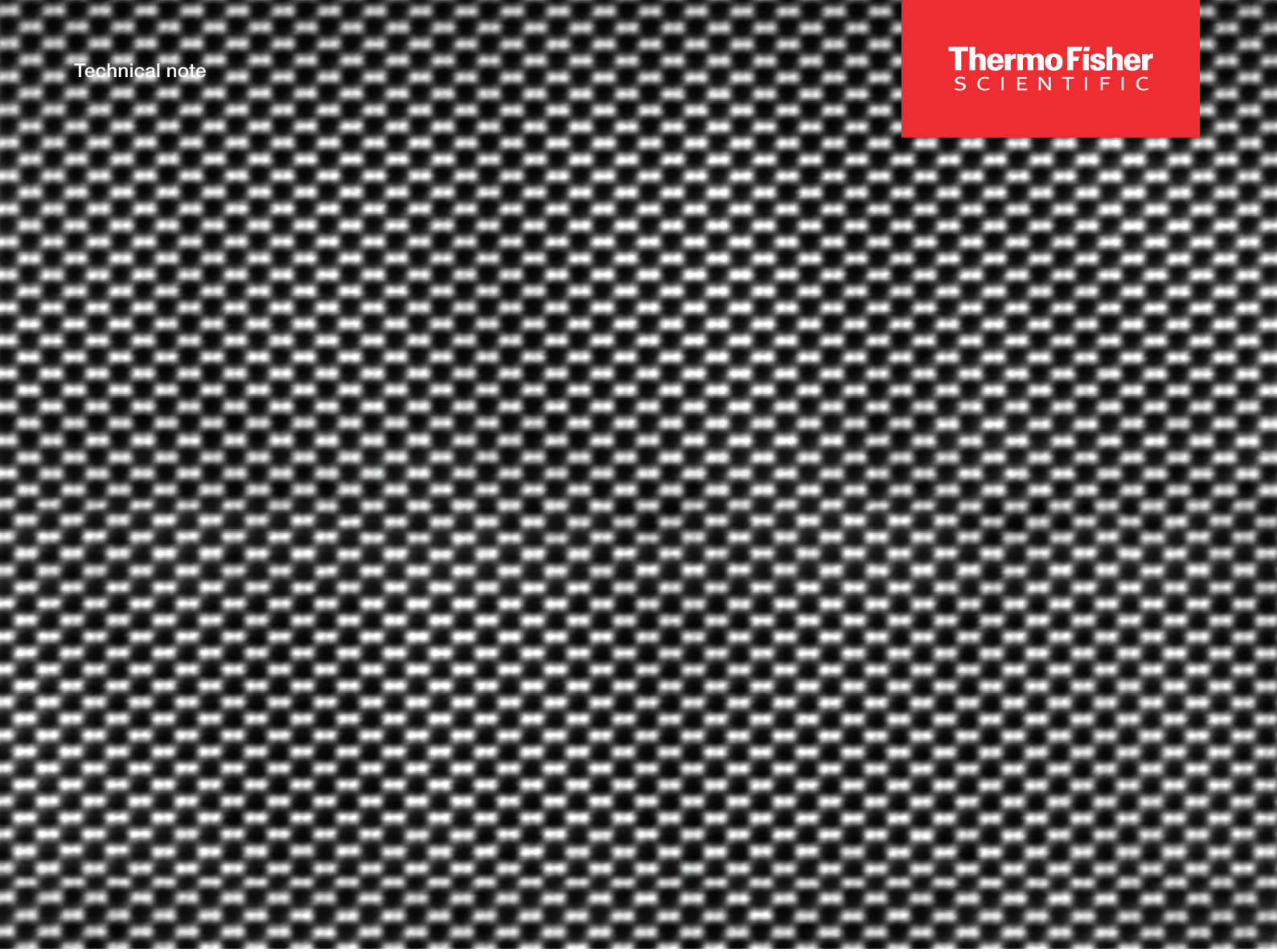
Technical note

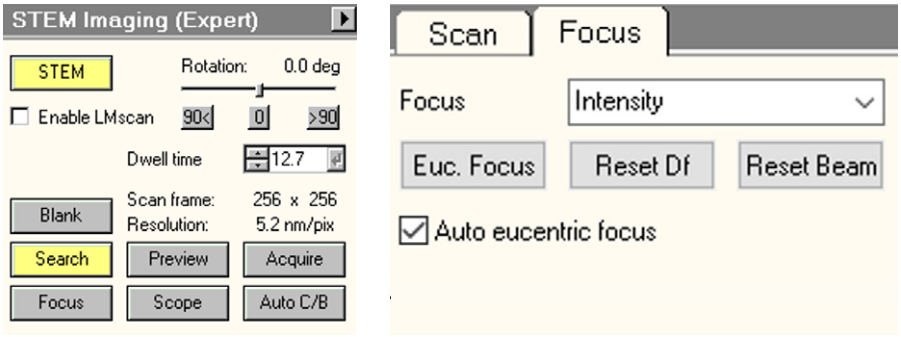


HRSTEM Imaging with Uncorrected Thermo Scientific TEMs

High-resolution scanning transmission electron microscopy (HRSTEM) is a highly valuable technique across a wide range of samples and industries. This technical note provides step-by-step instructions for aligning uncorrected STEM systems for such high-resolution studies, including Thermo Scientific™ Talos™ and Tecnai™ TEMs, as well as uncorrected Thermo Scientific™ Titan™, Spectra™ and Iliad™ TEMs with field emission guns (FEGs). This procedure was created using a Thermo Scientific™ Talos™ F200X TEM with an X-TWIN pole piece.

