

FEI Titan 60-300 Image Corrected STEM

Standby Condition

- HT setting at 300kV, **Col. Valves Closed**
- **RESET Holder** and remove sample.
- Mag ~ 5-10kX
- Objective and SA apertures out, C2 aperture at 150 μ m
- Record filament time and negative numbers.
- Computer monitors and room lights off.
- Run **Cryo Cycle** (unless someone will be following you on the microscope within 3 hours.)

If you step away from the microscope select **Col. Valves Closed**.

Startup

- Fill dewar and let chill for 15 min.
- Log into Windows, start Titan 80-300 **LAUNCHER** software
- Record starting time on logsheet.
- Load sample
- Verify Column Vacuum (IGP1) < 20 before opening Column Valves. Normal value is 6.

Sample Loading/Unloading

Loading

- Load sample in rod. (Be sure that the small end of the nut is against the sample.) Do NOT overtighten; use only your fingertips. Wear clean gloves!
- Verify stage coordinates are all zero; align pin (~ 5 o'clock position) and insert rod in airlock. Be sure the O-ring has seated and pin is in slot.
- Select the appropriate holder (usu. FEI Compustage, double tilt), press \downarrow , connect goniometer plug (for the DT holder), press \downarrow .
- Wait for vacuum cycle to finish (3 minutes) and the red light to go out (V8 = closed), then **immediately** rotate the sample rod **CCW**. Watch Column vacuum (IGP1) value while rotating the holder to the insert position. Slide the sample rod in to the viewing position slowly. Hold the sample rod with your finger tips (3 fingers) to minimize the applied force.

Unloading

- Select **Col. Valves closed** and **Reset Holder**.
- Gently pull the holder out from the microscope until it stops. Be careful not to allow lateral deflection.
- Do not keep pulling on the rod once you have reached the end of its travel. After rotating the sample rod fully **CW** to close airlock door, break the vacuum seal by holding the rod and pressing against the plate with your thumb. DO NOT apply any net force to the goniometer.

NEVER move the sample rod with **Column Valves open**.

Alignment

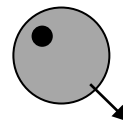
Each user inherits the Supervisor alignments. These are superseded by alignments the user saves. You can load a set of alignments from the Alignment workpanel and FEG register values from the FEG Register workpanel. These can be saved on exiting TUI and will be automatically recalled on the next login.

For basic work, you will only need to adjust

- FEG and C2 aperture
- Sample eucentricity
- GIF and CCD camera (if needed)

Gun Fine Alignment

- Select/load desired parameters from FEG register.
- Select and center largest C1 and C2 apertures
- Set sample to eucentric and focus.
- Remove sample or put beam through hole. (If you remove the sample, close V7 first!)
- Set Mag ~ 115kX
- Set C2 slightly stronger than crossover to see the bright spot within the illumination disk.
- Center the bright spot within the illumination
 - Center C2 aperture
 - Stigmatize C2
 - Shift beam with **Gun Tilt** in arrow direction to center the bright spot
 - Correct **Gun Trans** at spots 1 and 9 (if needed)
 - Repeat until satisfactory
 - Insert and center desired C1 and C2 apertures.



Digital camera

The CCD camera is located at the far end of the GIF. You need to verify the beam passes through the GIF before using the CCD. Also note that the CCD viewing area on the screen is about 2mm diameter, centered 1mm inside the small circle at ~10:00 position.

Set **Preview** mode to 512 x 512 imaging (full CCD, binning = 2, 0.5 sec) and **Acquire** mode to 1024 x 1024 (full CCD, binning = 1, 2 sec). With these settings, you will have the same field of view and exposure between **Preview** and **Acquire**.

Basic Imaging

- Select **EFTEM** mode and lift the screen.
- Select **Align ZLP**
- Verify that the **CCD/TV camera** controller is set to *Gatan Ccd* not *TIA Ccd*.
- With no sample (i.e. a hole - no substrate) in the field of view, select **Preview** (binning = 2, 0.5 sec) and verify that the CCD has even illumination across the image. If not, run “Acquire Gain Reference” in DM.

