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USER GUIDE

Very high energy resolution with X-FEG UltiMono electron microscopy techniques

Alignment information for the XFEG UltiMono.

The benefits of X-FEG UltiMono

Thermo Scientific™ X-FEG™ UltiMono is an electron microscopy source for ultra-high-resolution applications. By using the X-FEG UltiMono package, one can achieve energy resolutions better than when using a standard monochromator, enabling research on phonon-related applications in materials science. X-FEG UltiMono features very-high-energy resolution (<55 meV @ 300 kV, <25 meV @ 60 kV) and enables the following applications:

a) Phonon band study (2D material)

b) Interaction of phonon and boundaries

c) Surface phonon and other applications

General alignment procedure:

1. Preparation for X-FEG UltiMono use
2. X-FEG UltiMono in TEM mode
3. X-FEG UltiMono in microprobe (µP) Scanning Transmission Electron Microscope (STEM)

> **CRITICAL POINT:** Do not leave the microscope in the UltiMono mode for more than 24 hrs. It is possible to reload a previously stored UltiMono alignment file and UltiMono FEG register, to bring the microscope back to an aligned UltiMono state.

Alignment procedure:

1. Preparation for X-FEG UltiMono:

Prerequisites:

Ensure the system is stable at the desired HT and the instrument has the UltiMono software or OptiMono+ software with the UltiMono option installed.

Ensure the Gatan Imaging Filter (GIF) is well-aligned and tuned before starting the UltiMono alignment procedure. The specimen that is loaded should have sufficient vacuum area, i.e., >tens of microns.

Check that an alignment file at the relevant HT is present. Preferably, this file should be a Transmission Electron Microscope (TEM) Field Emissions Gun (FEG) register with default monochromator potential (3kV) and 2-condenser uP-STEM FEG register.

- Load an up-to-date alignment file and a TEM FEG register with default potential of 3kV.
- Go to a vacuum area.
- Press **Presets** in OptiMono UI. This brings the microscope to filtered 2-condenser TEM mode at spot size 1 and lowest SA magnification (**Figure 1**). Regarding the use of OptiMono, please refer to the dedicated user manuals for OptiMono.

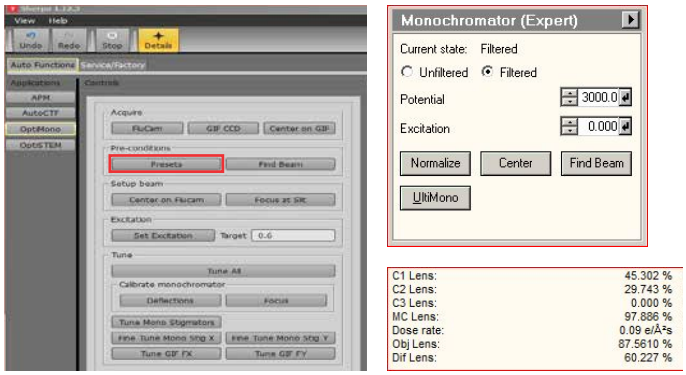

Figure 1. Screenshots showing the activation of filtered 2-condenser TEM mode.

- Lower the monochromator potential to 800 V (**Figure 2**), using one of the following two methods:

1. Enter 800 V into the field, and then use the **Find Beam** routine by selecting the button in the monochromator panel or in OptiMono.
2. Lower the potential step-by-step (e.g., 100 V steps), while keeping the beam centered using Monochromator Shift.

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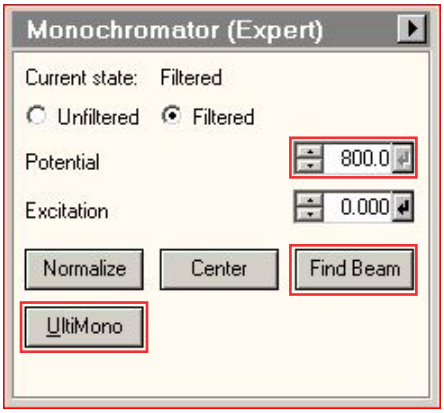

Figure 2. Screenshots showing the activation of UltiMono optics and relevant functions in monochromator control panel.

- Perform direct alignments while paying particular attention to the rotation center.
- Check the slit and hole centering of the C1 apertures intended to use and recenter if necessary.
- Roughly center and stigmate the monochromator using shift and stigmator in the Monochromator Tune panel (**Figure 3**).
- Focus and normalize the monochromator and re-center. Press both **Store** buttons in the Monochromator Tune panel (**Figure 3**).
- Save this TEM FEG register for reference.
- Click the **UltiMono** button in the monochromator control panel (**Figure 2**) or in the OptiMono UI (**Figure1**) to get the optical set-up for UltiMono. The change in the optical settings will take a few minutes. It takes roughly 30 minutes to achieve the thermal stability necessary for good performance. During the stabilization time the gun alignment can be performed (next bullet point).
- Perform the full gun alignment in the **Alignment** panel (**Figure 3**). Achieving the highest energy resolution requires a well-aligned gun (e.g., minimizing the coma in the gun that originates from a misaligned gun tilt). Due to the changes in optics, it is necessary to save this as a new alignment file. Careful attention must be paid to find the edge of the 1 mm hole in four directions with the same symmetrical features as possible. In this way, there is the least chromatic anerration, with the highest energy resolution. It is strongly advised that users not quit until the full gun alignment is completed.

