ULSAN NATIONAL INSTITUTE OF SCIENCE AND TECHNOLOGY

Transmission Electron Microscopy

Normal-TEM Operation Training Course

UCRF TEMs Specification

Advanced TEM (Titan^3 G2 60-300)	CS-STEM (JEM-ARM300F)	HR-TEM (JEM-2100F)	Normal TEM (JEM-2100)	Bio TEM (JEM-1400)
				
B115, Bldg.102	B102, Bldg.108	B106, Bldg.102	B104, Bldg.102	B104,B105, Bldg.102
Collaboration	Request	Request/Self	Request/Self	Request/Self
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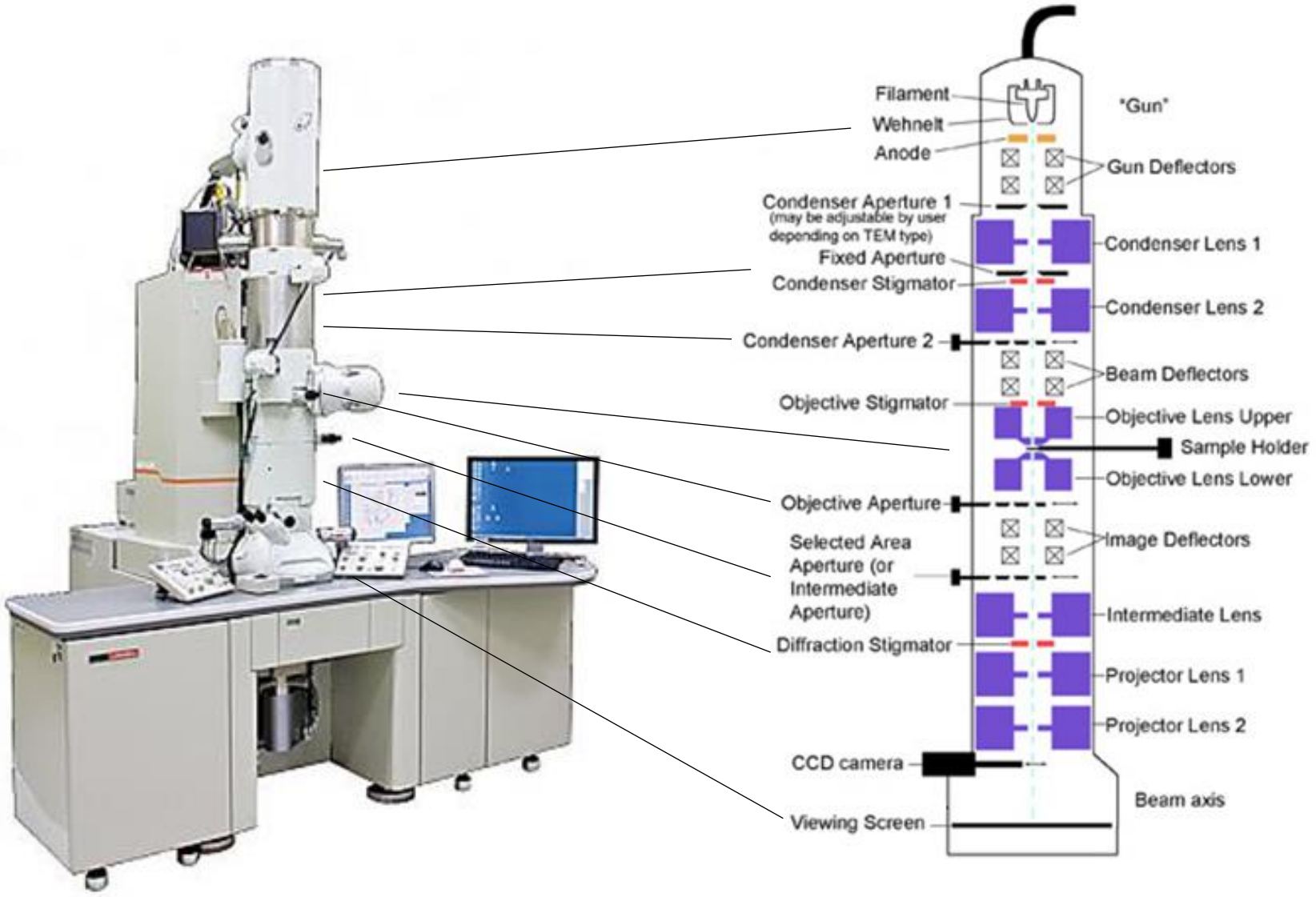
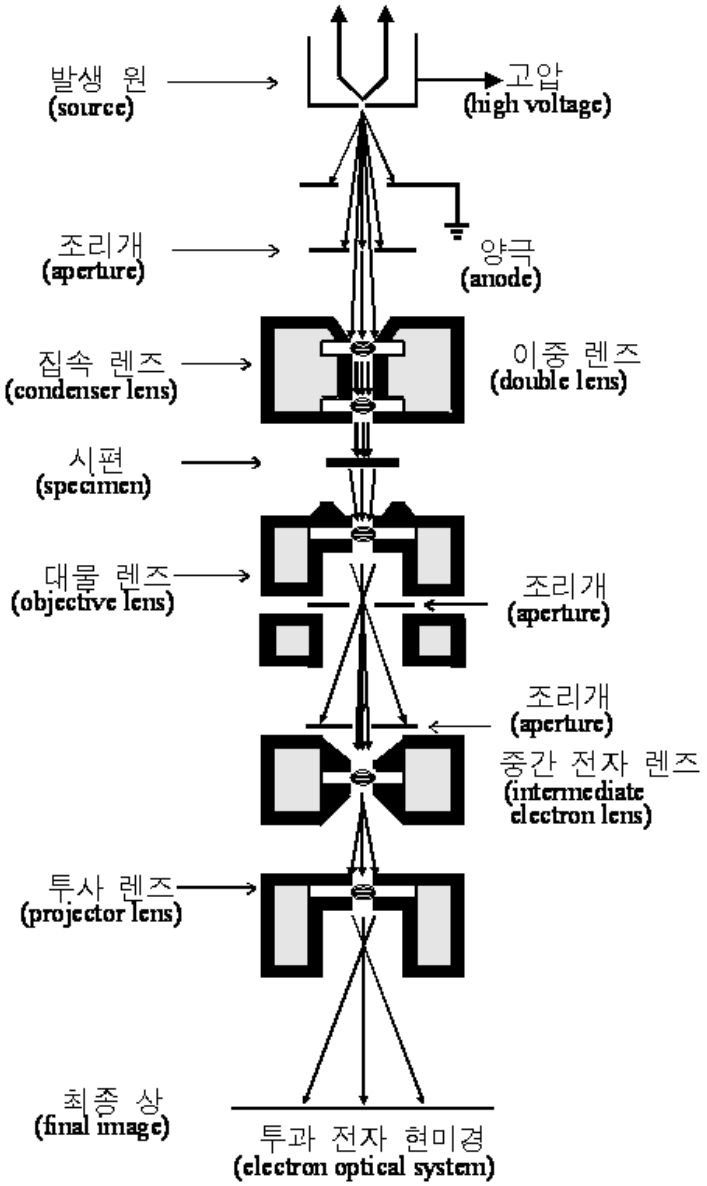
UCRF TEMs Specification

