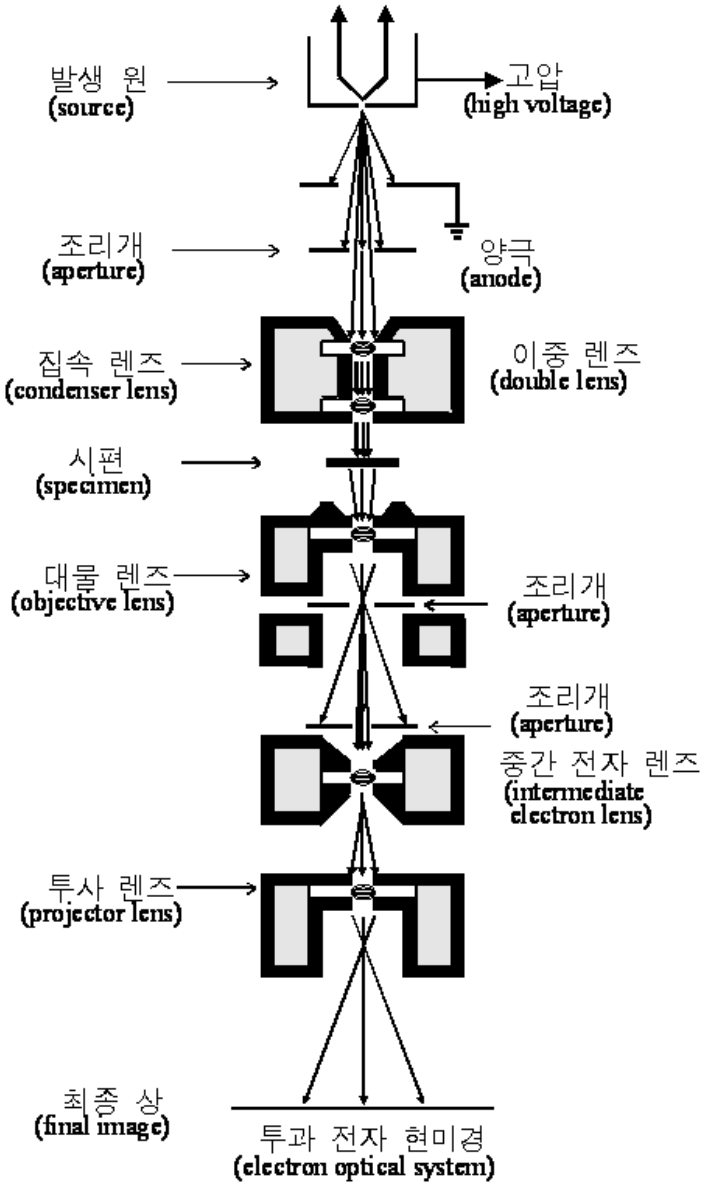
Advanced-TEM

모델명 (제작사)	Grand ARM-300F (JEOL)	Titan³ G2 Cube 60-300 (Thermo fisher scientific)
Accelerating voltage(kV)	60/80/160/300(not 200 kV)	60/80/200/300
Lattice resolution(nm)	0.06	0.07
HR-TEM image	O	O
Diffraction pattern image	O	O
Probe-CS corrector	O	O
Image-CS corrector	X	O
Atomic STEM image(nm)	0.058	0.07
EDS	O	O
EELS	O	O
분석료(원/시간)	313,200	417,600
비고 및 특징	원자 수준의 이미지, 60~300 kV 가속 전압 변화, 의뢰 전용 장비	Monochromator 장착, 이미지 & 프로브 수차보정기, 원자 수준의 이미지, 60~300 kV 가속 전압 변화, 의뢰 전용 장비(월, 화)
담당자(연락처)	이종훈(4171)	이종훈(4171)

The Structure of TEM



The Structure of TEM



- **Gun** : LaB6
- **Condenser lens** : 빛이 밝고 평행 광선이 되도록
- **Condenser aperture** : 시편에 조사되는 전자 빔을 평행하게 하고 전자빔의 크기 조절
- **Objective lens** : 결상하는 역할
- **Specimen chamber** : 시편을 넣어서 조작
- **Objective aperture** : 전자 회절상의 한 점, 또는 몇 개의 점을 선택하여 결상
- **Selective area aperture** : 제한 시야 회절을 위해 확대된 상에서 일부 영역 선택
- **Projector lens** : 확대하는 역할
- **Detector** : 전자와 시편의 상호 작용으로 생기는 전자와 파 감지
- **기록장치, 펌프 등...**

Sample preparation

1. Drop-casting process(nanoparticles)

a. Choice of solvent to disperse particles (ex. Ethanol, DI water, toluene..)

b. Adjusting concentration of solution

c. Ultrasonic dispersion of your solution

d. Selection of suitable support grid and grid frame

1) Carbon formvar grid - normal grid

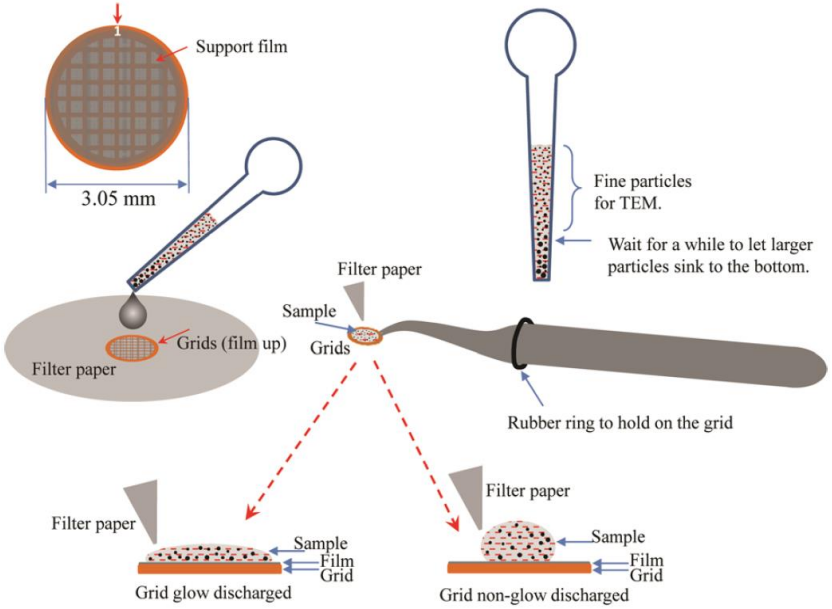
2) Holey carbon grid - sample size below 5nm

3) Lacey carbon grid - carbon materials

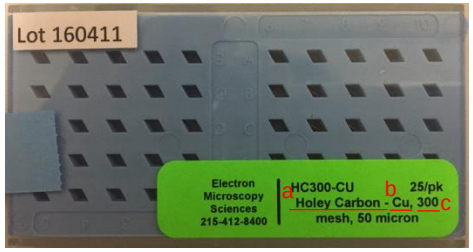
4) If you want to EDS analysis, you avoid the same material (sample, grid frame)