> **NOTE:** Often, performing the gun alignment provides enough stabilization time for the microscope. It is recommended that this should be done at a given high tension the first time UltiMono is used and then regularly repeated. The interval depends on the stability of the surroundings, but a reasonable timeframe is to repeat the full gun alignment every few months.

While the full gun alignment has been completed and saved, it is strongly advised to reset the monochromator table (**Figure 3**). This allows for the **Find Beam** routine to work properly. More importantly, it allows the **Find Beam** routine (**Figure 3**) to work efficiently while exciting the monochromator. Otherwise, “find beam” routine may fail or take very long time due to the wrong interpolation of excitation-dependent monochromator shift.

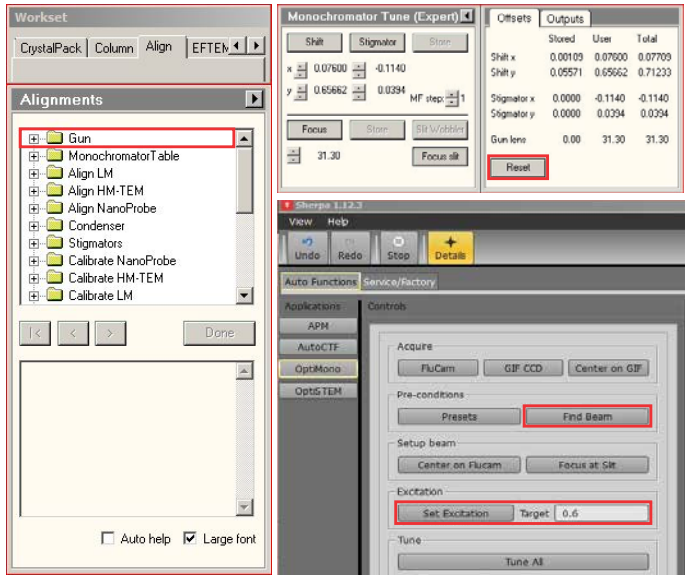

Figure 3. Screenshots showing the full gun alignment module and subsequential “Reset” procedure to get “Find Beam” routine to work while exciting the monochromator.

2. X-FEG UltiMono in TEM mode:

2.1 Basic alignment

- Re-check all direct alignments and in particular paying attention to the rotation center as well as C1 slit and hole centering.
- Normalize monochromator and, if necessary, find the beam’s back; Save an intermediate FEG register as a reference.
- Roughly center the beam to the GIF entrance aperture (beam shift) marked in the Thermo Scientific SmartCam™ camera viewer, fine-tune the beam shift to maximize the EELS (electron energy loss spectroscopy) zero-loss peak (ZLP) intensity, and set up a low dispersion (e.g., 1 eV/ch) in EELS mode in the **Digital Micrograph (DM)**.
- Acquire a live electron energy loss (EEL) spectrum with the ZLP (**Figure 5**) and ensure a proper EELS set-up in the **DM**. Click **View** in the **EELS** toolbar in the **DM**, commencing from the lowest exposure time (1E-6 second). Lift the viewing screen and increase the exposure time to get a proper view of the ZLP. By doing this, the risk of damage to the GIF camera is reduced.
- Center ZLP up to a high dispersion (e.g., 0.005 eV/ch) in EELS mode, with a suitable exposure time. Select the smallest entrance aperture to avoid high order aberrations and center ZLP on camera center by clicking **Align ZLP** (**Figure 4**).
- Coarsely adjust FX, FY, SX, SY, if necessary. Click **Focus X**, **Focus Y**, **SX**, **SY** under the drop menu **Adjust** tab of the **Filter Control** panel to coarsely adjust GIF focus and stigmation in **DM** (Figure 4). Note that ultimate fine adjustments can be done by clicking **Focus**, accessing the **Manual Tuning** window on the left side of the **Tune GIF** panel, and choosing a proper strength of FX, FY, SX, SY per click (**Figure 4**).
- Press **Focus at slit** in the OptiMono (**Figure 3**) and excite the monochromator within a range of 1.6 – 1.8, either using OptiMono (preferred) or the monochromator control panel. Again, it is strongly recommended to reset the Monochromator Table in the Monochromator panel if the total Monochromator shift is too large, prior to exciting the Monochromator.