	Advanced TEM	HR-TEM	Normal TEM	Bio TEM
Model	Titan^3 G2	JEM-2100F	JEM-2100	JEM-1400
Acceleration voltage(kV)	60 ~ 300	200	200	120
Specimen tilting(°)	α=±35, β=±15	X=±25, Y=±25	X=±35, Y=±30	X=±25, Y=±75
Cs corrector	Image Probe(On going)	Probe	X	X
Image resolution(nm)	0.065	0.1	0.14	0.2
STEM	O	O	O	X
HAADF resolution(nm)	0.07	0.1	X	X
EDS resolution(eV)	128	128	132	X
EDS window size(mm²)	120 (30 X 4)	80	50	X
EELS resolution(eV)	0.12	0.8	X	X

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

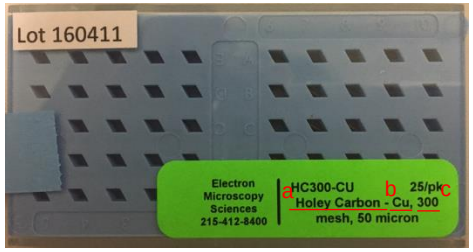
3) Lacey carbon grid - carbon materials

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

e. Drop on your grid 2~3 point

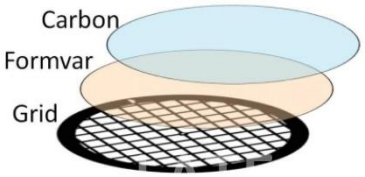
f. Dry

2. Grid description

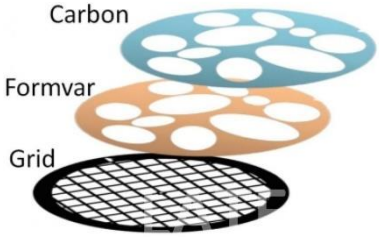


a. Grid type
b. Grid frame
c. How many mesh in grid

3. Grid type



- Carbon film grid



- Holey carbon grid

1. Check the equipment before use

a. Error message

b. Column vacuum

c. LN_2

2. Increase the accelerating HT voltage

a. HT: 160keV → 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

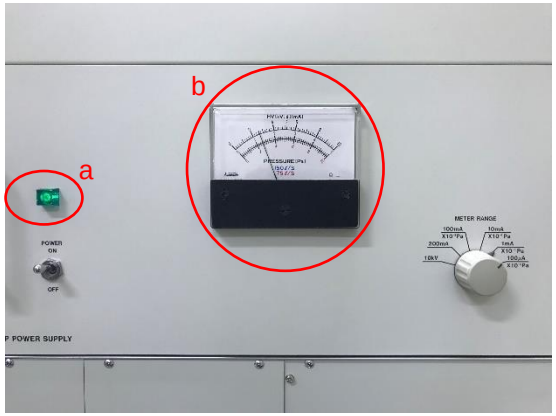
a. Column vacuum

b. Error message

c. LN_2

Check the Column Vacuum

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



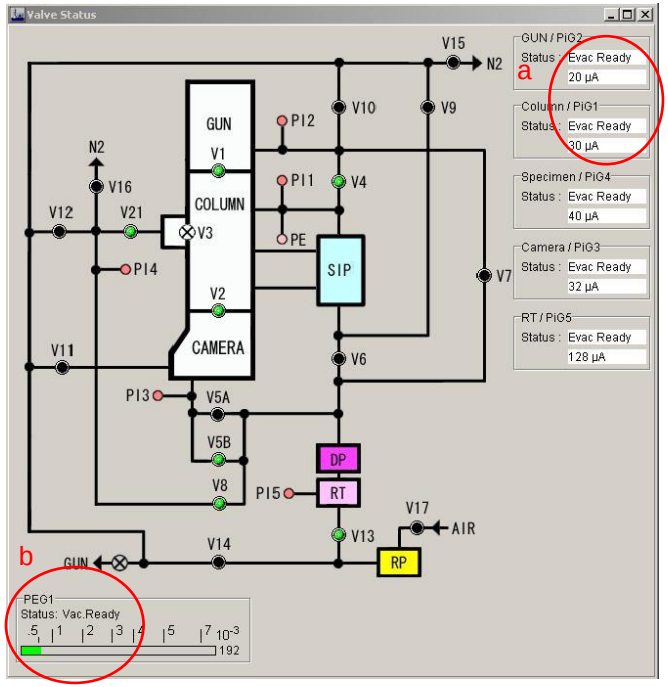
a. Check that the power lamp of the SIP is green