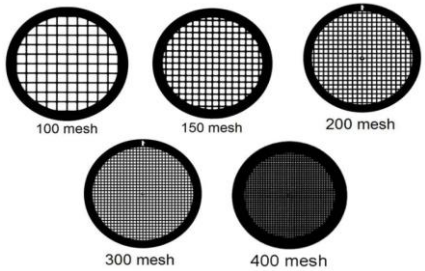
e. Drop on your grid 2~3 point

f. Dry



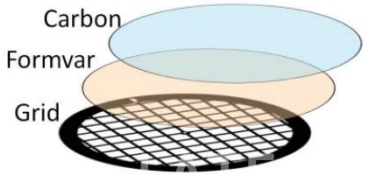
2. Grid description



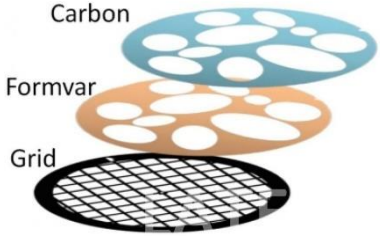


a. Grid type
b. Grid frame
c. How many mesh in grid

3. Grid type



- Carbon film grid



- Holey carbon grid

Guideline of Normal TEM operation

1. Check the equipment before use

a. Error message

b. Column vacuum

c. LN_2

2. Increase the accelerating HT voltage

a. HT: 160kV → 200kV

3. Loading of the specimen

a. Sample loading on the specimen holder

b. Dry ion pumping station

c. Inserting and removing the specimen holder

4. Beam alignment

a. CL aperture centering

b. CL astigmatism correction

c. High voltage centering

d. OL astigmatism correction

5. Check the equipment after use

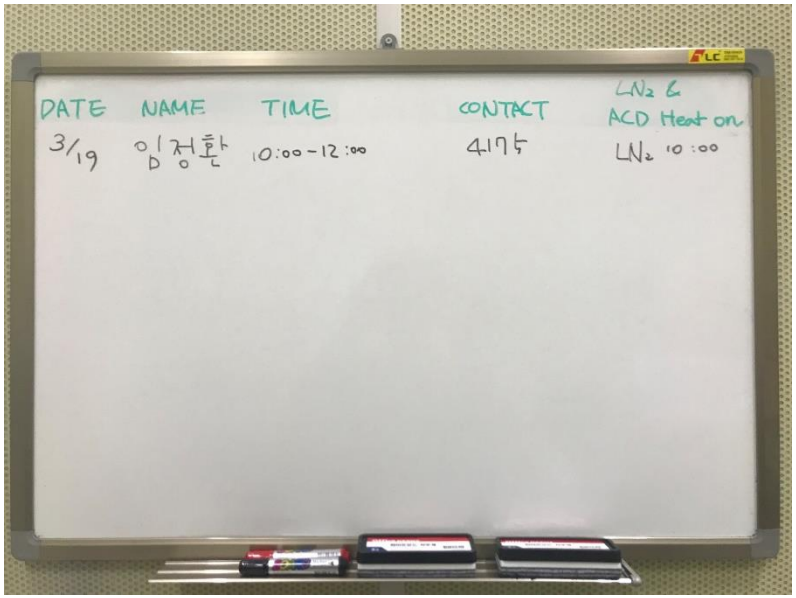
a. Column vacuum

b. Error message

c. LN_2

Check the equipment before use

Check the white board

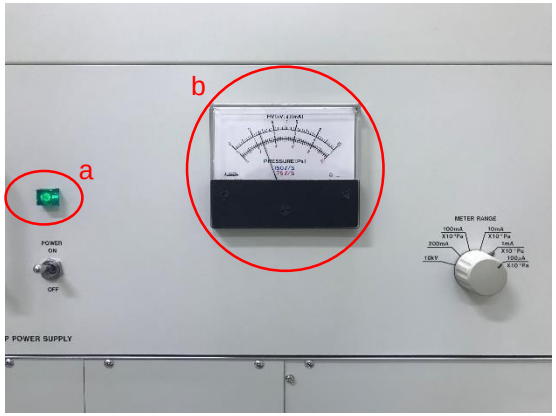


1. **Check notice and the message on the board (If there are something wrong, you can't use the equipment)**

2. **LN_2 should be filled below 4 h on the board**
   - **200 kV as it is**
   - **Refill LN_2**
   - **Write the refill time on the board**

Check the Column Vacuum

1. Check the control panel before use the equipment(SIP power supply)



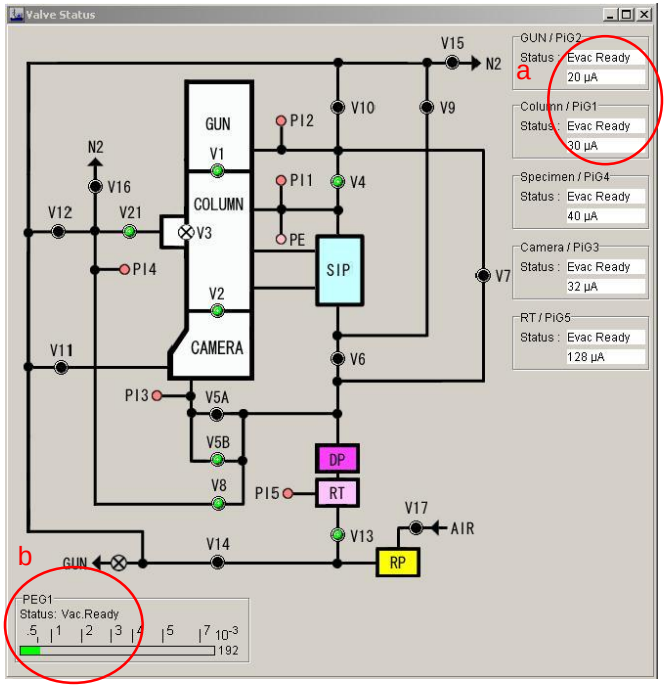
a. Check that the power lamp of the SIP is green

b. SIP vacuum gauge should be 1~2

(Meter range knob → 1mA x 10)

c. When it exceeds 3*10 Pa, you can't use the equipment

2. Check the vacuum system on the program



a. Check the vacuum of Gun & column

1) Status: Evac Ready

2) Both values should be less than 30uA

b. Check PEG1

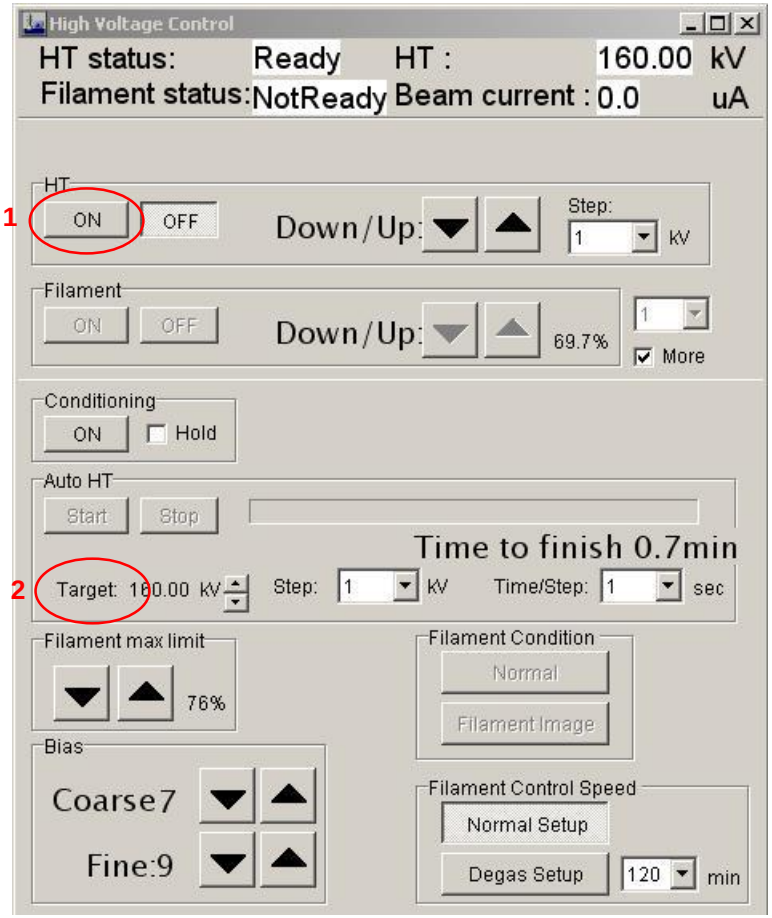
1) Status: Vac. Ready

2) Bar: Green

Increase the HT voltage

▶ **If you are seeing that message 'ACD Heat ON' you are first user at today.**

So, You have to do increase the HT voltage step.



