

## HD-2300A Daily Operation Guide

Last modified on October 2015 by Dr. Jinsong Wu

Note: This guide is NOT complete operation manual of HD-2300. 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 send a copy of your publication (e-format preferred) to Jinsong (jinsong-wu@northwestern.edu) if you use any TEM results taken with this microscope! We highly appreciate if you include NUANCE Center in the Acknowledgement.
2. Adjust the alignment settings ONLY when necessary.
3. Refill Liquid Nitrogen if your experiment goes over 2 hours.

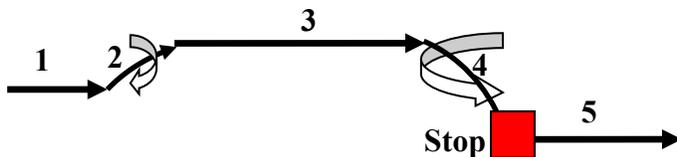
### BEFORE YOU START

- Check conditions of HD-2300A before logon the access control.
  - Vacuum statue (Turbo: normal; IP1: <8E-9; IP2: <6E-8; column reading: <6E-5 Pa); (On the main menu, select [Setup] and then [Evac Control])
  - Sample position (X=0, Y=0, tilt=0); (On the main menu, select [Operate] and then [Stage Control])
- Check FOM-online to see notes left by previous user.

### START-UP

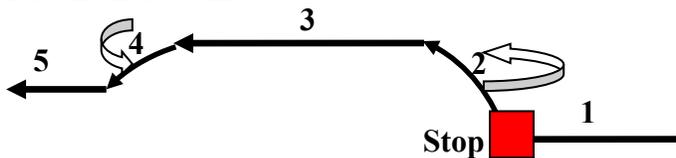
1. Fill LN2, Log in Access Control in FOM, <http://www.fom.northwestern.edu/>.
2. Load Specimen. Remember to check the O-ring!

### Take the holder out



- 1: Gently pull it out till it stops
  2. Turn it 15° clockwise
  3. Gently pull it out till it stops
  4. Turn it 30° counterclockwise till it stops.
- Stop:** Set the EVAC-AIR switch to AIR.
5. Gently pull it out straight (so we do not bend the holder).

### Put the holder in

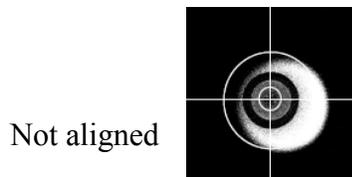


- 1: Gently push in till the holder stops
- Stop:** Set the EVAC-AIR switch to Evac.
2. Once green light on (alarm on), turn it 30° clockwise.
  3. Let it slide in (hold it so it can slide in gently).
  4. Turn it 15° counterclockwise
  5. Let it slide in gently.

3. Turn on HV: Open “HV control” menu (On the main menu, select [Setup] and then [HV Control]), choose kV (80, 120 or 200 kV) and click “HV on”. If you wish to change to 120 or 80 kV: click “Standby” firstly and then select 120 kV (or 80 kV) and click “HV on”.
4. Open gun valve: turn the “GV Lock” switch from close to AUTO (open).

### Column Alignment

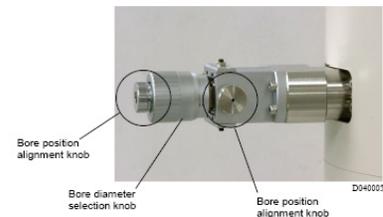
5. Alignment of movable aperture position
  - On the main menu, select [Operate] and then [Alignment], select ‘Aperture alignment’ and click “On”.
  - By moving the aperture (turn the two knobs on the aperture) to center it. Click “off” when it is done.



Not aligned



aligned



6. Set up Eucentric height: Find a suitable area; ABCC (auto adjustment of brightness, contrast); Reset lens current and adjust height to focus.



Lens reset .....Resets the lens current (C1, C2, OBJ and PROJ).

7. Optional Auto Alignment (On the main menu, select [Operate] and then [Auto Alignment Mode])

- current center alignment: select “SE” icon on the toolbar; set magnification to 20K-200K; adjust focus (you can use Auto Focus function); select [SE alignment] and click “Start”.

ABCC button



or Area ABCC button



- Auto compensation for image escape in astigmatism correction: this can be done in any of detection mode: SE, ZC and TE; to get a clear and focused image and then select [Stigma alignment] and click “Start”.