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

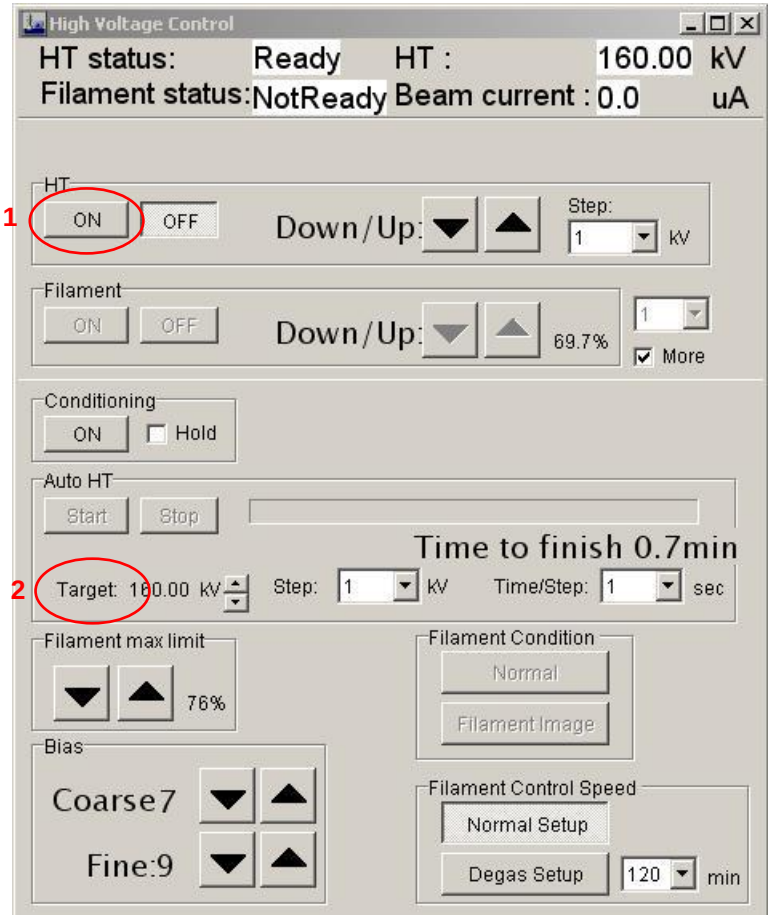
b. Check PEG1

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

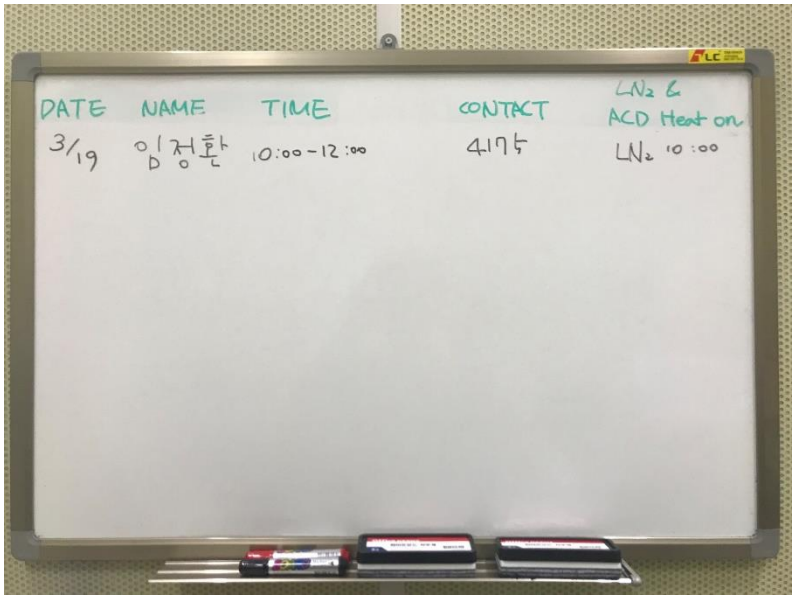
1) Target : 200kV, Step : 0.1kV, Time/Step : 3 sec

2) Click start button of auto HT

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

Check the equipment before use

Check the white board

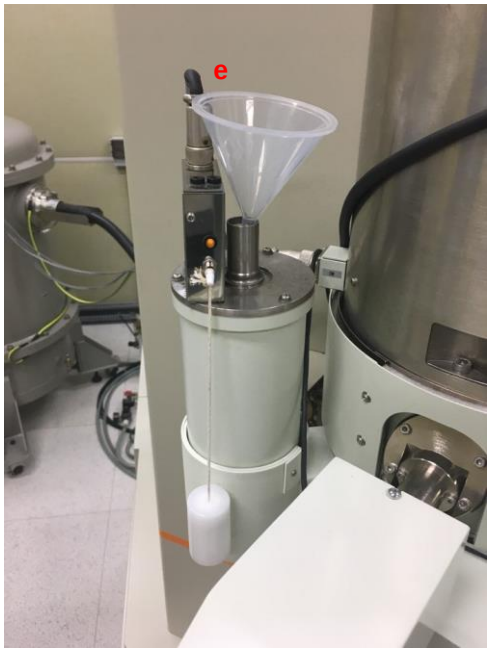


1. **Check notice and the message on the board (If there are something wrong, you can't use the equipment)**

