HF2000 Daily Operation Guide

Last modified on Apr. 2004 by Dr. Shuyou Li

Note: This guide is NOT complete operation manual of HF2000. It is only for your daily operation quick reference. For detailed operation manual, please refer to http://www.nuance.northwestern.edu/epic/manuals.htm

Remember:

1. Please remember to send me a copy of your publication (e-format preferred) if you have TEM results taken with this microscope.
2. **Do not open gun valve until all vacuum gauges and pumps work properly.**
3. **In emergency, turn off FE rapidly, then gun valve.**
4. **Refill Liquid Nitrogen if your experiment goes over 2 hours.**

BEFORE YOU START

- Check conditions of HF2000 before login the access control PC.
  - Vacuum gauges (3 Ion Pumps, pressure gauge on control rack~$2x10^{-6}$Pa)
  - Sample position (X=0, Y=0, tilt=0), PEELS switch OFF, HV=200, ANA mode off (left panel).
  - Obj Apertures out, EDX detector out.
- Check logbook to see notes by previous user.

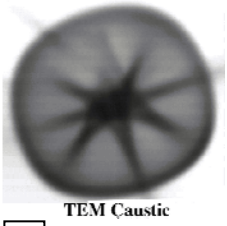
START-UP

1. Fill LN2
2. Log in Access Control with FOM.
3. Load Specimen. Remember to check the O-ring!

Start Emission

4. Wait until pressure reach $2x10^{-6}$ Pa.
5. On Control Rack, press **MANU** to turn it on, then **IF2** (setting data shows ~4.42)
6. Press **FLASH**, keep an eye on V1 and I1 values. At the beginning I1 is very low (~0.08). We repeatedly FLASH the tip until I1 reaches 0.2~0.3. Wait 20 seconds between each FLASH, or press FLASH only when V1 goes back to 0.00.
7. When I1 reaches 0.2~0.3, look at "FLASHING" that appears in the setting display. When the "FLASHING" word disappears and changes to some number, push **FE** button IMMEDIATELY (<10 seconds)
8. Wait until the setting display shows ready (RDY). Record V1 and I1 values in logsheet.
9. Open gun valve (FE control rack) and you should hear it click open.

TEM alignment



TEM Caustic

1a

1. Find beam, increase mag. to 150kx, move sample to a hole, center beam, go to crossover.
2. Remove all apertures. Use condenser stigmator (right sub panel) to make caustic round and use 3rd stigmator (left sub panel) to make it symmetrical, as shown in fig. 1a.
3. Insert a condenser aperture to be used (size 1 or 2). Align aperture position with condenser aperture X/Y knobs. Align stigmatism with condenser stigmator (left subpanel).
4. Set OBJ current at 6.35 and align sample eucentric height.
5. Reduce Mag to 50kx, change wobbler angle to fully clockwise and turn on wobbler. Adjust BTX and BTX vert knobs (left of CRT) until swinging beams overlap. Turn wobbler angle 3 clicks counterclockwise to adjust BTY and BTY vert knobs. Turn off wobbler when done.
6. Turn on either Current Center wobbler (right panel) or HV wobbler (on FE control rack) to align beam tilt. Current center alignment is good for High resolution imaging, while HV center is good for EELS and EFTEM.

GIF alignment and digital imaging

7. Check GIF hardware: GIB=ON; MSC=ON; Cooler=cool; shutter=AUTO; STEM detector=OUT.
8. Turn on TV monitor (Pelco PMM901), Turn on CCD camera (Beam stop=OUT; Camera=OUT; gain=AUTO), Open N2 gas for GIF (main valve only), Start FilterControl and DigitalMicrograph software.
9. TEM mag=10kx, move sample hole to screen center, focus beam to ~6mm, center it to GIF entrance aperture.
10. Make sure STOP light on CRT is lit. Push Photo to raise screen.
11. Insert TV camera into electron path (in FilterControl). Adjust Energy Shift value if no light shown on TV.
12. Click AlignZLP (1min), click TuneGIF (5min). Adjust beam intensity if it is prompted (Press Enter to exit intensity adjustment window). If emission current too low, press I1c (FE control rack) to increase. After push I1c "RDY" will not come back but you should wait until emission is stable (5 minutes).
13. Find amorphous area on specimen, center on TV monitor, click Preview-idle to view CCD image on PC. Use live reduced FFT (process menu) to correct obj stigmatism. Insert TV camera to relocate sample if necessary.
14. Alt-Acquire to adjust exposure time, then click Acquire to record image. Save image on your own folder in DM3 format.

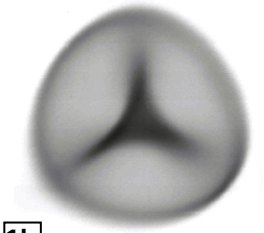
EFTEM (energy filtered imaging)

15. Zero-loss imaging: choose slit width (in FilterControl, ~20eV). Insert slit. If image disappears redo Align ZLP.
16. Low loss imaging: With slit in, change Energy Offset to a desired value (20-70eV). Refocus image if necessary.
17. Elemental mapping: Align ZLP again. Hold Alt key, Click Filer-Elemental mapping in DM window.