- Auto centering of visual field (in TE imaging mode): Click on “TE” icon to choose TE mode; select [**Bright area centering**] and click “Start”.
  - Focus: mag>50K, select [**Focus**] and click “Start”
  - Stigma: mag>50K, select [**Stigma**] and click “Start”.
8. Fine Column Alignment (manually)
- Find an interesting sample area and go to desired magnification. Go to low-magnification mode if necessary.
  - On the main menu, select [**Operate**] and then [**Alignment**], select ‘Column alignment’ and click “Start”.
  - Lens ISF reset and sample height adjustment:  
 **Lens reset** .....Resets the lens current (C1, C2, OBJ and PROJ).
  - Turn Z-control knob to adjust the sample height to focus (Eucentric height).
  - Click “Next” and followed the instruction in the menu to do fine “AL” alignment (aperture alignment): by turning the two Stigma knobs to stabilize the image.
  - Click “Next”, by turning the two Stigma knobs to stabilize the image when TEM starts stigma coil modulation along X.
  - Click “Next”, by turning the two Stigma knobs to stabilize the image when TEM starts stigma coil modulation along Y.
  - Click “Close” once you get to the last step.
  - Go to “TE” mode; go to 2-4K mag; select “DEAL Alignment”; center the bright spot by using the two multifunctional knobs.
9. Optional Auto Alignment wizard: select [**Operate**] and then [**Alignment**], select ‘Auto alignment’ and click “Start”. As guided by the wizard, you can easily carry out the alignment to astigmatism correction.
10. Astigmatism adjustment by Ronchigram: Under Gatan Digitalmicrography, find “Camera View” sub-window, make sure explosion time is “0.001” second, click on “Start View”. Click on “yes” to insert camera if the camera is not inserted. Retract the TE detector by checking “TE detector out”. Switch to spot scanning mode, use condenser aperture No.1, turn focus knob to have a clear Ronchigram; adjust Stigm knobs to make the Ronchigram as round as possible; point the mouse to the center of the Ronchigram; use a smaller condenser aperture and center it. Now we can switch back to normal scanning mode. Select “TE” detector; go to 2-4K mag; select “DEAL Alignment”; center the bright spot by using the two multifunctional knobs.

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### Image Observation

11. Scan speed settings: fast (TV rate) and slow scan mode: from 0.5s/frame (slow 1) to 20 s/frame (slow 4).
12. Selection of image observation signal: SE (secondary electron image), ZC (Z contrast image) and TE (phase contrast image).
13. Image movement by means of Image Shift: the Image SHIFT knobs. ISF reset (right icon underneath the “RESET” to reset once it is done).
14. Raster rotation: The direction of images can be adjusted by rotating the raster scan direction of the beam. Click the [Raster rotation button] or select [**Raster rotation**] from [**Operate**] menu.
15. Focus and stigma: can be done manually or automatically (see7).
16. Different lens operation modes: [**Setup**] menu select [**Column setup**]: (when you change the mode, you have to do the alignment again).  
 In terms of probe current: Decan>EDX>Normal>Nano-diffraction>High Resolution>Ultra-High Resolution (more probe current, more beam damage).  
 In terms of image resolution (from the best to the worst): Ultra-High resolution>High Resolution>Normal>Decon.  
 Decon is the de-contamination mode used to reduced contamination;  
 Nano-diffraction mode: Probe has a small convergent angle to see clearly the nano-diffraction patterns (by using live-diffraction camera).
17. Select right condenser aperture: Aperture 1 (150 um) > 2 (75 um) > 3 (50 um) > 4 (30 um); the bigger the aperture, the larger the probe current.
18. Select appropriate camera length: [**Setup**] menu select [**Column setup**]: in ZC mode, you can use either ZCM (Z-contrast; 62-330 mrad) or DCM (diffraction contrast; 46-245 mrad); in TE mode, you can use either PCM (phase contrast; 3 mrad) or WAM (wide angle; 30 mrad).
19. Nanodiffraction: change operate mode to “Nano-diff”, under PDS load in the alignment data for nanodiffraction at current kV; in STEM image to adjust focus (or sample height if it is necessary) to get a clear image, use the Gatan diffraction camera (as instructed in the next page to obtain a Ronchigram) to get a diffraction pattern, switch to spot scan mode and put beam on the spot you wish to get nano-diffraction. Use “Hitachi-Nano” as password to switch to the Staff Mode and open the “Lens Control” menu (if it is not open). In “Lens Control” select “Proj”, and use the focus knob to adjust the projection lens current to have an appropriate camera length. In DM software click on ‘acquire’ to acquire a diffraction pattern and save the diffraction pattern.

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### Take Picture

20. Set capture conditions: from [**Setup**] select [**Image setup**]: you can change the capture speed and image resolution.
21. Click the capture icon to record and then save the file to the TEM Server.

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### EDS Spectra collection and spectral imaging

22. On the Thermo computer, open the software: NSS; select your Project or set up a new one.
23. Insert both of the EDS detectors; Put in EDX aperture ([**Setup**] menu select [**Column setup**]). Without the aperture, all you got is the noise from the grid!
24. In NSS software, select “Both detectors”. Click on the yellow “Acquisition Property” icon, set up collection time for both detectors, set “low energy cutoff” as either 100 or 200 eV.
25. Find the interested area and select “Spectrum” icon in the NSS to collect an EDX spectrum; You can use other functions like “Point and shoot” and “Line Scanning”; Select “Spectral Imaging” to do spectral imaging.
26. Save your data. You also can generate a report by clicking the “Export to Word” button. The EDS spectrum can be saved as JPG file by highlighting the spectrum and then choosing “Copy To”.
27. Please retract both detectors and take out the EDX aperture once you are done.