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**

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

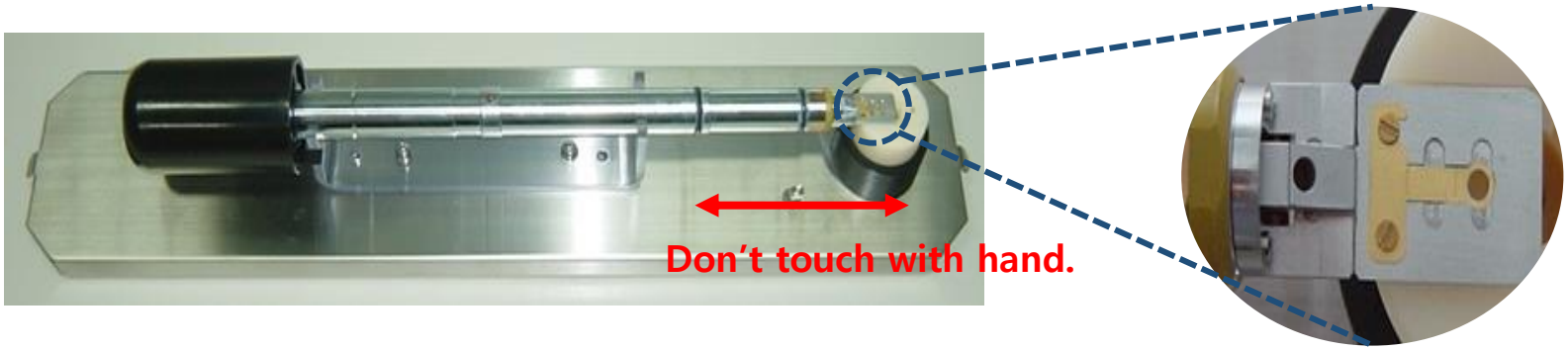
d. Fill LN2 to about 1/3 of the container

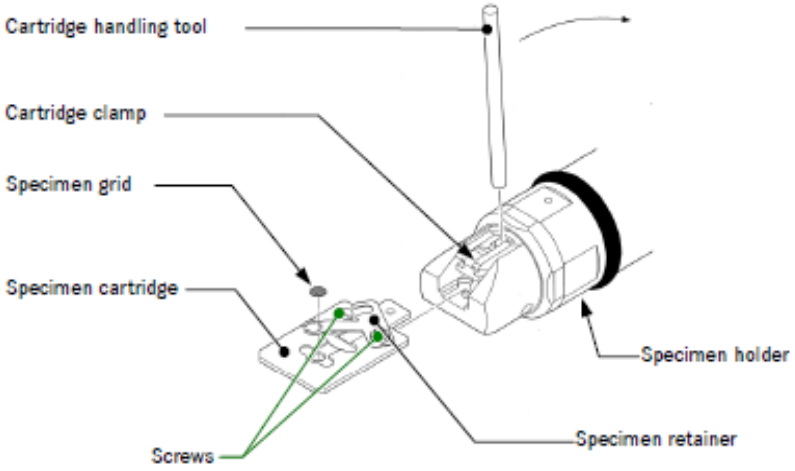
e. Put the funnel into ACD liquid nitrogen tank fill LN2 fully

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

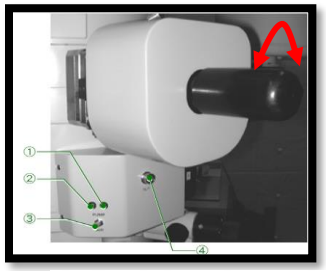
❖ Caution! If you tighten too much the screw, the screw line will be break .

Dry pumping station

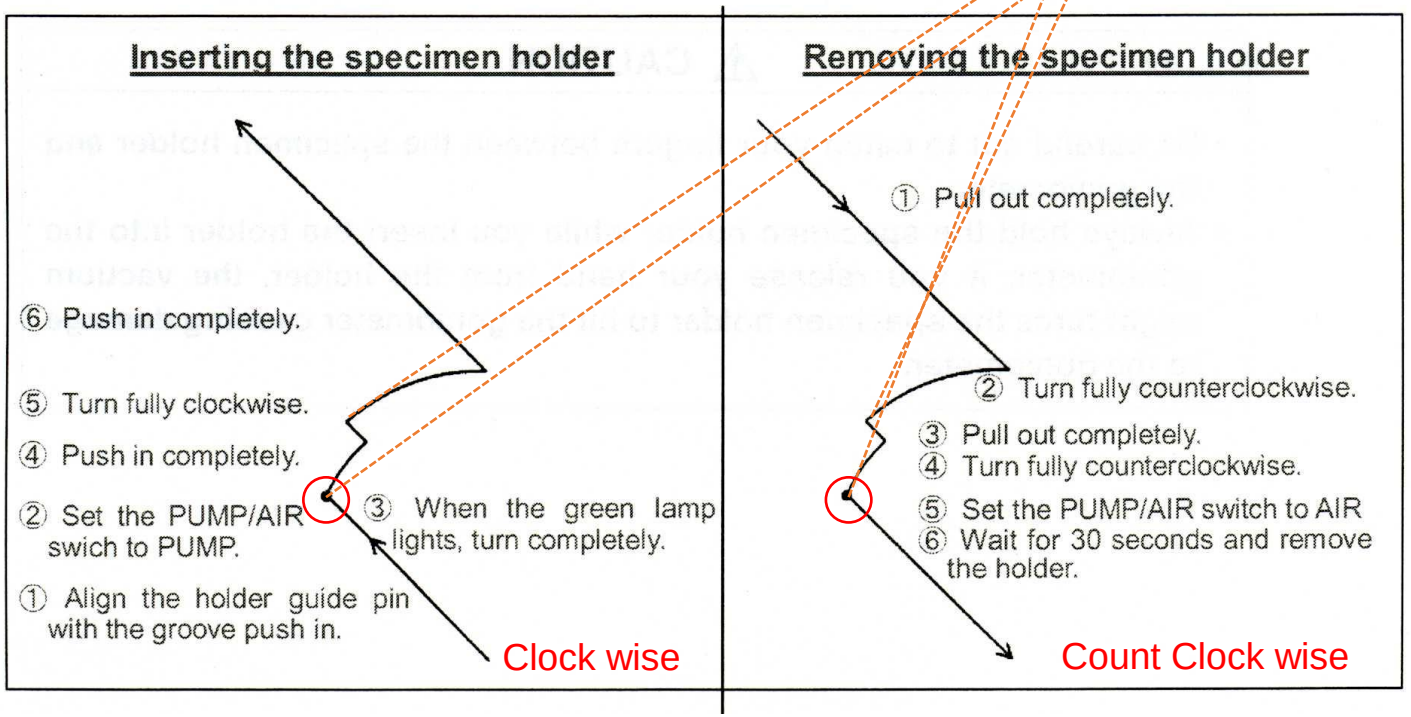
1. Insert the holder
2. Check power on status
3. Click Evac button
4. After 5 minutes, click the Vent button
5. The Vent light will be stop, you can pull out the holder straightly