1. Turn ON the HT

1) Click HT ON

- HT status : Ready → ON
- HT : 160kV
- Beam current : 0 uA → 80 uA

2. Increase the HT

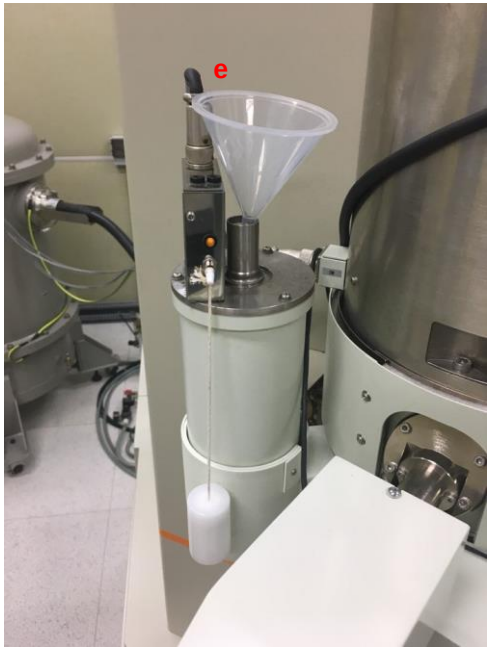
1) Target : 200kV, Step : 0.1kV, Time/Step : 3 sec

2) Click start button of auto HT

- HT : 160kV → 200kV
- Beam current : 80 uA → 102 uA

Fill LN_2

1. Fill LN_2



a. When you fill LN2, your eye must be upper the ACD tank using a ladder

b. Put the container to the tube entrance

c. Lift the green valve toward to the top

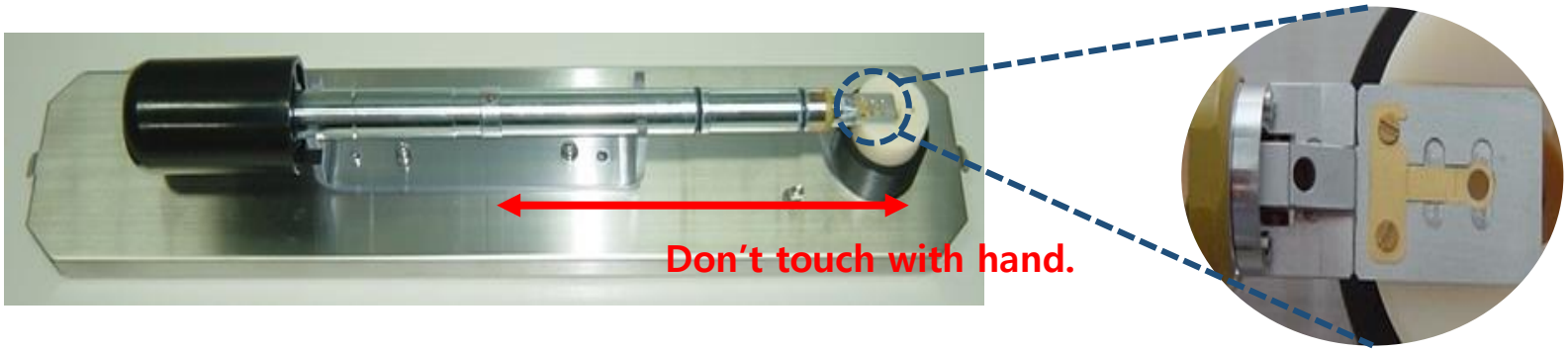
d. Fill LN2 to about 1/3 of the container

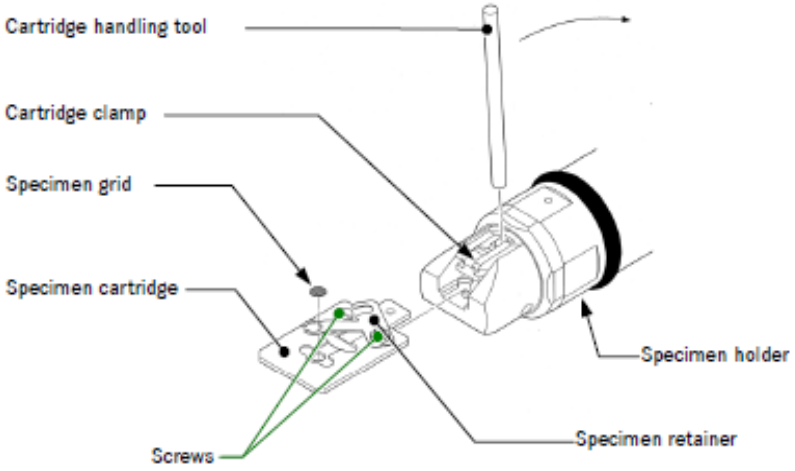
e. Put the funnel into ACD liquid nitrogen tank fill LN2 fully

f. After filling LN2, close the cap

- Be sure that view chamber cover

Load a sample





1. Load the specimen where the upside of target face is located on upside
2. Put the spacer on the sample
3. Put the plate on the spacer and screw on the plate

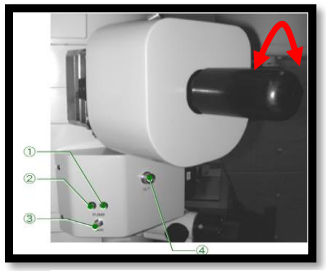
❖ Caution! If you tighten too much the screw, the screw line will be break .

Dry pumping station

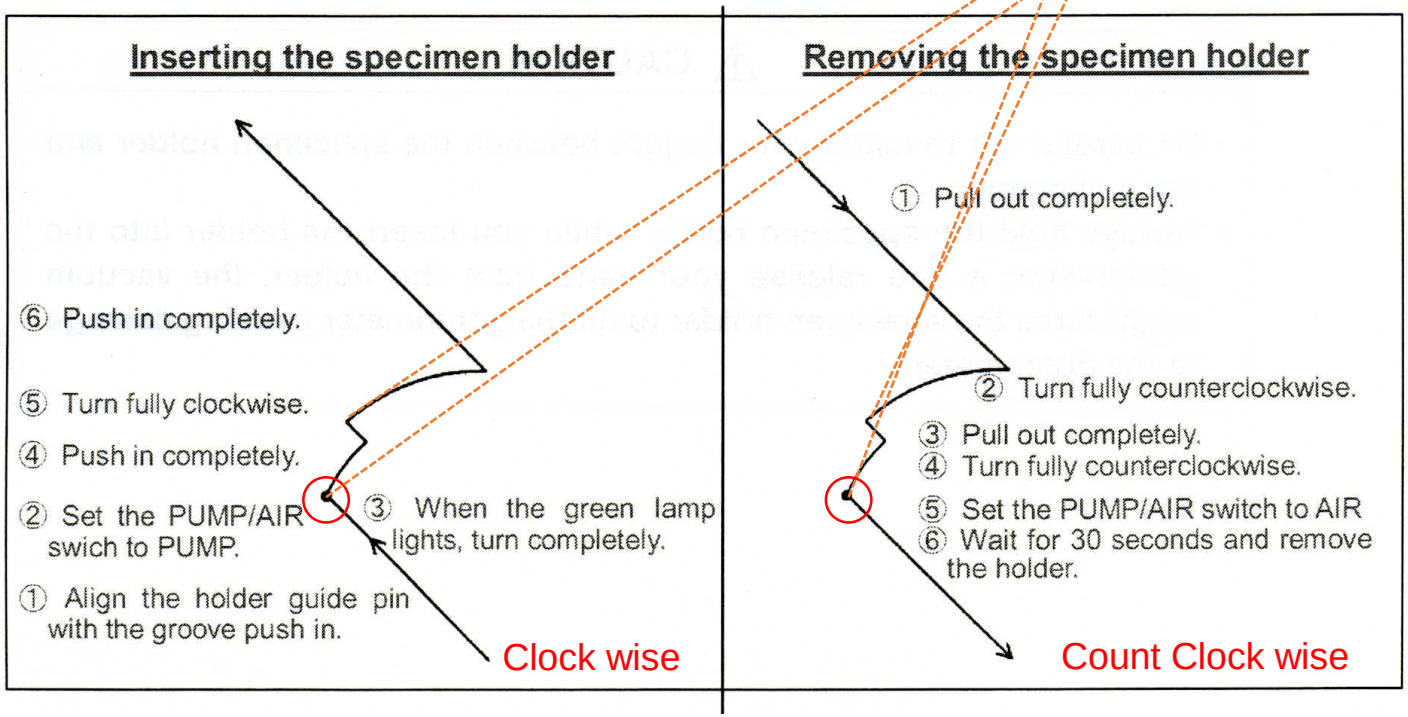
1. Insert the holder
2. Check power on status
3. Click Evac button
4. After 5 minutes, click the Vent button
5. The Vent light will be stop, you can pull out the holder straightly

Inserting the Specimen Holder

1. Inserting the specimen holder



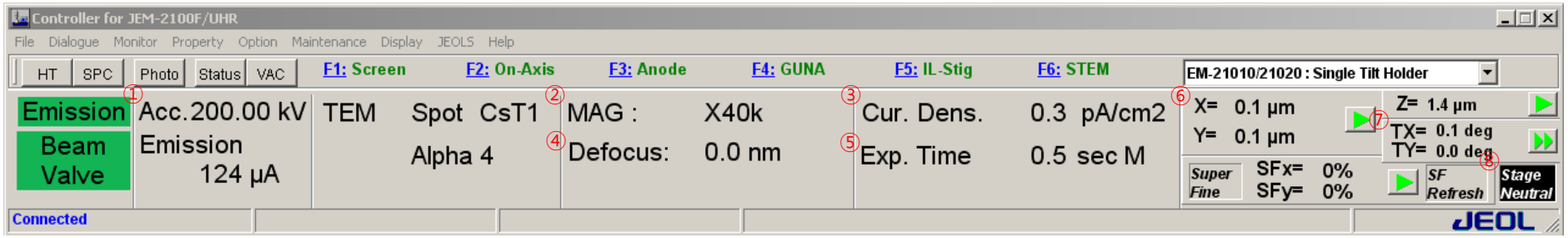
Guide pin



If you break the vacuum of TEM because you use inappropriate, you can't attend the training anymore.
You just fail, so be careful in use.