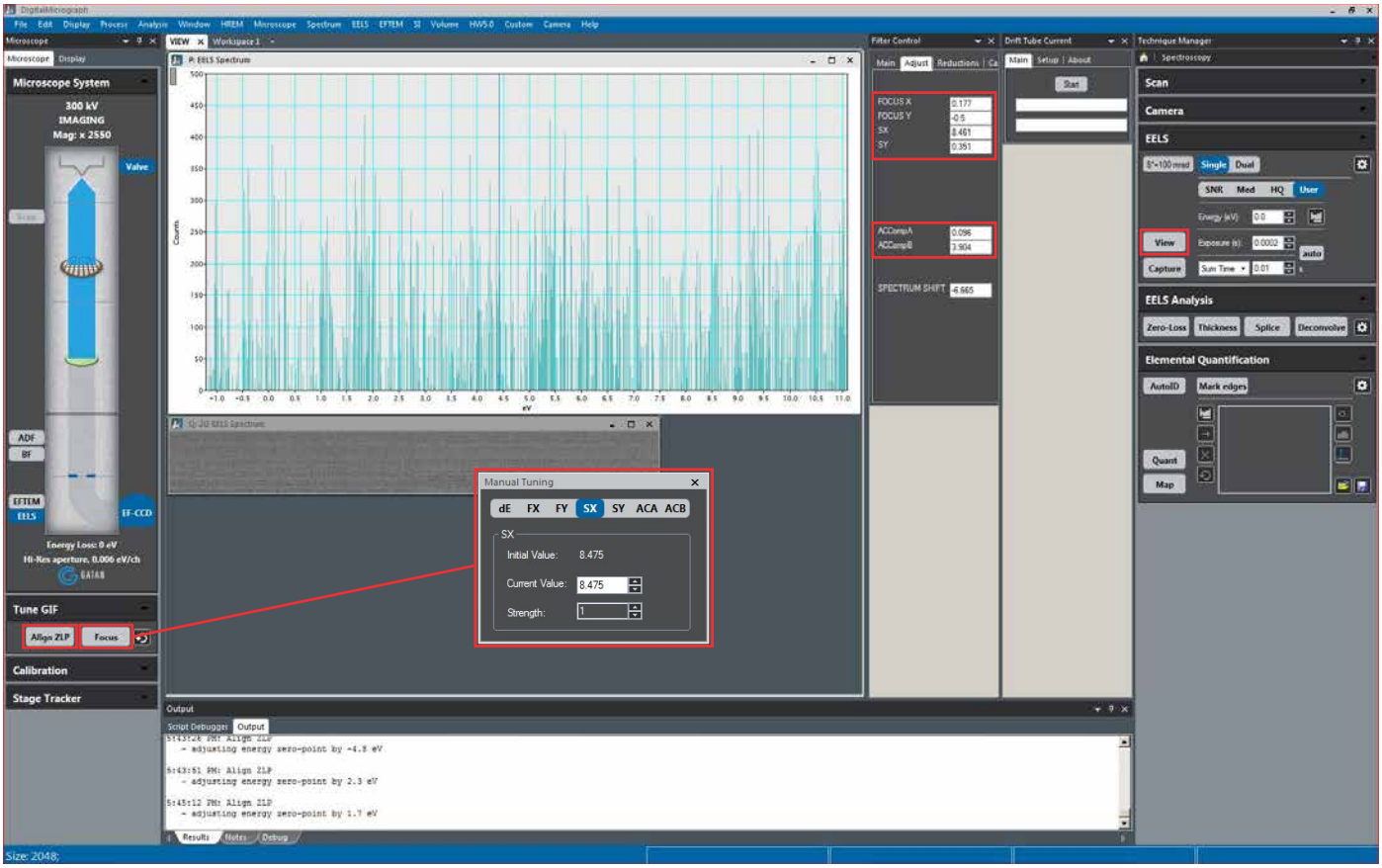

Figure 4. Screenshots showing the functionalities in Digital micrograph to be used for X-FEG UltiMono.