Inserting the Specimen Holder

1. Inserting the specimen holder



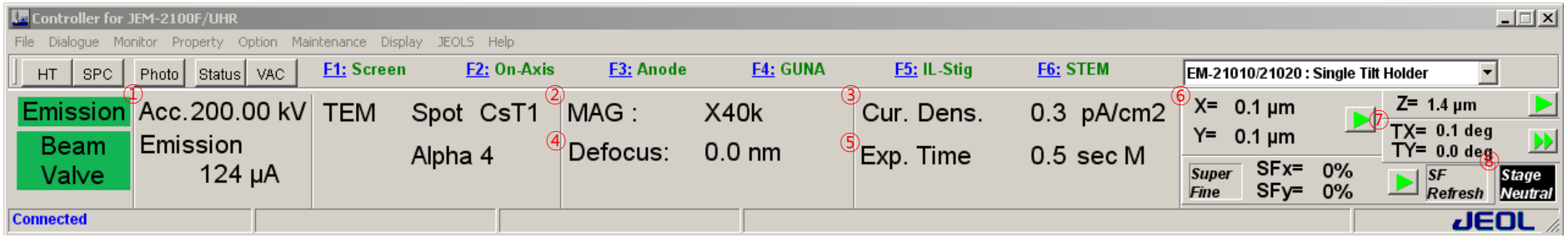
Guide pin



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

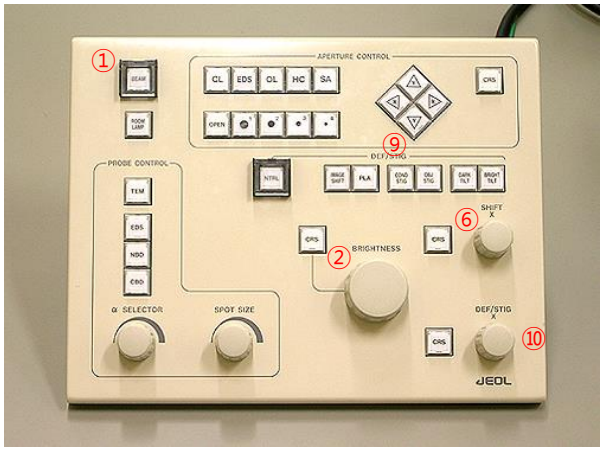
⑥ X, Y, Z: Specimen position in the X, Y, Z direction