TEM controller window

1. TEM controller window



① HT Acc.: Accelerating voltage

② MAG: Magnification

- LOW MAG: x50~x8000, MAG1, MAG2: x6000~x1.5M

③ Cur. Dens.: Beam current density on the fluorescent screen

④ Defocus: Defocus length

⑤ Exp. Time: Exposure time

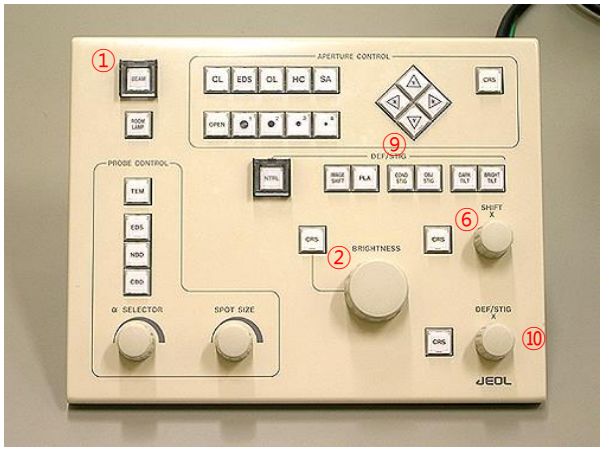
⑥ X, Y, Z: Specimen position in the X, Y, Z direction

⑦ TX, TY: Specimen-tilting angle in the X, Y tilt

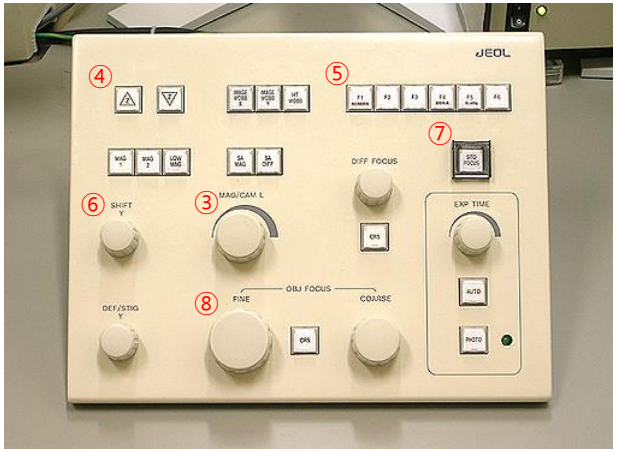
⑧ Stage neutral

Control Panel

1. TEM control panel



Left panel



Right panel

① Beam switch: Turn on the beam

② BRIGHTNESS knob: Converges and spreads the electron beam

③ MAG knob: Varies the magnification

④ Z switches: Shift the specimen in the vertical(up/down)

⑤ F1 switch: Screen up/down

⑥ SHIFT X, Y knobs: Shift the electron beam in the X, Y direction

⑦ STD FOCUS: The objective lens current to the original reference

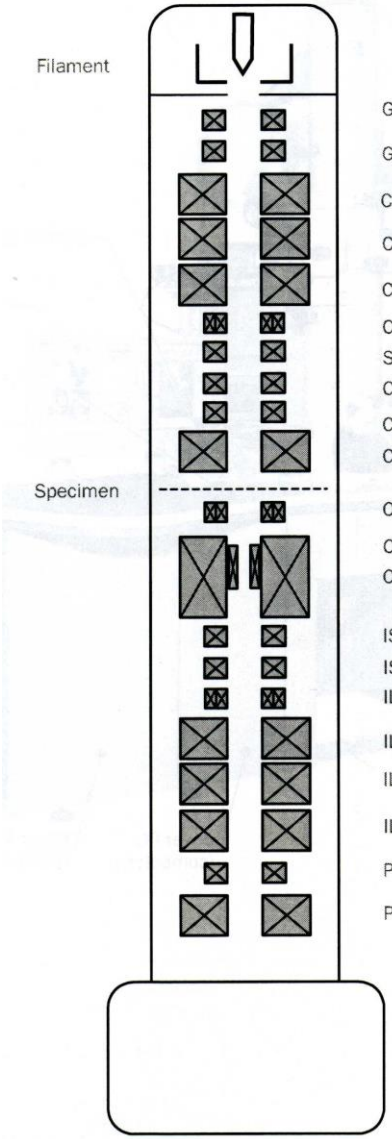
⑧ OBJ/FOCUS: Focuses the image

⑨ COND STIG button: Adjust the condenser lens stig when correcting the beam shape

⑩ DEF/STIG X,Y knobs

The Flow Chart of Beam Alignment

1. Normal-TEM



Gun1	Electron gun 1st beam deflector
Gun2	Electron gun 2nd beam deflector
CL1	1st condenser lens
CL2	2nd condenser lens
CL3	3rd condenser lens
CLS	Condenser lens stigmator
Spot	Spot alignment
CLA1	Condenser lens 1st deflector
CLA2	Condenser lens 2nd deflector
CM	Condenser mini lens
OLS	Objective lens stigmator
OL	Objective lens
OM	Objective mini lens
IS1	Image shift 1st deflector
IS2	Image shift 2nd deflector
ILS	Intermediate lens stigmator
IL1	1st intermediate lens
IL2	2nd intermediate lens
IL3	3rd intermediate lens
PLA	Projector lens deflector
PL	Projector lens

2. Z-Axis (40K)

STD Focus - Image Wobb X – Z button ▲▼

3. Flow chart of beam alignment

CL aperture centering

↓

CL astigmatism correction

↓

High voltage centering

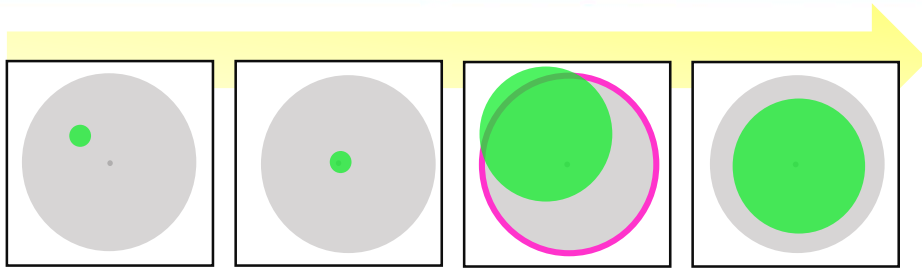
↓

OL astigmatism correction

CL Aperture Centering

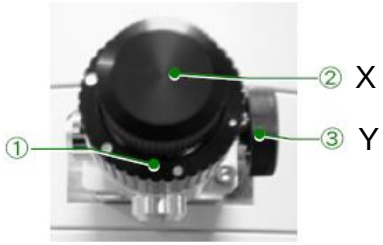
1. Condenser lens aperture centering

1) Adjust the magnification to over x40k
2) Beam centering
3) Spread the electron beam (80~90% of large screen)
4) The beam should be center of the large screen