Basic EFTEM

- Start and align TEM. Select a high beam current. Note that after a gun change, it may be necessary to re-**ALIGN ZLP** several times until the FEG tip stabilizes.
- Set the sample to a hole.
- Select **EFTEM Tab** | Autofilter | **EFTEM**.
- Select **Align ZLP** button, view beam on CCD camera. If it looks uneven, collect a new Gain Reference in DM.
- Select **Tune GIF**.
- Locate your area of interest.
- In the AutoFilter panel, select **EFTEM**, Filtered. Search mode will show the filtered CCD image.
- For mapping, insert TV, select **Filtered** and **Zero-Loss**, locate the area of interest and go to the CCD.
- In **Preview** mode, adjust the CCD intensity to ~9k – 10k counts (not e^-). This gives you a zero-loss image on the CCD.
- For an elemental map, select **Custom** | **EI. Map**. Select the element of interest and verify that the various regions will work for your sample. Select OK in the map window and when it asks for the exposure time cancel the acquisition.
- Refocus the image (There WILL be a focus change from the zero-loss.). You will now need to calculate the image acquisition time from the intensity in the Preview mode. The Preview frame rate is set to 1 sec. Look at the counts in the image and calculate the time necessary to get 9k – 10k counts.
- Select **EI. Map** and set the Acquisition time to the calculated value, Then **Acquire**.

Note: to change the CCD intensity readout from e^- to counts, unclick the ruler in the DM Target window.

Notes

TUI – Titan 80-300 User Interface – Basic Microscope control software

TIA – Titan 80-300 Imaging and Analysis – STEM and spectrum imaging software. Also known as ESVision.

DM – Digital Micrograph – CCD image collection software which also controls the GIF acquisitions.

GIF Filter control – controls the GIF hardware. Also known as IFC (Image Filter Control)

DM expects GIF Filter control to be already running.

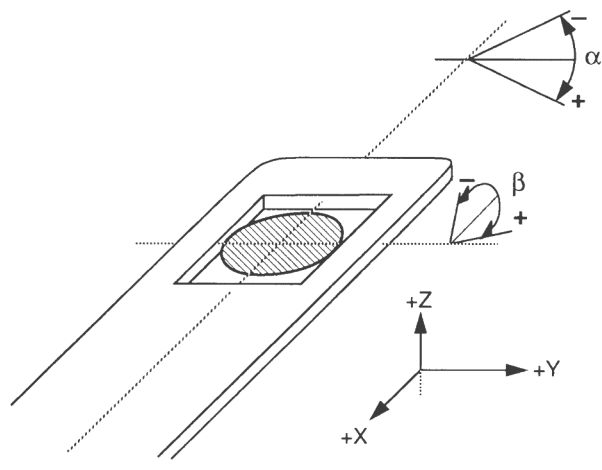
TIA expects DM to be running.

Appendix

Apertures (μm)