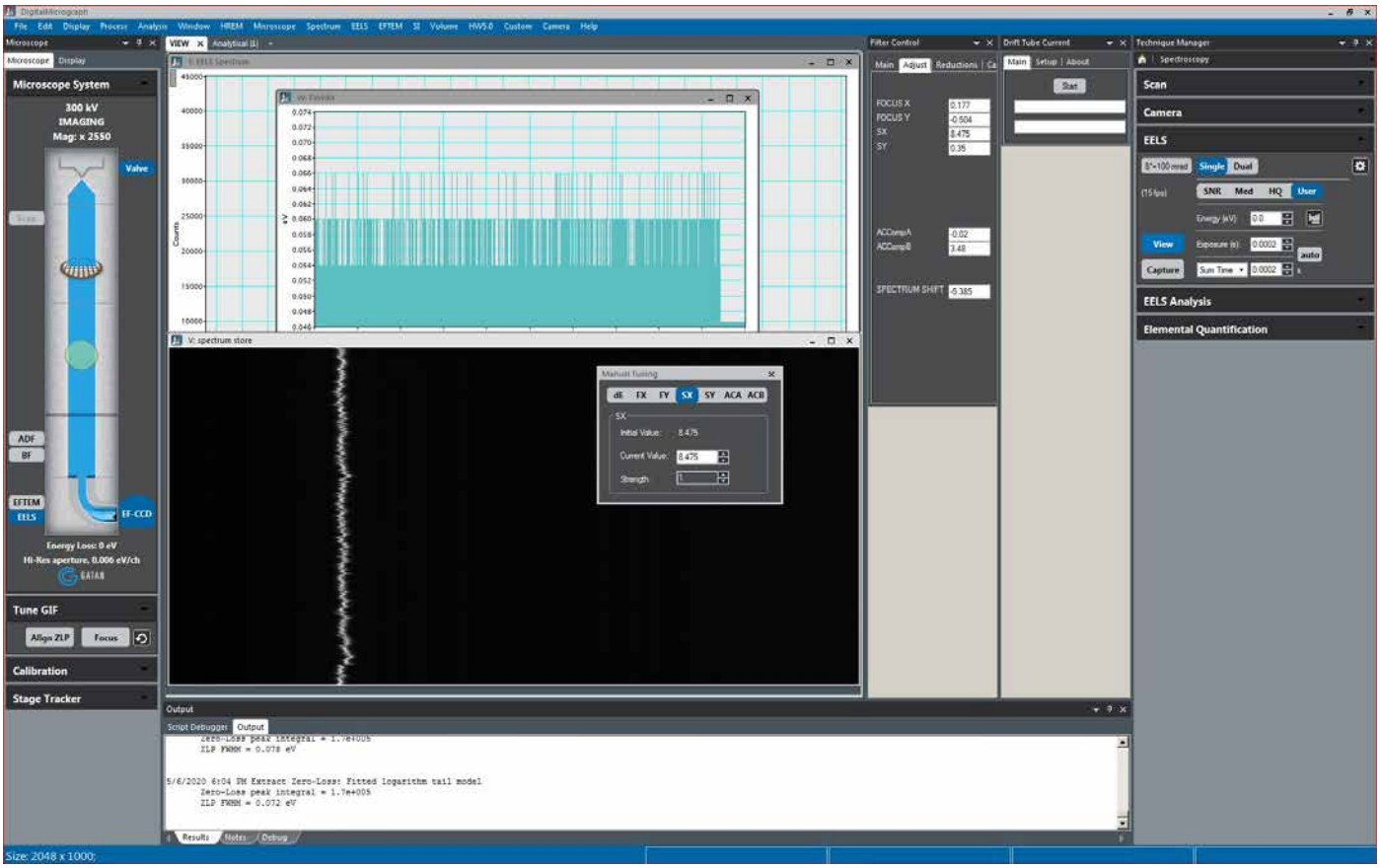

Figure 5. Screenshots showing the functionalities in Digital micrograph to be used for X-FEG UltiMono.

- Press **Tune All** in OptiMono (**Figure 3**) to optimize the stigmation for both monochromator and GIF in an efficient and reproducible way. Regarding the usage of OptiMono, please refer to the application notes for OptiMono, i.e., either press **Tune All** or follow the tuning step by step. Note that the monochromator and GIF stigmators can be further tuned to achieve the highest energy resolution by following this order: **Fine Tune Mono Stig Y** > **Fine Tune Mono Stig X** > **Tune GIF FY** > **Tune GIF FX**.
- Correct 50/60 Hz frequency (AC compensation) to minimize the effects of stray magnetic AC fields, if necessary. Please refer to *Appendix A* for detailed instructions on how to correct 50/60 Hz frequency disturbances.
- If necessary, manually fine-tune FX, FY, SX, and SY in **DM** to optimize the energy resolution. For Continuum and Continuum S spectrometers, press "**Alt+M**" in **DM** to activate an instantly live graphical representation to conveniently tune further to achieve the highest energy resolution (**Figure 5**). For an older instrument, such as the GIF QuantumTM energy filter from Gatan Inc., this functionality is not available, but manual tuning is still achievable to obtain the optimized energy resolution.
- Acquire the EEL spectrum. Click "**Capture**" in the EELS toolbar in "**Digital Micrograph**", using the SNR setting for the camera and a proper exposure time (**Figure 6**). Ensure to have the suitable capture setting for Sum Time or Frame Integration. Alternatively, acquire a series of EEL spectra and align the spectra to remove any movement of the zero-loss peak.
- Measure the energy resolution using the ZLP. Click "**Extract Zero-loss**" in "**Zero-Loss**" that is in the drop menu of the "**EELS**" in **DM** (**Figure 6**) and it will measure the energy resolution. Note that the measurement accuracy depends on the fitting method used. If necessary, adjust the settings to get a more accurate fit.
- Normalize monochromator, recenter the monochromated beam and save the FEG register for UltiMono-TEM. This FEG register can be used together with the UltiMono alignment file to recall the proper alignments in future use.