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

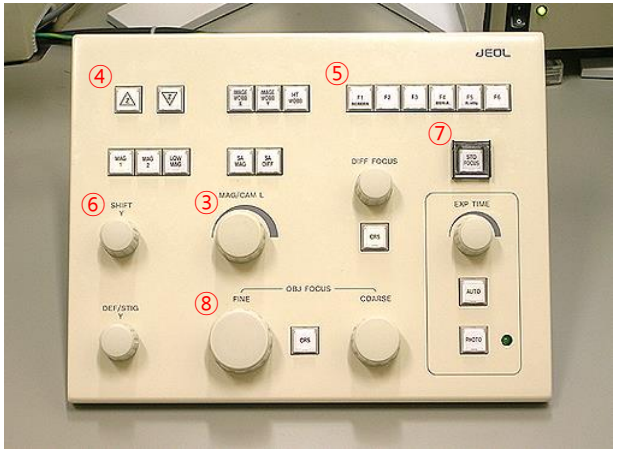
⑧ Stage neutral

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

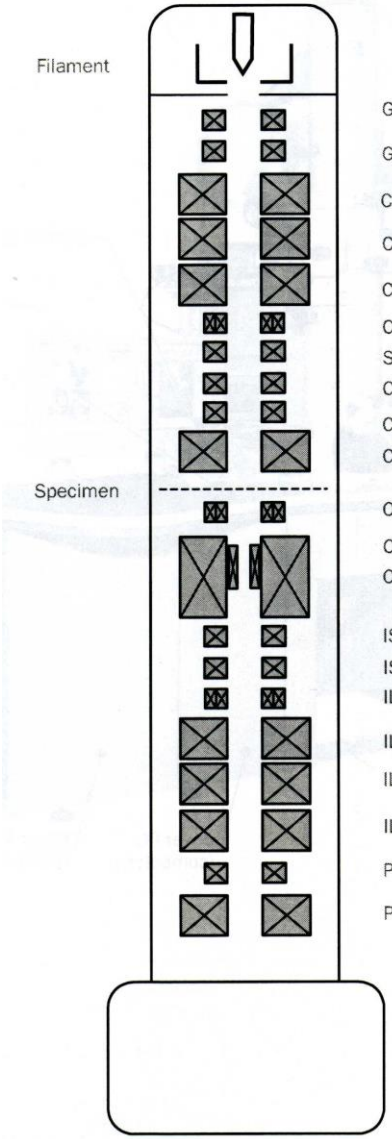
⑧ OBJ/FOCUS: Focuses the image

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

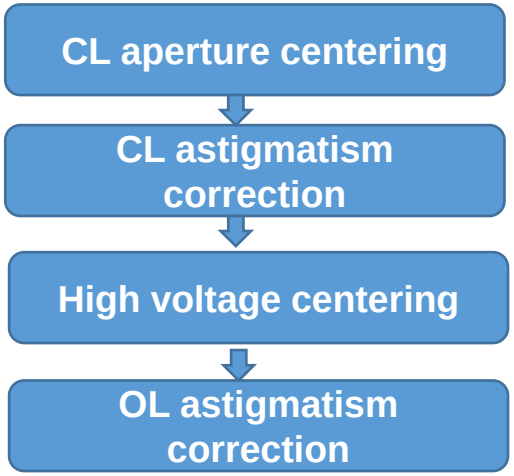


Gun1	Electron gun 1st beam deflector
Gun2	Electron gun 2nd beam deflector
CL1	1st condenser lens
CL2	2nd condenser lens
CL3	3rd condenser lens
CLS	Condenser lens stigmator
Spot	Spot alignment
CLA1	Condenser lens 1st deflector
CLA2	Condenser lens 2nd deflector
CM	Condenser mini lens
OLS	Objective lens 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

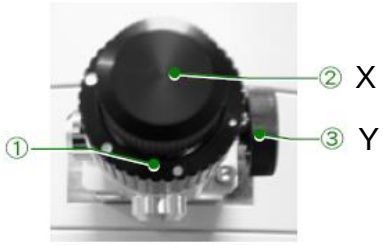
1. Condenser lens aperture centering

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

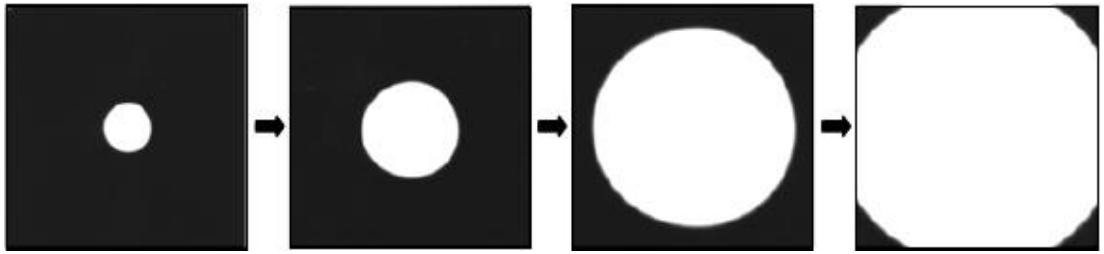


CL aperture X/Y knobs

- 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

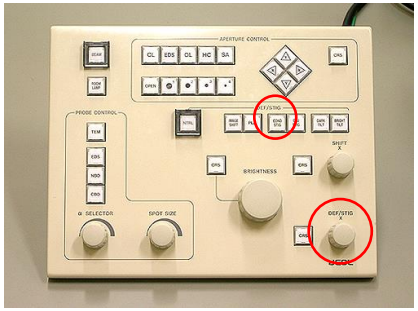
a. Adjust the magnification to over x60k

b. Beam centering

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

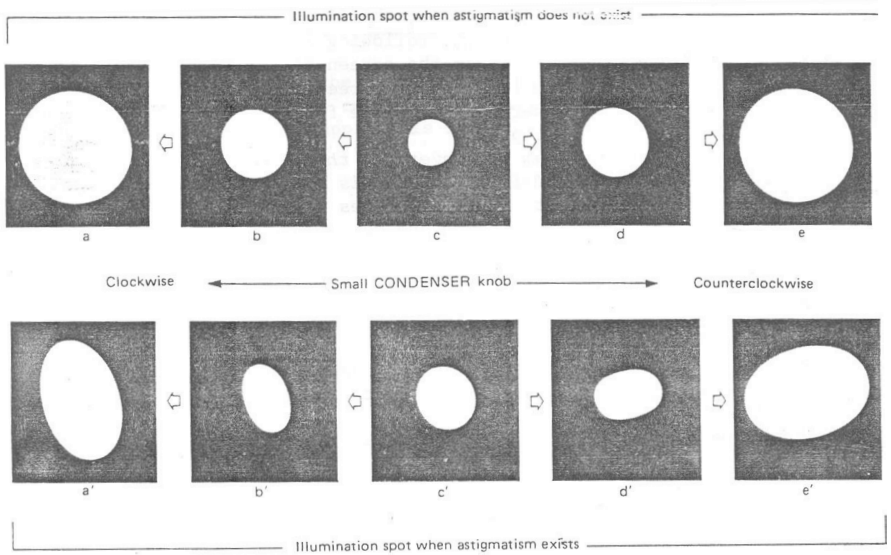
COND(CL) Stig button +

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

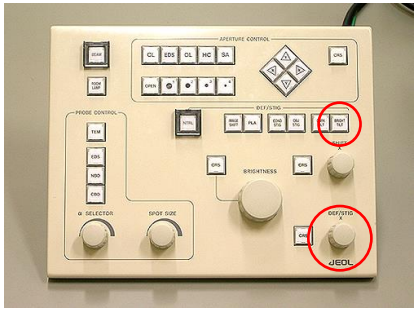
e. Let's make the edge point of the sample do wobbling at the small screen center