	4	3	2	1
C2	150	100	70	100
Obj	8 apertures 100 - 10			
SA	800	200	40	10

Compustage Goniometer Axes



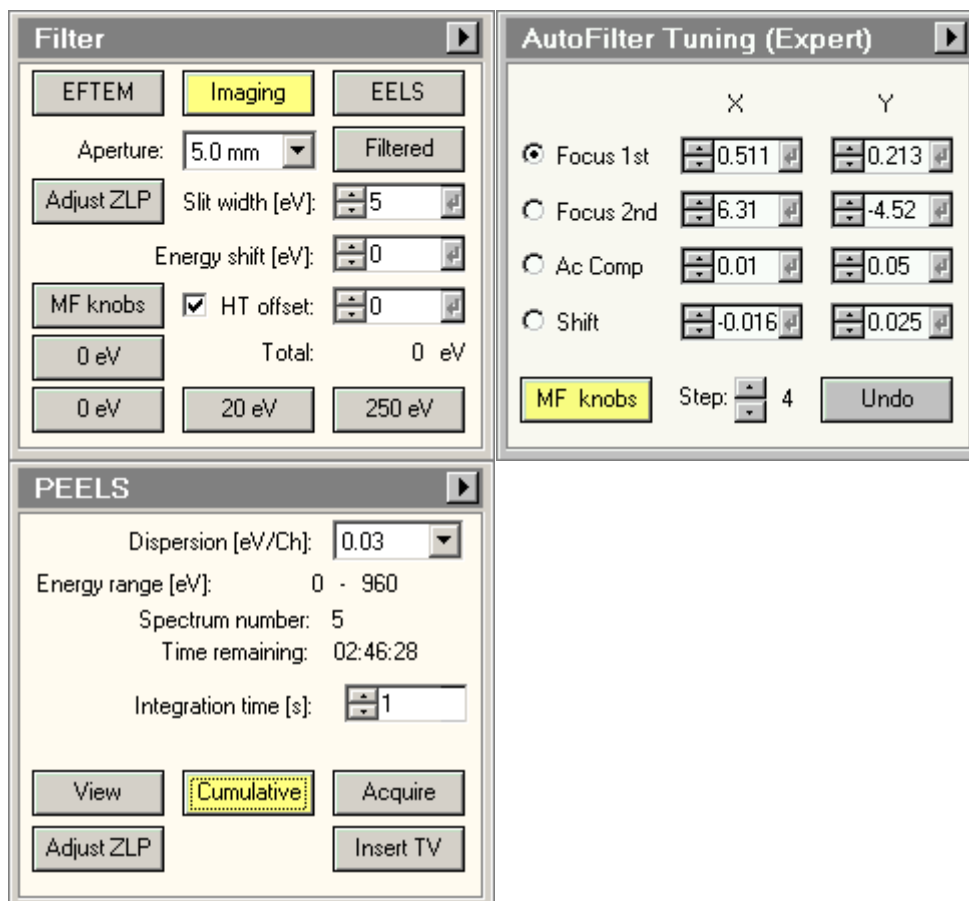
EELS Alignment addendum

General Notes:

- For Higher energy resolution, choose $V_{\text{ext}} = 3400\text{V}$ and tune at 0.1eV per channel, 2mm aperture
- **DM | Filter | Spectroscopy Setup** – allows you to adjust the time etc for different view modes
- **<CTRL>** - drag will expand spectrum around cursor.
- **DM** sums vertical column of pixels on the GIF CCD to get the spectrum.
- You can do manual GIF tuning either through *Gatan FilterControl* or through *TUI*. The *TUI* workpanels will allow you to use the MF knobs.
- 2mm aperture gives $\sim 0.9\text{eV}$ resolution, 0.6mm aperture gives $\sim 0.75\text{eV}$

TUI setup

In *TUI*, set up a tab with the following workpanels.



In **DM | AutoFilter**

If AutoFilter is not visible, open it from **DM | Windows | Floating Windows**

- **Align ZLP**

- **DM | Camera | Prepare Gain Reference** (Set CCD Gain correction)
- **Tune GIF** (repeat till there is no change in the parameters)

You can do manual GIF tuning either through *Gatan FilterControl* or through *TUI*. The *TUI* workpanels will allow you to use the MF knobs.

Set up a tab with “Filter”, “Filter Tuning (Expert)”, and “PEELS”

TUI | Filter | EELS

TUI | PEELS | Insert TV

TUI | Filter | Adjust ZLP (MF-Y) to get ZLP on TV .

TUI | Filter | Aperture | Mask

Do preliminary manual Fx, Fy, Sx, Sy using mask

Best seems to be with Fy at ~11:59 rather than 12:00 (on TV!)

Further GIF Tuning

Spread Beam in TEM, switch to DM

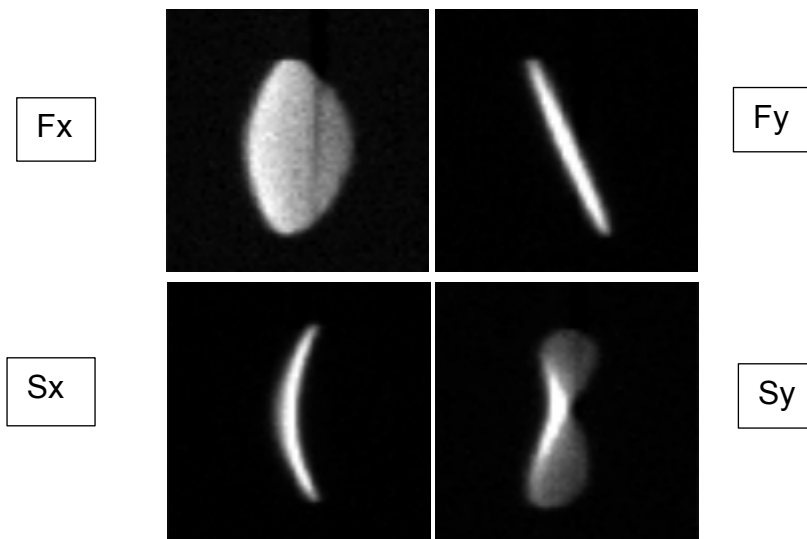
DM | AutoFilter | Turbo View

- Adjust intensity

DM | AutoFilter | View

- Insert Mask,
- Adjust Fx, Fy, Sx, Sy

Switch to CCD View mode



- Spread beam
- Correct Aberration Figures:

- Fine tune Spectrum with image on CCD

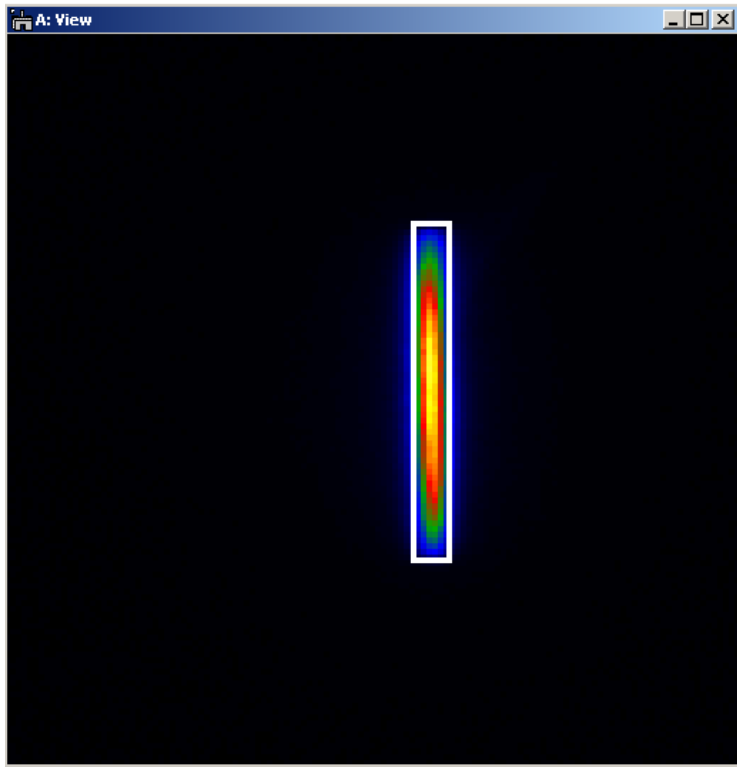
DM | Display | Image Display | Display | Color | Temperature

DM | Display | Image Display | Display | Contrast

Set contrast max level to ~5k

Draw box around ZLP line to serve as a reference

Fine tune Fx, Fy, Sx, Sy using color to assist.



1. Electron Microscope Identification
Manufacturer: FEI Company
Model: Titan 60-300 Image Corrected
Serial Number: D3824
Location: 110 CEMAS
Unit is **Operable**

2. Principal Investigator: R Williams
Telephone: 614/643-3407
E-mail: williams.2156@osu.edu

3. Additional Contact Person: Ashley Swartz
Telephone: 614/643-3467
E-mail: Swartz.202@osu.edu

4. Name of Electron Microscope Operator(s)
Cf. FOM scheduler list

5. General Radiation Safety Policies:
Only personnel trained and approved by the Principal Investigator may operate an electron microscope.
 - a. Radiation Safety must be notified prior to any changes in location, disposal, transfer, or acquisition of any electron microscope. Radiation Safety must also be notified of any plans for modification made to the unit, including built-in shielding and viewing ports.
 - b. Use interlocks, barriers, or administrative controls to ensure no one can gain access to the primary beam or high scatter radiation areas. Stop the primary beam by secured shielding that cannot be readily displaced. Secure unused ports to prevent accidental exposures.
 - c. Secure electron microscopes against unauthorized use by using a unit key control or the room lock. d. Units must be labeled with a readily discernable sign that bears the radiation symbol and the words
“Caution – this equipment produces radiation when energized”.
 - e. A readily visible warning light labeled “X-ray On” or symbols with a similar intent, must be located near the x-ray source and its controls and be illuminated when the x-ray source is energized for equipment installed after 2/10/06.

- f. An operating log should be maintained including the date, operator, beam voltage and current, and total exposure time.
- g. All locations should have a copy of the OSU Radiation Safety Procedures Manual of Radiation- Producing Devices (Non-human Use) which includes the applicable sections of the Ohio Administrative Code, including 3701:1-68-04.

6. ALARA Philosophy

ALARA is an acronym that stands for As Low As Reasonably Achievable. It is the policy of the University to maintain radiation exposure levels not only below applicable legal levels but to also keep the radiation exposure levels as far below the applicable levels as reasonable.

ALARA means making every reasonable effort to maintain radiation exposures as far below dose limits as is practical consistent with the purpose for which the activity is undertaken, taking into account the state of technology, the economics of improvements in relation to the benefits to the public health and safety, and other societal and socioeconomic considerations.

7. Emergency Contact Information

The 24-hour Emergency Response Number is 614-561-7969.

Any individuals have non-emergency questions, concerns, or inquiries pertaining to radiation safety may contact the Radiation Safety Section of Environmental Health and Safety during normal working hours at 292-1284.

8. The Radiation Safety Section of EHS shall be notified immediately of any radiation producing device that is stolen, lost, or missing.

9. Emergency Contact Information

The health physicist on-call can be paged at any time at 614-561-7969.

If any individuals have non-emergency questions, concerns, or inquiries pertaining to radiation safety, contact the Radiation Safety Section of Environmental Health and Safety during normal working hours at 292-1284.

10. Specific Standard Operating Procedures –
See notes above.