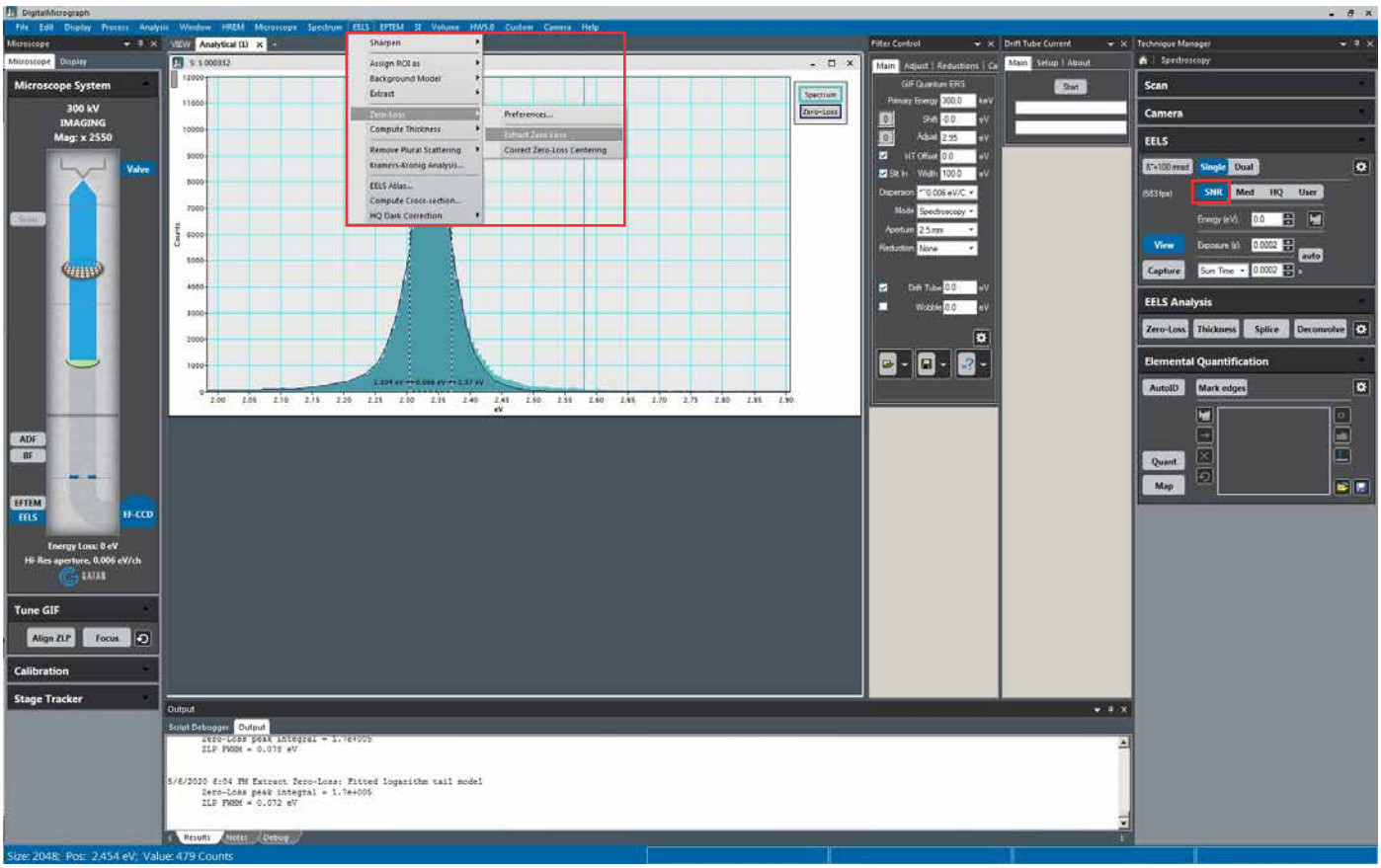

Figure 6. Screenshots showing the measurements of energy resolution.

2.2 Finalize the beam set-up amd measurement of energy resolution

- Change the beam to spot size number 7 – 9 (maximize beam current with monochromator shift) and align beam shift. This minimizes the effect of geometrical aberrations in the beam that are favorable for high spatial resolution and slightly improved energy resolution.
- Insert and center a C1 0.5 µm hole aperture by maximizing the beam current.
- Insert and center the 50 µm C2 aperture, maximizing the beam current. Write down the present highest beam current as a reference to detect any drift.
- Go to a medium magnification (e.g., 50 K) and make the beam as small as possible with intensity using the C2 lens. For a more detailed explanation of why the C2 lens intensity knob can be used for high spot size number 7 – 9, please refer to the Monochromator User Manual.

- Carefully center beam on GIF entrance aperture using beam shift. Click **View** in the **EELS** toolbar in the **DM** starting from the lowest exposure time. Lift the SmartCAM viewing screen and increase the exposure time to achieve a sufficient signal. For Continuum and Continuum S spectrometers, press **Alt+M** in **Digital Micrograph** to activate a live graphical representation of energy resolution measurements, and optimise FX, FY, SX, and SY. Otherwise, use the EEL spectrum for the optimisation of FX, FY, SX, and SY.
- Acquire an EEL spectrum and measure the energy resolution. Click **Capture** in the EELS toolbar in DM under SNR setting for camera and a proper exposure per pixel. Then, click **Extract Zero-loss** in **Zero-Loss** that is in the drop menu of the **EELS** in DM (**Figure 6**). Note that the measured energy resolution also depends on both the microscope and the GIF performance.
- For further optimization, if necessary, change Mono Stig X and recheck Mono Stig Y with Optimono at SS1. Go back to the high SS used and repeat procedure 2.2.

3. X-FEG UltiMono in µP-STEM mode:

3.1 Basic alignment

- Activate **STEM** in the STEM control panel and ensure the activation of Microprobe mode in the beam settings control panel (**Figure 7**). Alternatively, load a pre-aligned microprobe STEM FEG register with an unchecked gun in the FEG register control panel.
- Choose either two-condenser mode (**C3 off** checked) in beam settings control panel (**Figure 7**) or three-condenser mode. Two-condenser mode is strongly recommended, as it is easier to tune the alignments. Three-condenser mode can be used as an exception when a specific or variable convergence angle is needed for the experiment.
- Select **Descan** in STEM control panel (Fig. 8).
- Select spot size 7 – 9 or the spot size which was optimized in the previous section.
- Out of diffraction, optimize the monochromator shift in the monochromator control panel by maximizing the probe current with largest C1 and C2 apertures.
- Perform all direct alignments