Beam Shift

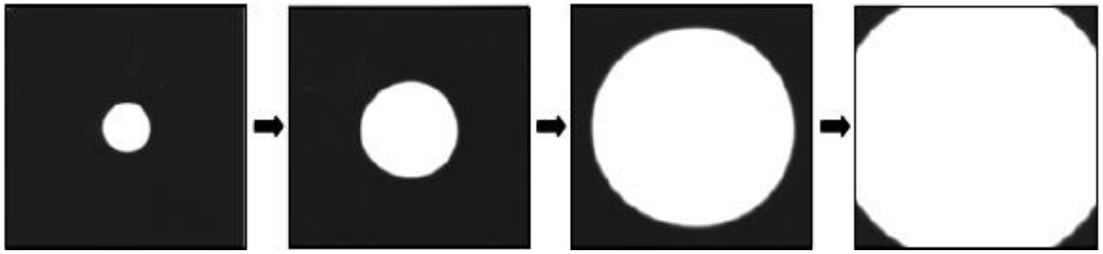
Aperture control



CL aperture X/Y knobs

- Do not touch ①
- When you turn ② or ③, do not turn too much

No good



Good

CL Astigmatism Correction

2. Condenser lens astigmatism correction

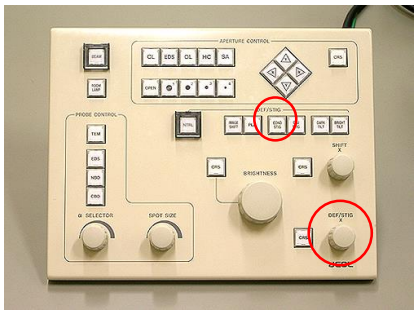
a. Adjust the magnification to over x60k

b. Beam centering

c. Make the shape of the electron beam to be circular triangle

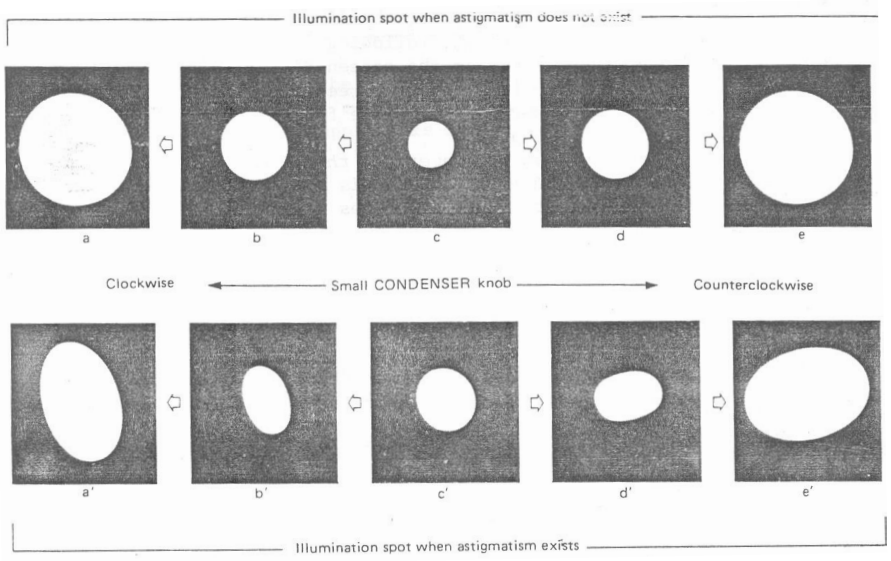
COND(CL) Stig button +

DEF/STIG X/Y knobs



Left panel

Right panel



High Voltage Centering

3. High voltage centering

a. Adjust the magnification to over x100k

b. Beam centering

c. Find the sharp edge of sample, and locate it the large screen center

d. Down the small screen

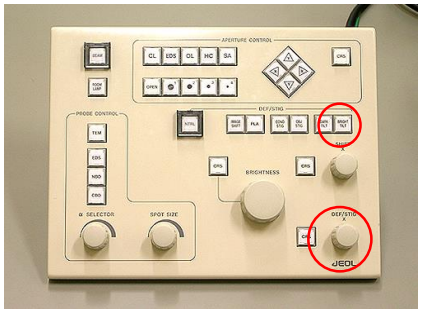
e. Let's make the edge point of the sample do wobbling at the small screen center

f. Make the movement of the edge point of the sample minimize

- If HV centering is well aligned, beam is wobbling at the beam center

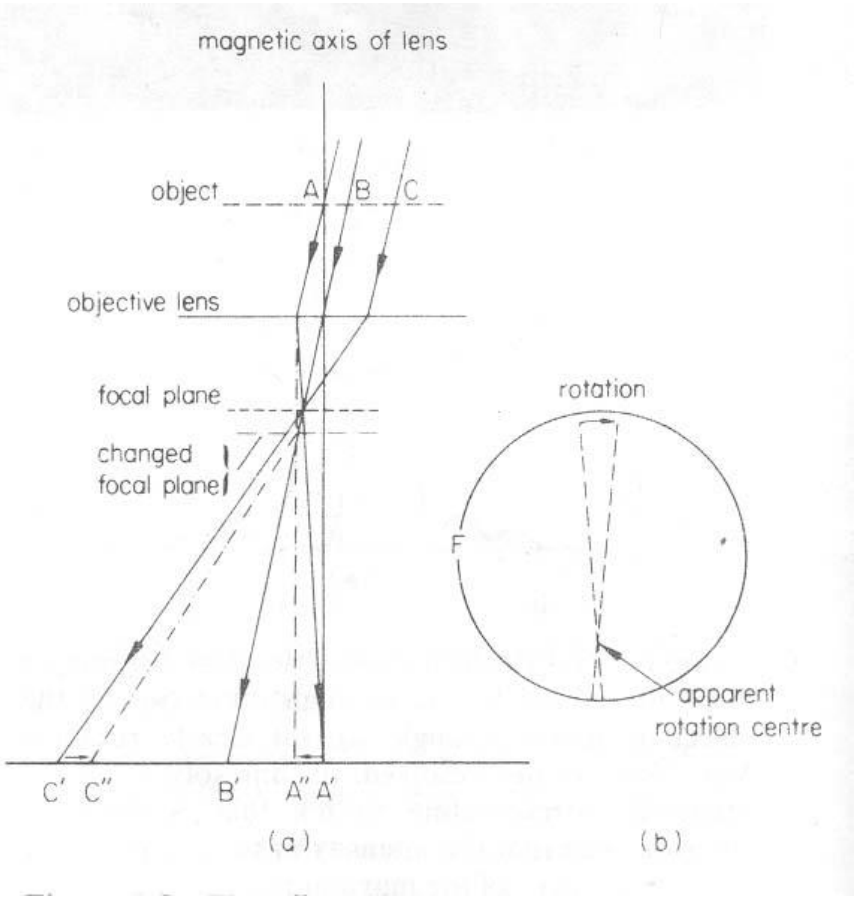
HT wobbler button +

Bright Tilt button + DEF/STIG X/Y knobs



Left panel

Right panel



OL Astigmatism Correction

4. Objective lens astigmatism correction

a. Adjust the magnification over x200k

b. Beam centering

c. You can use the amorphous phase of the sample or amorphous carbon grid

d. Make Cur.den to under 40pA/mm2
- Spread the beam with brightness knob

e. Up the large screen with F2 button

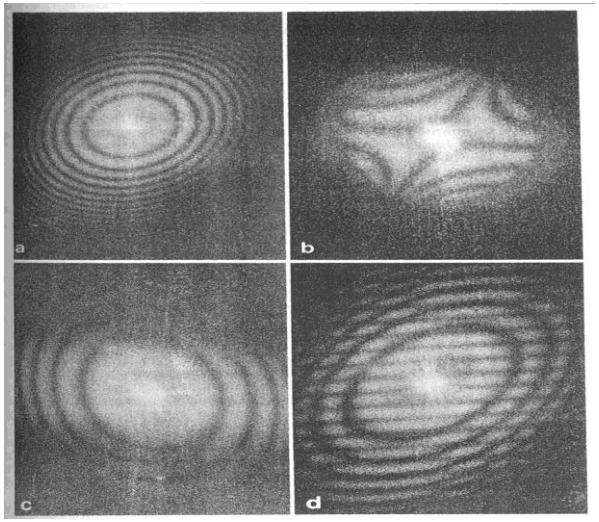
f. Start view and Process-Live-FFT

g. Make the FFT image to be perfect circle

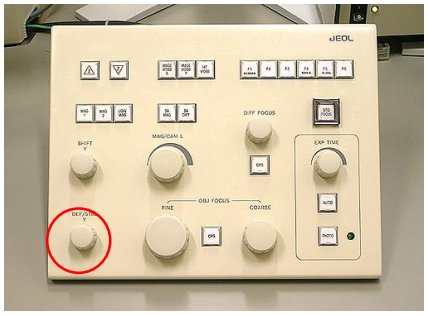
OBJ(OL) Stig button +

DEF/STIG X/Y knobs

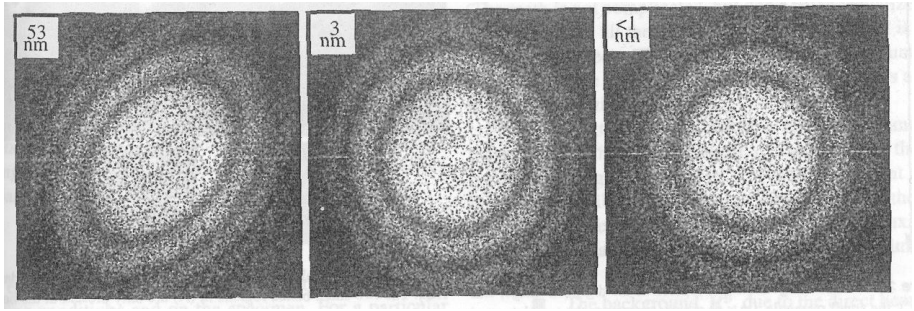
When OL lens is well aligned, FFT shape is circular