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### EELS Spectra collection and spectral imaging

28. Gatan: On the Gatan computer, open the software: DigitalMicrograph (DM); In 'Help', select "Power user" for user mode; In "Window", click "floating window layouts" and select "EELS-2012". On the "Enfina Control", type in beam energy (EELS is only aligned for 200 and 80 kV).
29. STEM-Control: Click on the red EELS button to select the EELS mode (in the Hitachi STEM control);
30. STEM-Control: Select UHR lens mode (to have a small probe); Find the area of interest and focus.
31. Gatan: In the Digscan window (of Digitalmicrograph), click "Search" to scan in a STEM image; Click on "Stop" to stop scanning and click on the beam control icon (red triangle) to park the beam to the interested area where a spectrum will be collect.
32. Gatan: On the EELS (in Digitalmicrograph), select 1 mm aperture, use 0.0025 s as the starting exposure time, and 0 as energy-loss, click on view to look at the zero-loss peak. (Convergent angle: aperture 1,2,3,4 – 24, 12, 8, 5 mrad; collection angle: 1,2,3,5 mm – 7, 14, 21, 30 mrad; please do not use 5 mm aperture: CCD will be damaged!).
33. Gatan: click on "Align" in the "EELS Auto Tune" to set zero for the zero-loss peak.
34. Gatan: On the "Enfina control", double click on "Focus-x" and then press on left or right arrow to focus (make the zero-loss peak as thin, as high as possible). Then repeat this with "Focus-y" to make the image on 2D view as a straight line.
35. Gatan: Set up "Acquire" condition and acquire a zero-loss EELS spectrum; Set up "energy-shift" and gradually increase exposure time to record a core-loss spectrum. You may select a larger EELS aperture different dispersion (ev/channel) for the core-loss spectrum. The residual left by strong zero-loss peak can be removed by select "Remove dark reference" in the "Camera".
36. Gatan: Get an STEM image; DM – Spectrum Imaging tab, click Assign Image; Draw a line or a subarea for spectrum imaging (collect set of EELS/EDS spectra), click Assign ROI; Click SI setup button at lower-right corner of Spectrum Imaging tab, set parameters for Spectrum Imaging. Note the total time needed for full set collection; Click Start button to start SI imaging.
37. Gatan: Get maps: Use "Spectrum picker" tool to pick up the spectrum, Click "Quantification" in the "EELS" (DM), select the core-loss edges to be analyzed; set up the background for the core-loss edges; click on "Qualify" in the "Quantification" window to get the maps.
38. Gatan: Release the beam-control (click on 'green triangle' icon to let it turn red) to let Hitachi to re-control the beam.
39. Gatan: Saved your data as .dm3 format and the data can be open in the TEM Server where DM is installed.
40. STEM-Control: Click on "EELS" icon again to quit the EELS mode (and return to the normal TEM mode).

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### STEM shutdown

41. Zero the stage: from [Operate] menu select [Stage control], set "Auto Drive" x to 0 and y to 0, click 'Start'.
42. For holder tilting along X and Y, you need manually tilt them back.
43. **CLOSE GUN Valve!!** (close gun valve whenever you need to change samples too).
44. take specimen holder out of the column; Remove specimen and insert specimen holder back to STEM.
45. If you are the last user of today, go to [HV control] menu and click "Stand By" (put HV in the stand by mode); turn off both monitors.

### Appendix: How to get a Ronchigram with Gatan diffraction camera?

- 1). At magnification 80K or higher, find an area of amorphous carbon (e.g. carbon supporting film).
- 2). Click on "Spot Scan" button twice to freeze the beam
- 3). Retract the TE detector, TE aperture and ZC detector (if any of them is in)
- 4). Under Gatan Digitalmicrograph, find "Camera View" sub-window, make sure the camera is "Orius"; make sure explosion time is "0.001" second, click on "Start View". Click on "yes" to insert camera if the camera is not inserted.
- 5). Retract the TE detector by checking "TE detector out".
- 6). Use condenser lens aperture No.1 (turn the knob to select aperture 1)
- 7). Adjust "Focus" knob and move beam to a fresh spot (or move specimen slightly) to get a clear Ronchigram. If the Ronchigram is not in the center, try to select "DEAL Alignment" for the multifunction knobs and center it.  
How to change diameter of the rings in a Ronchigram? You can adjust "Focus" knob (when it is selected for obj lens, which is the default for the "Focus" knob).  
How to change magnify/reduce Ronchigram as a whole? Click on "set-up", select "lens Control"; in the Lens Control menu, click on "proj" to select projection lens; now when you turn "Focus" knob, the Ronchigram will be magnified/ or reduced.
- 8). Select "stigma" the multifunctional knobs, to make the Ronchigram as round as possible.
- 9). Point the mouse to the center of the Ronchigram
- 10). Put desired condenser lens aperture back (normally 2 or 3) and center it to the point where the mouse is.
- 11). Done!
- 12). Click on "Stop View" to stop the diffraction camera; Click on and uncheck "Camera Insert" to retract the camera.
- 13). Click on "ZC" on Hitachi STEM control to put in ZC detector. Click on "Normal" scan button to get back to normal scanning mode (quit from spot scan).