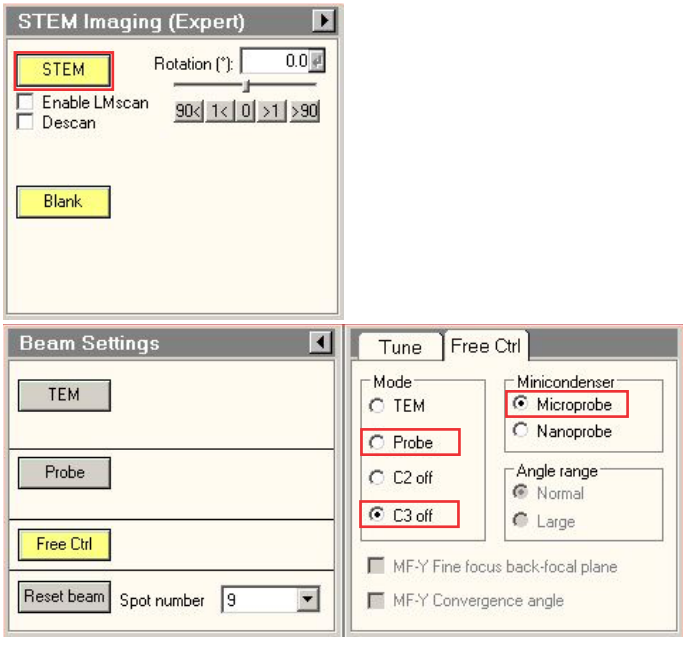

Figure 7. Screenshots showing the preferred STEM mode within UltiMono.

3.2 Descan alignment

- Acquire a STEM image in TIA, right click on the image, and select **Info…**, scroll down to see the **STEM rotation correction** (**Figure 9**), and write down the angle.
- Enter the opposite value as the STEM rotation in the STEM control panel (**Figure 8**).
- In diffraction, optimize Descan pivot points as far as possible by minimizing the movements of the diffraction disk at lowest STEM magnification for **Search** to >15 µsec.
- Exit diffraction, increase dwell time for **Search** to >15 µsec, and activate **Scope** in STEM control panel (**Figure 8**).
- Optimize Descan shift line and frame (**Figure 8**):
  - Line (fast direction), to minimize spot movement
  - Frame (slow direction), to rotate scan rotation 90° in STEM imaging control panel and minimize spot movement
  - Iterate between the two directions until they converge
- Switch to diffraction and double-check the Descan pivot point. If necessary, iterate the process of Descan pivot points and Descan shift line and frame until the movements are minimized.
- Ensure C1 and C2 apertures are centered.

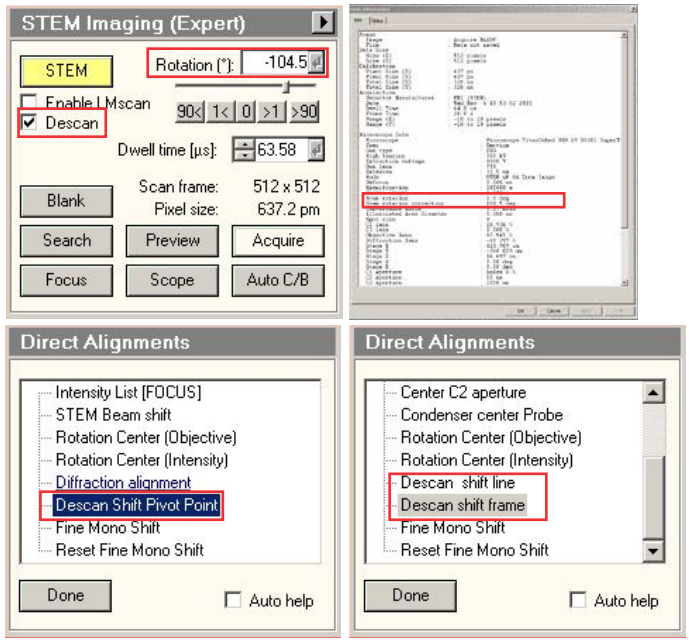

Figure 8. Screenshots showing how to acquire STEM rotation correction angle and Descan alignments.

- Ensure proper direct alignments have been done. Pay particular attention to the **Intensity List [Focus]** and **Beam Tilt/ Rotation Center**.
- Ideally, re-check the energy resolution in 2-condenser probe mode. This should be as good as when it was measured in TEM imaging during the set-up.
- In diffraction mode, center the central bright field disk on GIF entrance aperture (diffraction alignment).
- Choose a proper camera length in STEM control panel for energy resolution and other measurements. The higher the camera length, the smaller the influence of aberrations from the projection system and filter, but the lower the beam current that gets through the GIF entrance aperture. One may want to optimize the camera length to minimize aberrations

of the projection system while ensuring that there is sufficient signal for measurements.

- Carefully center the beam on GIF entrance aperture using diffraction shift. Press **Alt+M** in **Digital Micrograph** to activate alive graphical representation of energy resolution measurements, and, if necessary, optimize FX, FY, SX, and SY. Click **capture** in the EELS toolbar under SNR setting for camera and a proper exposure. Then, click **Extract Zero-loss** in **Zero-Loss,** which is in the drop menu of the **EELS,** for energy resolution measurements (Figure 6). This needs to be done carefully to reach the best possible performance (Figure 9).
- Normalize monochromator, find beam via monochromator shift, and save an UltiMono STEM FEG register.
- Move to the area of interest and perform the spectral imaging experiment for your own research/applications (*Appendix B*).