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

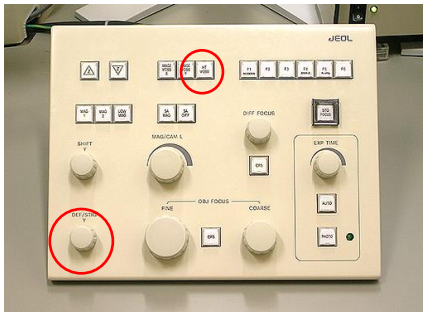
- If HV centering is well aligned, beam is wobbling at the beam center

HT wobbler button +

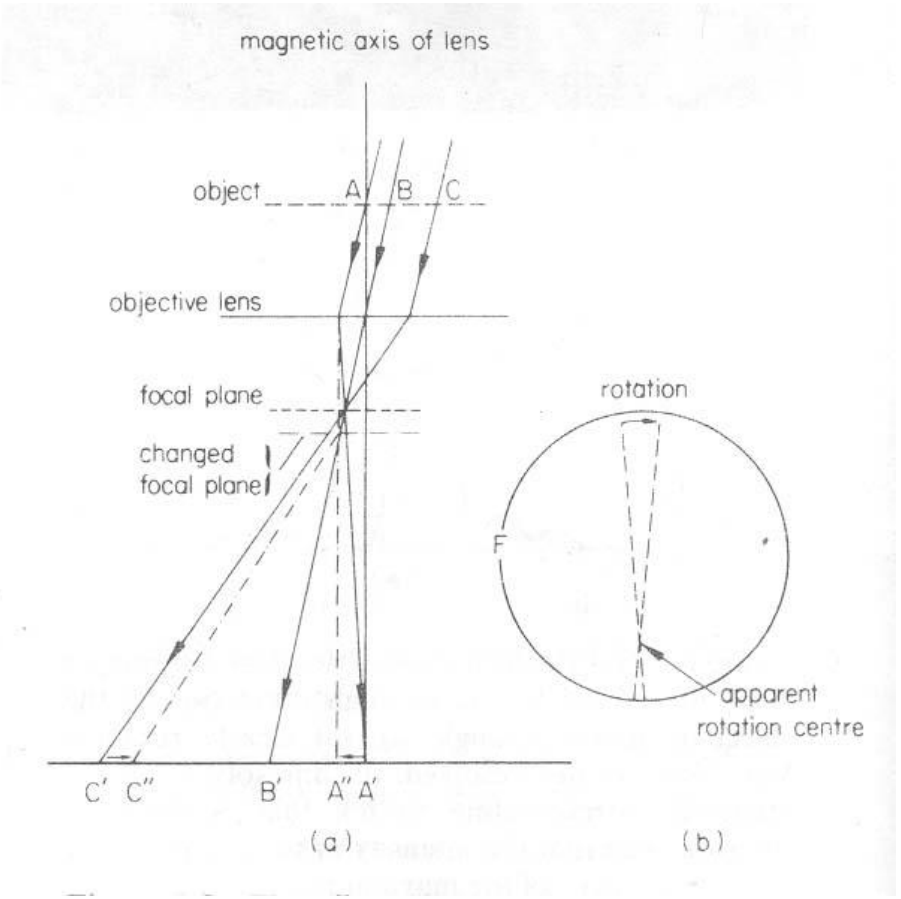
Bright Tilt button + DEF/STIG X/Y knobs



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 F1 button

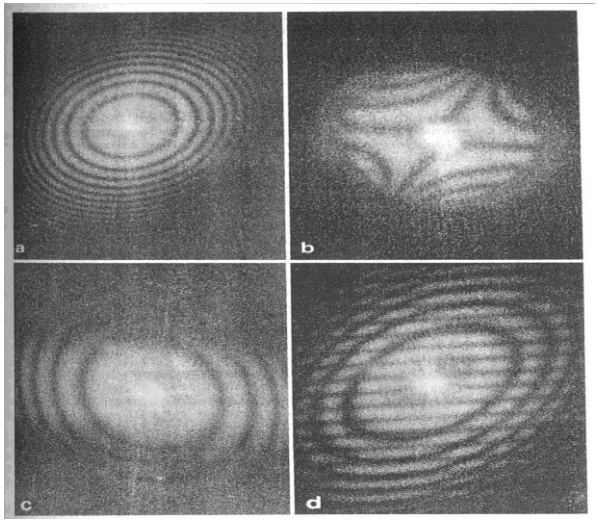
f. Start view and Process-Live-FFT

g. Make the FFT image to be perfect circle

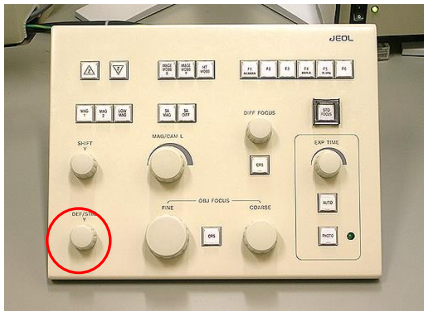
OBJ(OL) Stig button +

DEF/STIG X/Y knobs

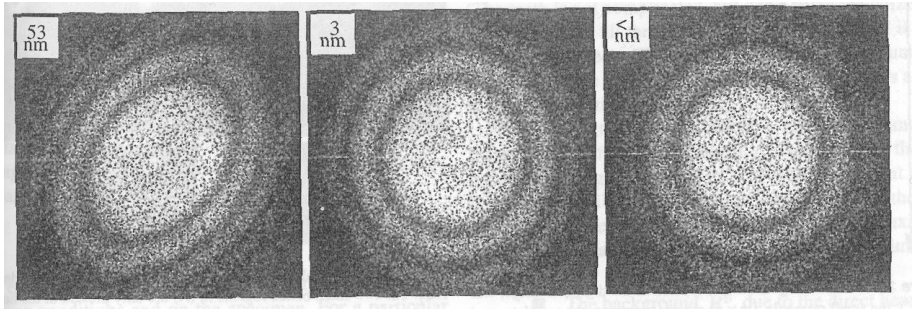
When OL lens is well aligned, FFT shape is circular