EELS in imaging mode

18. Click EELS to switch to spectrum mode. Alt+click Turbo button to set exposure time (start with 0.005s).
19. Select desired dispersion and entrance aperture size. Click Idle button to view EELS on PC.
20. Insert TV to view shape of ZLP. Adjust Focus X, Focus Y, ACComp to get ZLP. Switch off CRT if necessary.
21. Remove TV camera and view EELS on PC. Change exposure time with up and down arrow keys to get enough counts and keep spectrum in green. Hit spacebar immediately if the spectrum turns into yellow or red.
22. Change Energy offset for core-loss spectroscopy.
23. Alt-click Acquire button to change acquire time. Click Acquire to record spectrum.

ANA mode Alignment



1b **Analysis Caustic**

1. Push ANA button to turn on ANA mode.
2. Mag=150kx. Find a sample hole. Remove all apertures, center beam and go to crossover.
3. Use condenser stigmator (right sub panel) to make caustic round and use ANA 3rd stigmator (left sub panel, different from TEM knob!) to make it symmetrical, see fig. 1b.
4. If caustic moves when changing Brightness, note the direction of the caustic movement when going to crossover. Use the beam tilt knobs (right sub panel) to shift the caustic in the same direction of the movement and brightness centering knobs (right panel) to return the caustic to the center of the phosphor screen. Recorrect both 1st and 3rd order condenser astigmatism.
5. Repeat last step until caustic does not move with directionality when passing through crossover. This correction requires significant beam tilting if the previous operator used the TEM alignment procedure. You may need decrease magnification to 70kX or less to make the beam tilting and condenser astigmatism corrections easier but complete your final corrections at high magnification.
6. Insert condenser aperture of choice (3 or 4) and "center" by minimizing the tails of intensity surrounding the central bright spot.

EDS spot analysis Note: you cannot get EDS signal under TEM mode. You must use ANA mode instead.

7. Turn on STEM electronics-mag. Selector (3 or 10), turn on Gatan STEM interface
8. Insert EDS detector. Turn X-ray top-hat aperture lever to left to insert the aperture. Turn off Vacuum gauge filament on HV control rack.
9. Start EDS software. You may use INCA (simple but no linescan function) or ES vision (more complicated)
10. Focus beam on sample area of interest, collect EDS specutra.
11. Retract EDS detector. Remove X-ray aperture. Turn Vacuum gauge back on.

STEM imaging

12. Turn on STEM electronics-mag. Selector (3 or 10), turn on Gatan STEM interface
13. Switch Monitor to Emispec PC. Open EsVision software. Select desired work place.
14. Move sample edge to screen center, adjust z-height to focus at 70kx at objective lens current 6.35, fine tune image focus with focus knob (right panel), use stigmators (left sub panel) to correct objective astigmatism.
15. Converge beam to smallest size at high mag. in ZOOM mode, start STEM scanning, make sure beam rasters between specimen and vacuum.
16. Change to DIFF mode, set camera length to 0.20m, use microscope keypad to highlight (asterisk at left) I2 current, turn diffraction spot knob to change I2 strength to 1.75. Highlight P2 and change P2 current to 1.75.
17. Highlight I1 and use diffraction spot knob to minimize the movement of diffraction pattern.
18. Use INT ALIGN (black box) to move transmitted spot 1mm southeast of crosshair, make sure STOP is ON (under CRT), press PHOTO to lift screen.
19. Insert a STEM detector (BF or DF on STEM aperture controller), image will appear on computer, use Gatan-STEM box (above PEELS equipment) to adjust contrast and rightness.
20. Use INT ALIGN to center diffraction pattern exactly on STEM detector.
21. Use the microscope focus knob to produce the sharpest image detail.

EELS in STEM/nanodiffraction mode

22. Make sure STEM detector is at DF or OUT position (BF detector will block GIF entrance aperture).
23. Stop STEM scanning. Spot beam on STEM image with beam spot locator.
24. Set Turbo exposure time to 0.005s, set dispersion, aperture size and start acquisition.
25. Carefully adjust INT ALIGN to get highest spectrum counts.
26. Adjust Focus X/Y, ACComp if necessary. Use smaller condenser aperture or GIF aperture if CCD saturated.

EDS in STEM/ANA mode

27. Get an STEM image and the cross-hair tool to locate beam on STEM image/sample.
28. Series EDS spectra along a line (linescan) can be done in "line profile" work space of EsVision software.

Shutdown

1. Remove EDS detector, Obj aperture, Turn off GIF CCD camera control unit and TV monitor, Close GIF N2 gas.
2. Turn MAG down to 8000, center beam and spread beam to cover whole screen.
3. Move specimen to origin: X=0, Y=0, Tilt=0, Then turn MAG to 60K
4. Press FE to turn it off. Close gun vlave.
5. Remove specimen, return specimen holder to standby position.
6. Record usage in paper logbook and log off Access Control PC.
7. Vent Camera chamber and exchange negatives (40 pcs total) if films are used.