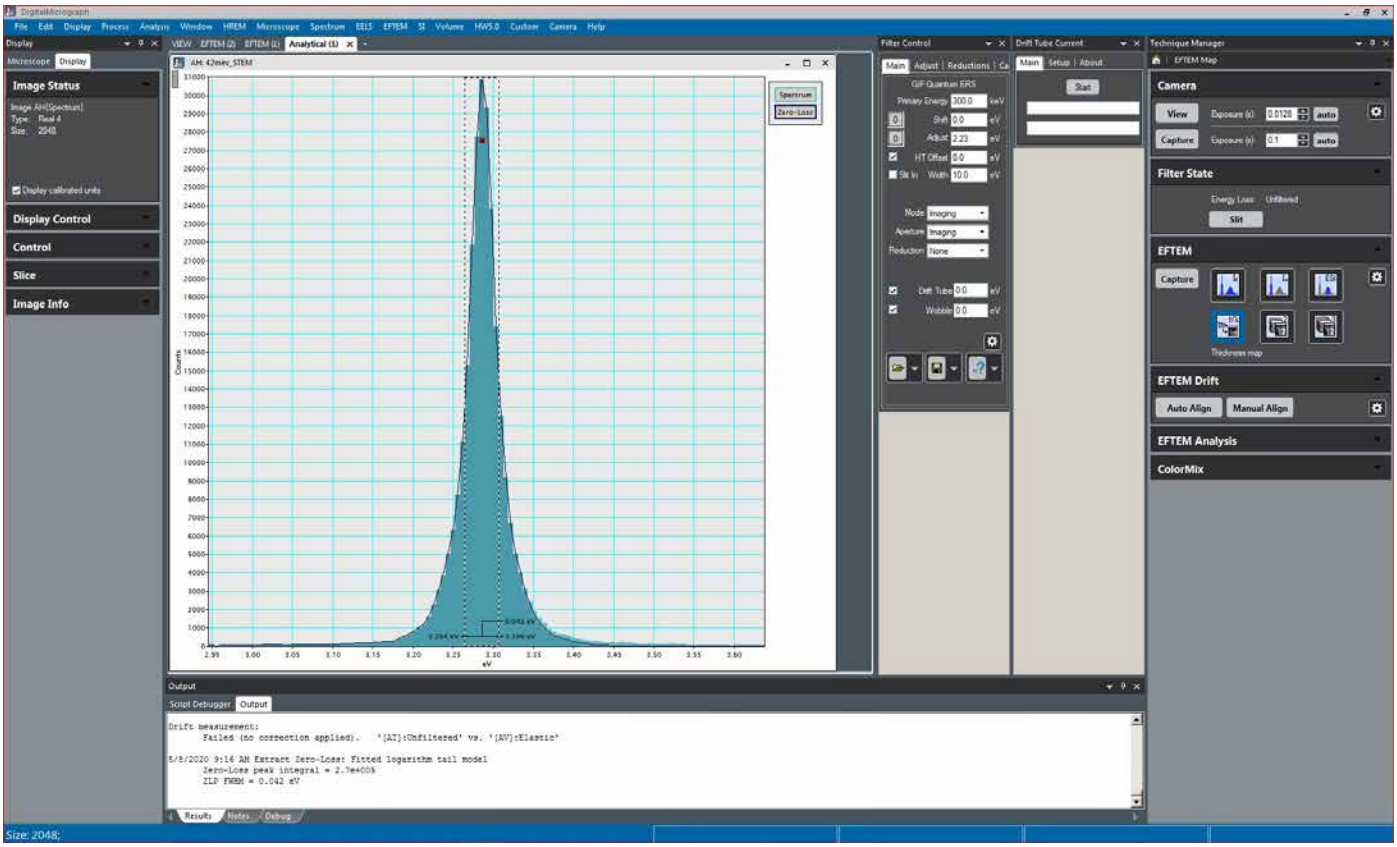

Figure 9. An example showing an energy resolution of 42 meV achieved at 300 kV with an excitation of 1.6.

Appendix A

Ensure that the aberrations of the GIF are well-tuned in the EFTEM mode, prior to AC compensation tuning.

Quantum filters

A 'streak-image' is made to visualize and minimize the effects of stray magnetic AC fields. To do so, this requires a set-up with no shutter, double-focused dispersion, and zero exposure time. The whole procedure can follow these steps:

1. Ensure **DM** is in **EFTEM mode** instead of **EELS mode**.
2. Lower the viewing screen.
3. Disable the fast shutter in the fast shutter control page within **DM**, and if not found, go to floating window and select fast shutter page that is to be displayed.
4. Select the 0.00 eV/pixel setting, selecting the lower 0.01eV/pixel setting if two 0.01 eV/pixel settings are displayed.
5. Set the exposure time to 0 seconds, choosing a relatively high magnification (e.g., 115 kx) or **STEM mode**. If using STEM mode, aligning AC compensation should have been performed before UltiMono. Ensure the beam is centered.
6. Start the CCD image acquisition, and wait until the first image is displayed by **DM**.
7. Raise the viewing screen. The image on the CCD will be a 'streak image,' that is a recording of energy versus time, in which 50 Hz (or 60 Hz) stray fields and other irregularities can be easily seen.
8. Adjust AC Comp A/B of filter control to minimize the 50 Hz or 60 Hz effects.
9. Lower the viewing screen and enable the fast shutter in the fast shutter control page within **DM**.
10. Perform the next step experiment.