Left panel

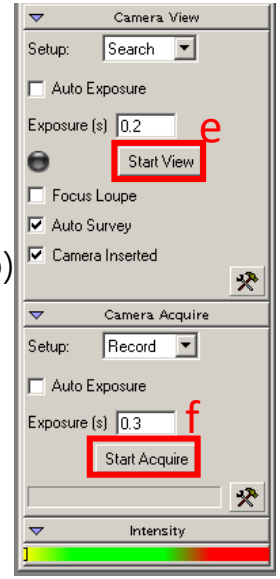


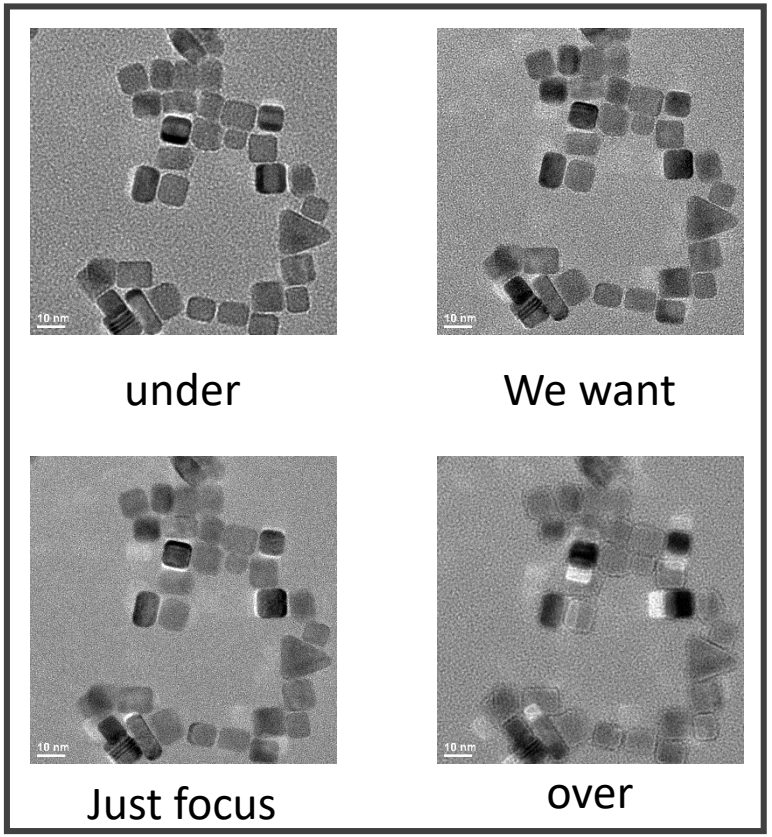
Right panel



Take images

a. Adjust the magnification that you want
b. Beam centering
c. Make Cur.den to under 40pA/mm2
   (Spread the beam with brightness knob)
d. Up the large screen with F2 button
e. Start view
f. Focusing and click to start acquire





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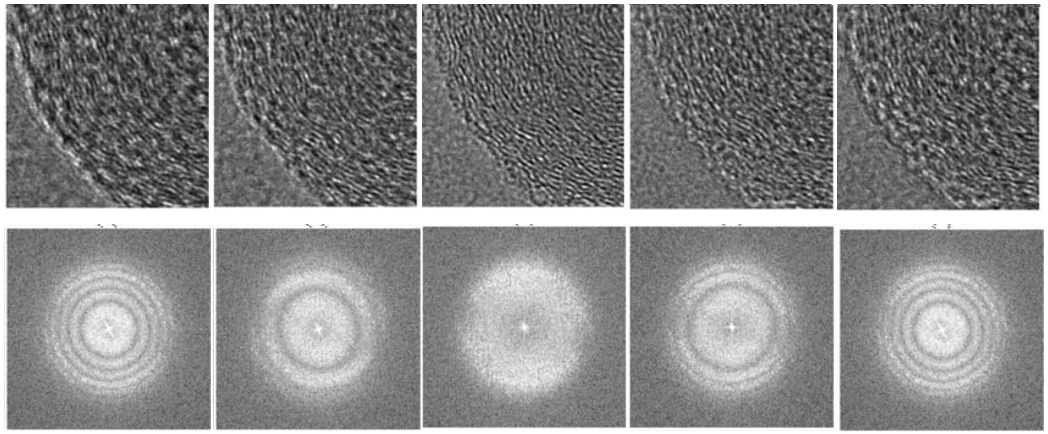
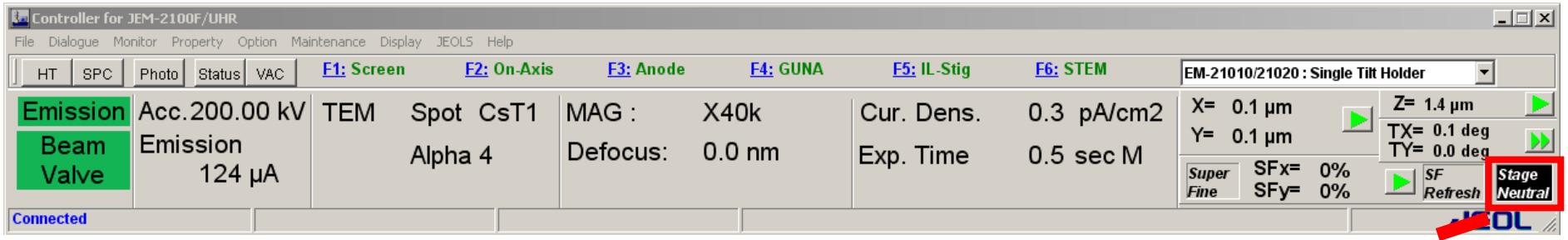