Left panel

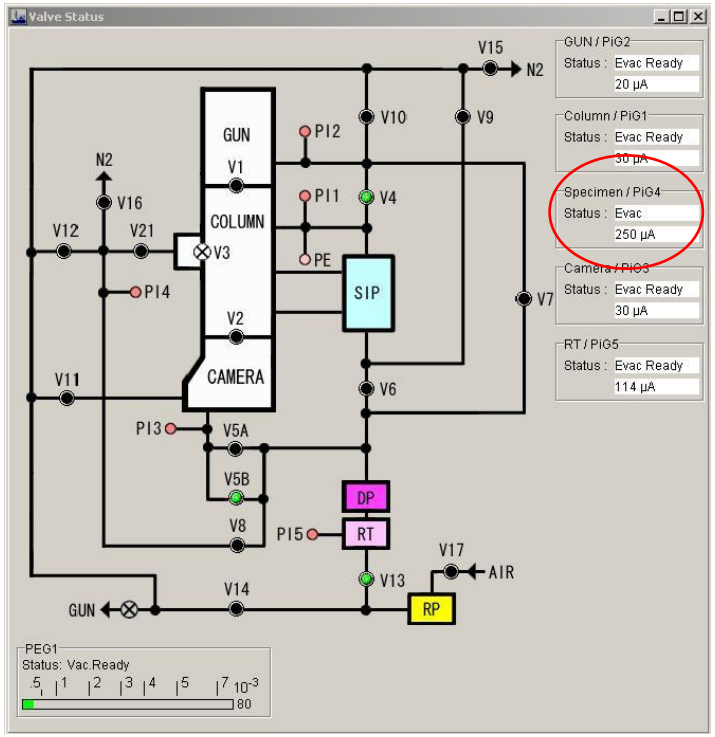


Right panel



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

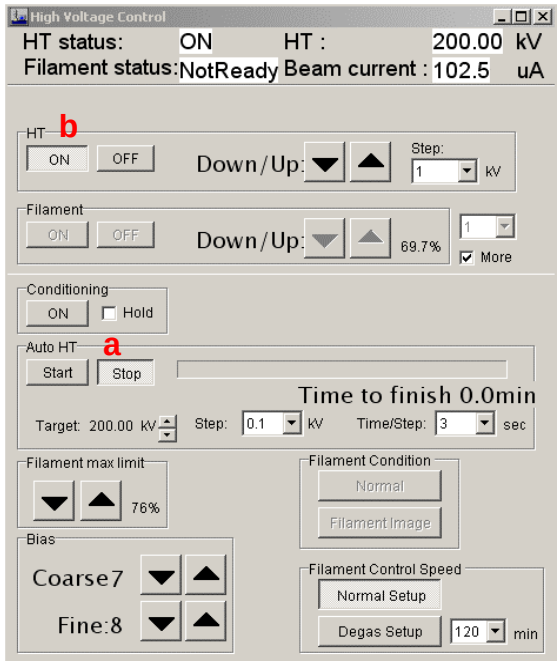
d. You have to wait until the value of specimen is over 220uA

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

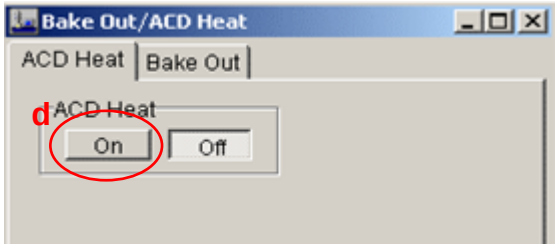
2. Turn off the HT

1) Click HT OFF (b)

- Beam current : 80 uA -> 0 uA

3. Plug ACD heater (c)

4. ACD Heat On (d)



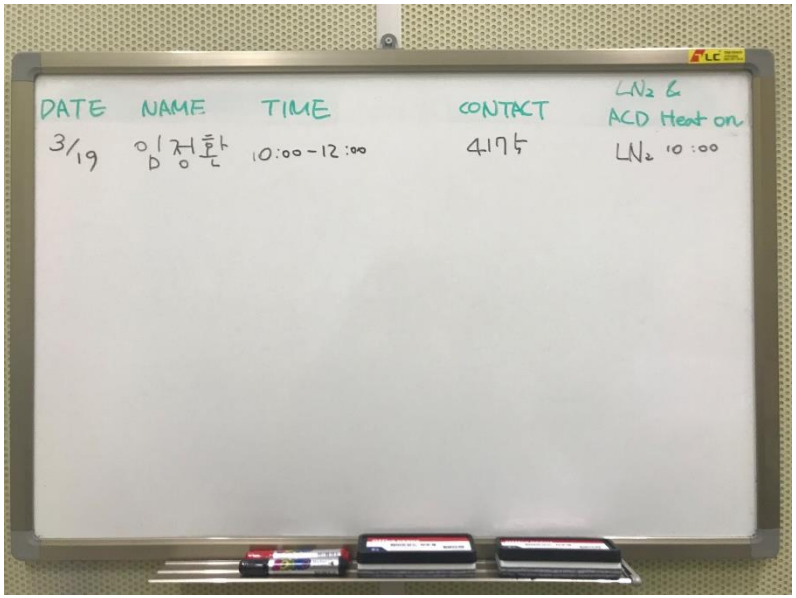
Check the equipment after use

- 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 on the google calendar
      1) 200kV as it is
      2) Refill LN_2
   b. When the blank time is over 5 hours on the google calendar
      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 (22page)
   - 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)	Chaeeun Hong 내선(Extension) (4185) ★ 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