Continuum filters

The AC compensation is performed in **spectroscopy (EELS) mode**. This requires a set-up of well-tuned GIF with a resulting well-tuned spectrum. First, selecting the smallest or second smallest dispersion is advised, so that the ZLP can be well resolved. Second, using a proper current is suggested, so an exposure time between 1 ms to 10 ms can be utilized to reflect the 50/60 Hz frequency. Then, follow the steps below for AC compensation adjustments if needed:

1. Click the spectrum and press **Alt+M** in **DM** to activate an instantly live graphical representation.
2. Click the ZLP position measurement after the ZLP and FWHM measurements appear in a pop up window.
3. Make a live FFT of the ZLP position measurement (via Process > FFT > Live) and tune AC Comp until any "non-zero" frequency peaks diminish (Figure S1).

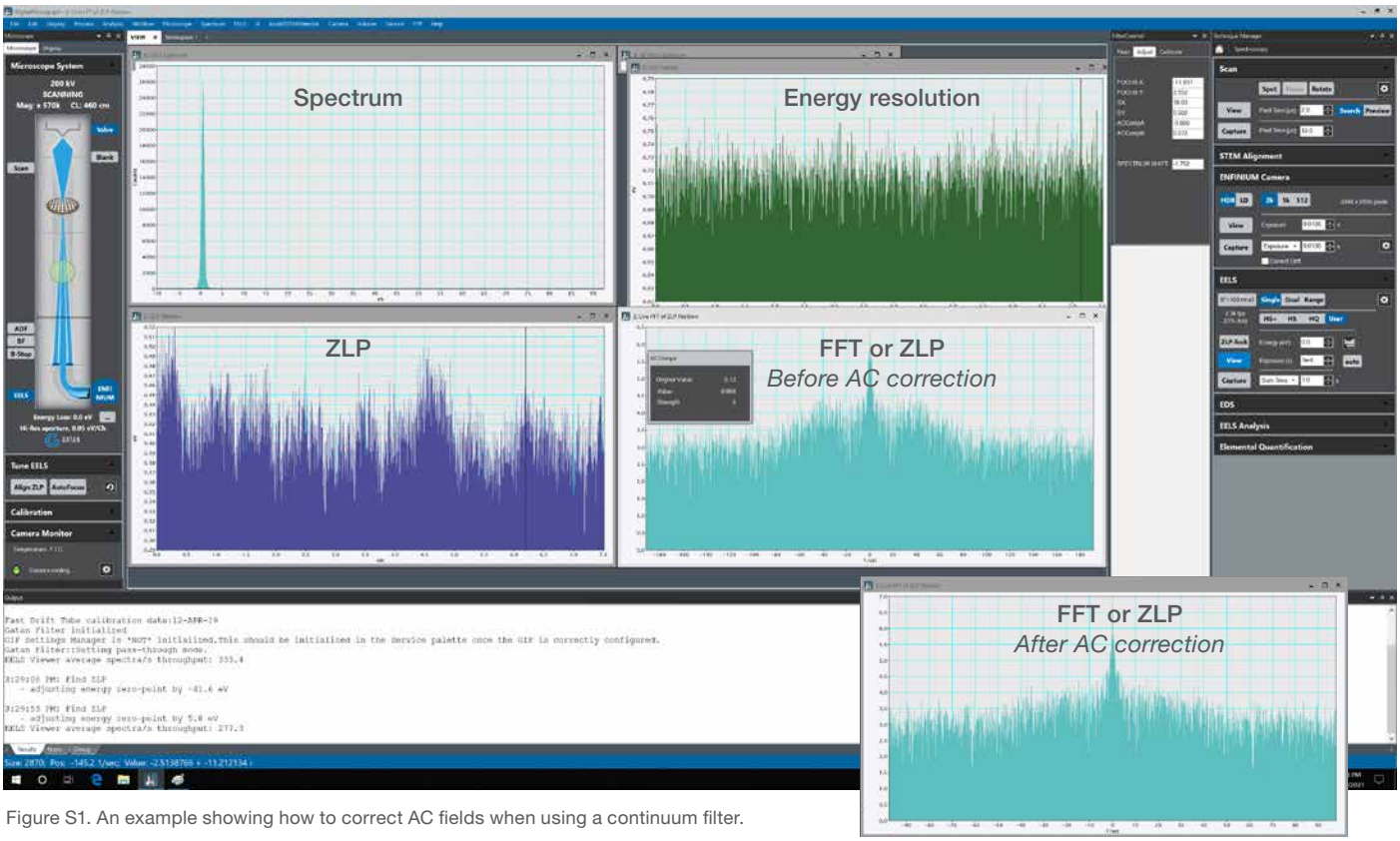

Figure S1. An example showing how to correct AC fields when using a continuum filter.

Appendix B

When performing vibrational spectroscopy related applications on polycrystalline/monocrystalline samples or regions where diffracted discs are close to central disc (microprobe STEM mode), it is of great importance to choose the proper camera length and use a sufficiently small, objective aperture to select only the transmitted discs and to screen out the diffracted discs. Otherwise, EEL spectra may contain misleading peaks or tails as a result of diffracted discs which should not be present in the spectra. Depending on experimental circumstances, this peak may vary from a few hundred meV to a few eV.

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Notes

Find out more at **thermofisher.com/spectra300**

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