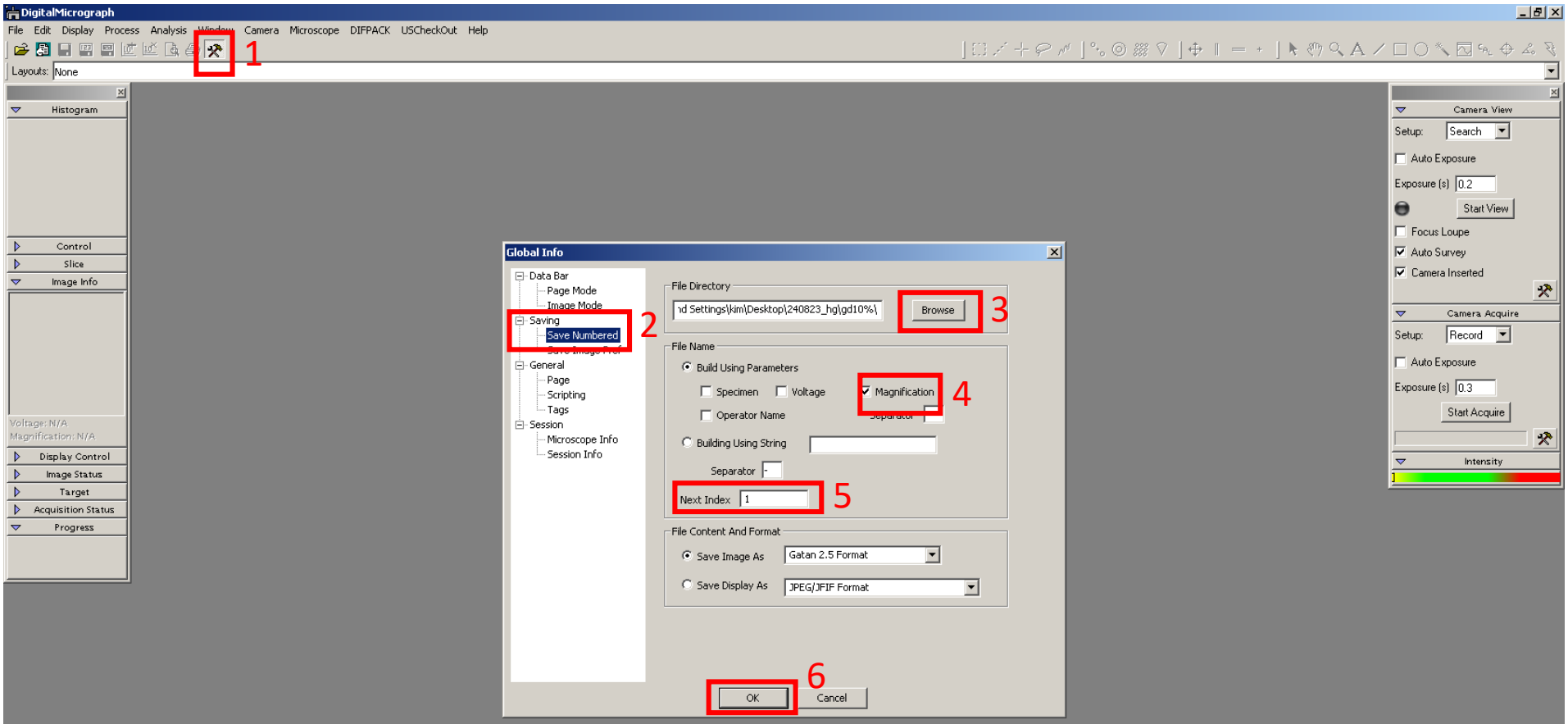
Before turn off the filament



1. Mag : 40K
2. Beam centering
3. Increase the beam size
4. Stage Neural (twice, doble check!)

And then you can turn off the filament.

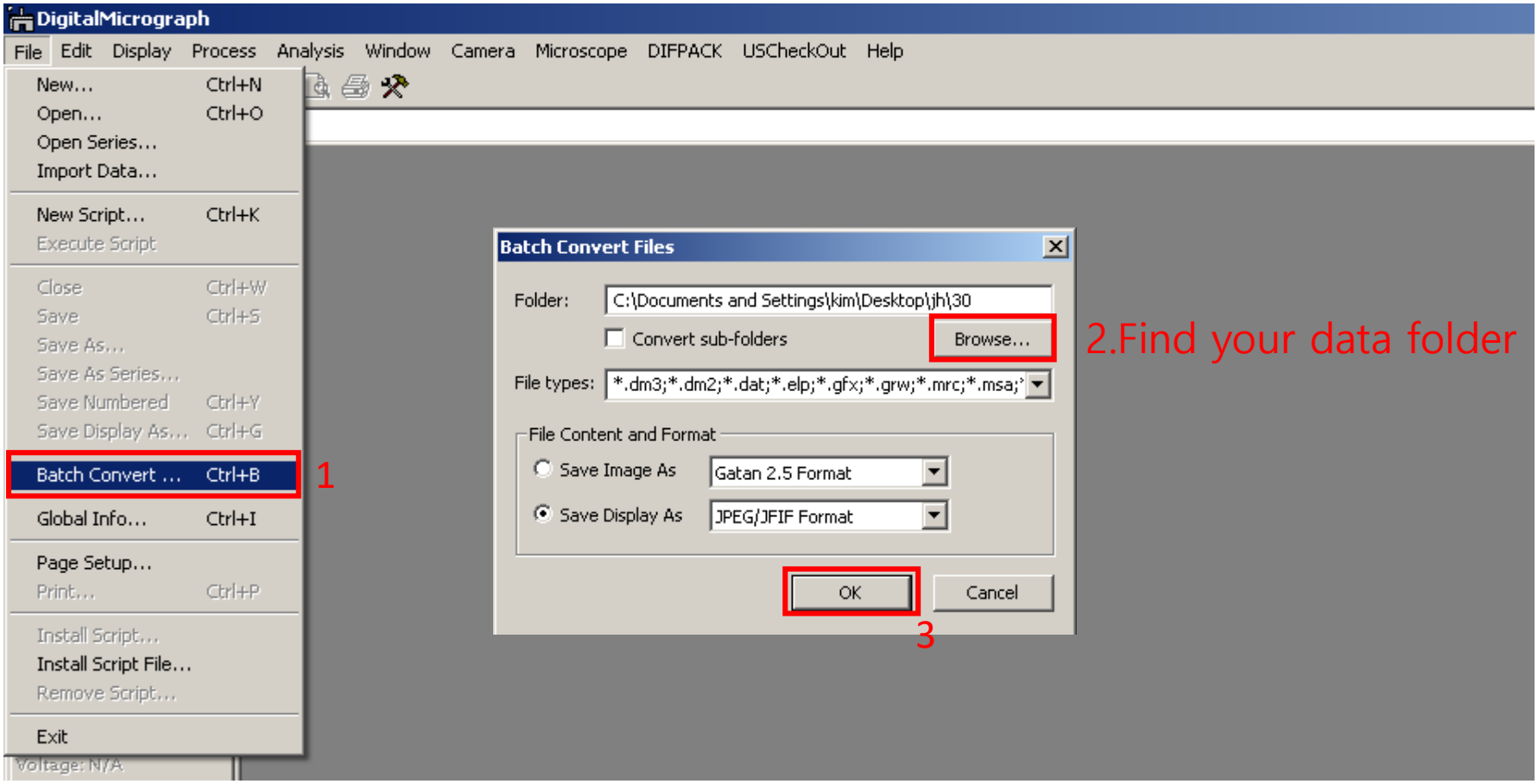
Save the data



7. If you click on each image file and press ctrl+Y, the data will be saved with the .dm3 extension.
Be sure to check whether the image file name has been converted to scale x index number.(ex. 40000 x 1)
Press Alt + Shift +  to close all image files.

HT: 200kV | Mode: IMAGING | Mag: 40k

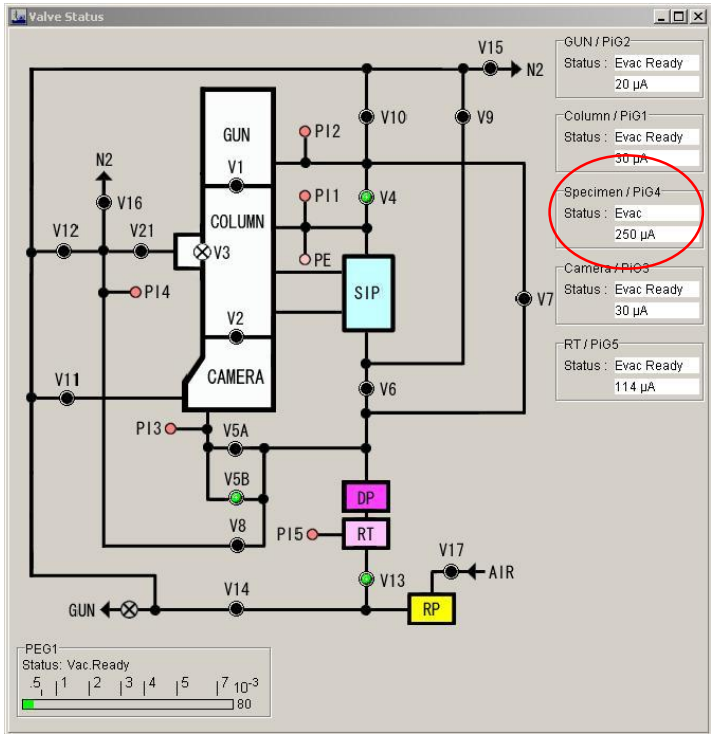
Data conversion(.dm3 → .tif, .jpeg, bmp)



4. After moving your data to the shared folder, upload the data to the Nas system.
If a problem occurs with the shared folder, you must re-configure the sharing settings on the computer using the NAS system.Use the manual in front of data pc.

Removing the Specimen Holder

1. Removing the specimen holder



a. Pull the specimen holder straightly

b. Turn the holder anti-clockwise and pull and turn the holder anti-clockwise

c. Down the switch ③ of the pump to air

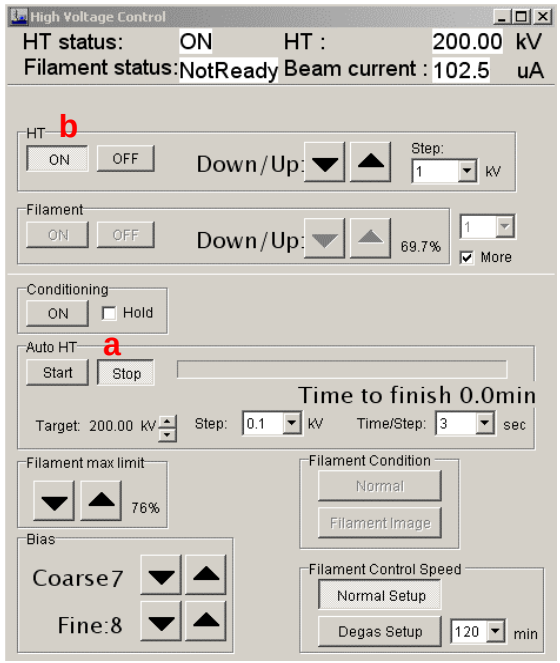
d. You have to wait until the value of specimen is over 220uA

e. Pull the specimen holder completely

- If you break the vacuum of TEM because you use inappropriate, you can't attend the training anymore. You just fail, so be careful in use.

Decrease the HT voltage & ACD Heat on

❖ When the blank time is over 5 h on the board or you are last user at today.
❖ You have to do decrease the HT voltage & ACD heat on step.



a. Decrease the HT

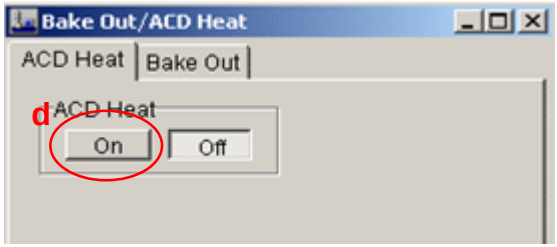
1) Target : 160 kV, Step : 1 kV, Time/Step : 1 sec
2) Click start button of auto HT (a)
- HT : 200 kV -> 160 kV
- Beam current : 102 uA -> 80 uA

2. Turn off the HT

1) Click HT OFF (b)
- Beam current : 80 uA -> 0 uA

3. Plug ACD heater (c)

4. ACD Heat On (d)



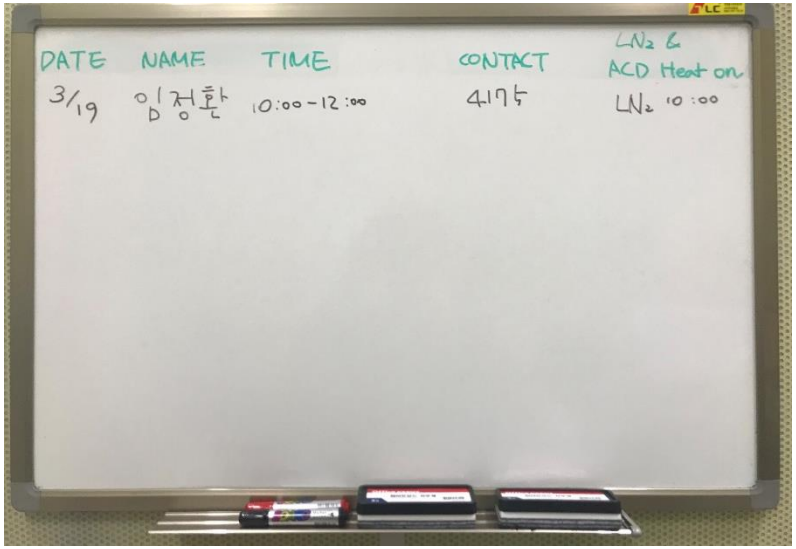
Check the equipment after use

- When you finished analysis, check the equipment again

1. Column vacuum
    a. SIP vacuum gauge
    b. Column vacuum on the program
2. Error message on the program
3. The reservation time on the google calendar
    a. When the blank time is below 5 hours
        1) 200kV as it is
        2) Refill LN_2
    b. When the blank time is over 5 hours
        1) Decrease and turn off HT voltage to 160kV
        2) Plug the ACD heater
        3) ACD heat on

Check the equipment after use

* Check the white board



1. LN_2 should be filled below 4 h on the board
   - 200 kV as it is
   - Refill LN_2
   - Write the refill time on the board

2. When the blank time is over 5 h on the board
   - Decrease HT voltage & ACD heat on - Write the ACD heat on time on the board

Emergency

연구실 번호 (Laboratory No.)	제1공학관 B104호 Engineering Bldg 102, B104
연구실명 (Laboratory Name)	투과전자현미경실 1 Normal-TEM lab.
연구실 안전담당자 (Safety Manager)	Jihyun Park 내선(Extension) (4177) ★ Please do not hesitate to contact "Safety Manager", if you have any queries or urgent business. (문의 사항 또는 급한 용무가 있을 시, "연구실 안전담당자"에게 연락 요망)

원외 주요 연락처

External Main Telephone

소방서 Fire Station	119
경찰서 Police Station	112
좋은삼정병원 Hospital	052)220-7500

화재,폭발,가스・화학약품 누출 등 응급상황발생시

Fire, Explosion, Gas and Chemical Leak etc.

응급상황 발생시 Emergency Call

052) 217-0119

UMCL user penalty points and action criteria

1. 벌점 부과 기준

가. 연구 활동 종사자의 부적절한 행동이 아래 표의 각 항목에 해당할 경우 벌점을 부과하며, 각 벌점은 중복 부과될 수 있다. (벌점의 소멸시효는 부과일로부터 1년)

순번	벌점부과내용	벌점
1	해당 장비에 대하여 직접 사용이 허가되지 않은 사용자가 장비 사용	5
2	장비 예약하지 않고 장비 사용(추가 예약 없이, 초과하여 장비 사용하는 경우 포함)	3
3	장비 사용 중 허용되지 않는 기능 조작	3
4	장비 사용 전/후에 장비의 이상 발견 시, 담당자에게 즉시 고지하지 않은 경우	3
5	사용자 부주의로 인한 기기 손상, 고장, 분실, 파손 * 해당 행위로 인해 비용이 발생할 경우 사용자 측에서 모든 책임을 진다.*	5
6	담당자가 장비 또는 시설의 정상적인 작동과 안전을 위해 반드시 파악해야 할 시료의 정보를 제공하지 않거나 허위사실을 고지하여 문제 발생	3
7	유독 물질 및 가스 누출, 화재 발생 위험 초래	5
8	공용 물품 및 타인 물품을 사전 동의 없이 사용하거나 소유·점유하는 경우	1
9	장비 사용 후 소등, 출입문 단속, 주변 정리 등을 확인하지 않고 퇴실	1
10	연구실 공통 안전수칙을 지키지 않는 경우(복장, 취식 금지 등 일괄 포함)	1

UMCL user penalty points and action criteria

2. 벌점 부과 후 조치 내용

가. 누적 벌점이 특정 기준을 초과하는 경우 조치 내용과 부합하는 제재를 가한다.

나. 사용 금지 조치 시행시, 해당 내용을 수칙 위반자 본인이 속한 학과 또는 기관(외부 기관일 경우)에 공문을 발송한 다.

다. 기준 이상의 벌점 합산에 따라 하기 조치 내용이 발생하였다 하여도 유효기간 내의 벌점은 효력이 있다. (조치사항 발생한 벌점이라도 유효기간 내에는 소멸하지 않음)

구 분	벌점	조치 내용
개인에게 부과된 벌점 합산	3점 이상	- 사용자 및 연구책임자에게 "벌점 5점 이상일 시 장비 사용이 1개월간 금지됨"을 이메일로 통보
	5점 이상	- 해당 장비 1개월간 사용 금지 - 사용 재개 시, 교육 및 평가를 다시 이수해야 함
	8점 이상	- 해당 장비 3개월간 사용 금지 - 사용 재개 시, 교육 및 평가를 다시 이수해야 함
동일 연구실에서 동일 장비에 부과된 벌점 합산	12점 이상	- 사용자 및 연구책임자에게 "벌점 15점 이상일 시 장비 사용이 1개월간 금지됨"을 이메일로 통보
	15점 이상	- 해당 연구실의 해당 장비 사용 1개월간 금지 - 소속 학과에 조치사항 공문 발송
동일 연구실에서 연구지원본부 전체 장비에 대하여 연구실 소속 학생들에게 부과된 벌점 합산	20점 이상	- 사용자 및 연구책임자에게 "벌점 25점 이상일 시 해당 연구실의 연구지원본부 전체 장비 사용이 1개월간 금지 됨"을 이메일로 통보
	25점 이상	- 해당 연구실의 연구지원본부 전체 장비 사용 1개월 간 금지