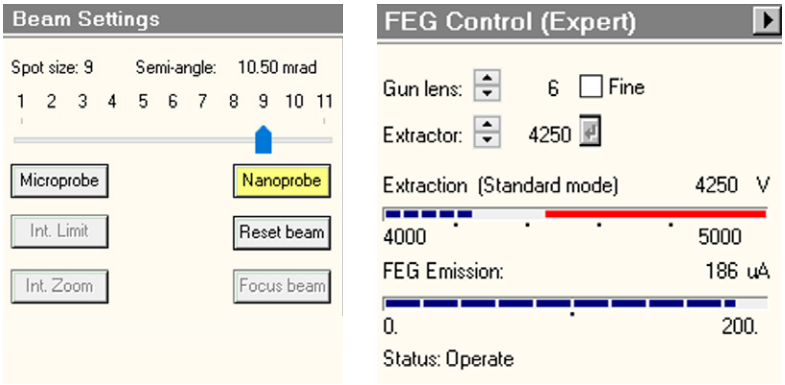
Basic microscope setup

1. Load the most recent column alignment file, with the appropriate high-tension settings from the “Alignments” OCX, if needed.
2. Load a STEM (nanoprobe) FEG register file, if one is available. If not, click “STEM” in the “STEM Imaging” OCX. In the STEM flap-out, ensure that “Intensity” is selected for “Focus” instead of “Intensity & Objective” or “Objective.”



Left) STEM mode activation. Right) STEM focus selected as “Intensity.”

3. Ensure that “Nanoprobe” is selected in the “Beam Settings” OCX.
4. Select an appropriate C2 aperture, such as 70 µm (which corresponds to a convergence semi-angle of 10.5 mrad at 200 kV).
5. Select a suitable combination of gun lens (e.g., 4–8) and spot size (e.g., 9–11). Generally, for high-resolution STEM imaging with an X-FEG, a probe current of ~30 pA is required, whereas with an S-FEG, it should be less than 10 pA.

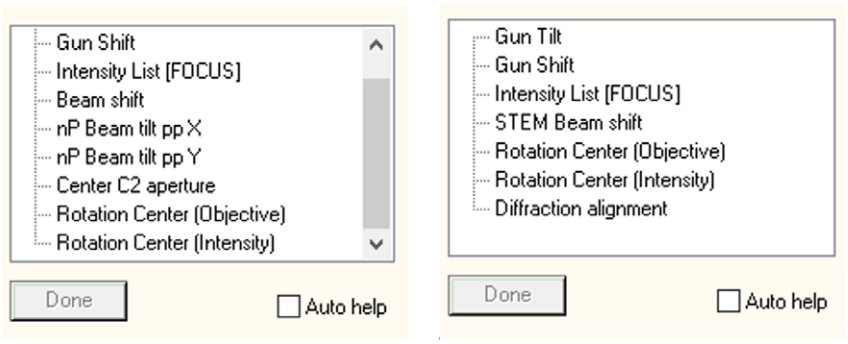


Left) Beam settings for probe selection. Right) FEG settings for gun lens and extraction voltage.

6. Acquire STEM images using Thermo Scientific™ Velox™ Software or TIA Software. Press the “Eucentric focus” button on the right-hand control panel and adjust the sample to the eucentric height using the z +/- buttons. Find a vacuum or amorphous region, go to high magnification above 1Mx, stop scanning, unblank the beam if necessary, and verify that the probe position marker is centered in the image. This ensures there is no additional beam shift away from the optical axis.

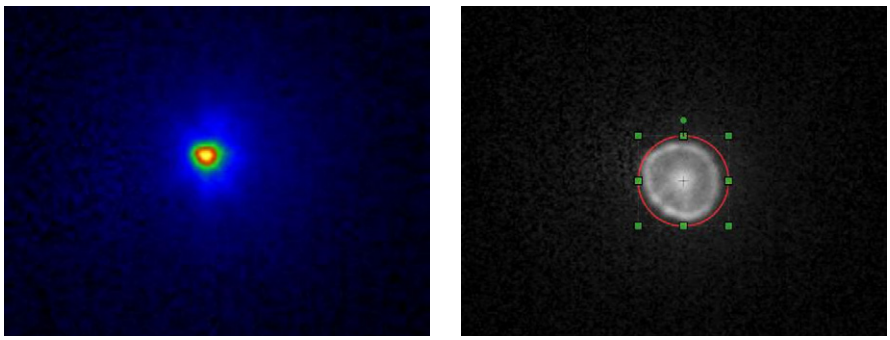
Alignments in imaging mode

7. Exit diffraction mode by clicking the “Diffraction” button on the right-hand control panel, switching to probe mode.
8. At low SA magnification, find the probe and center it using the “Beam shift” option in the “Direct Alignments” OCX. Precisely align the C2 aperture by checking the beam’s concentricity as it opens and closes while wobbling the C2 lens (via the focus knob). C2 wobbling can also be performed by clicking “Center C2 aperture” in the “Direct Alignments” OCX. The beam’s concentricity should now be optimized for accurate C2 aperture centering.



Left) Alignment elements in probe mode. Right) Alignment elements in diffraction mode.

9. In the “Direct Alignments” OCX, click “Intensity List [FOCUS]” and focus the probe using the focus knob, then center it using the “Beam shift” option. Increase magnification to maximum using the magnification knob on the right-hand control panel.
10. Click “nP Beam tilt pp x/y” to correct the pivot point. Use the “Multifunction x/y” knobs to overlap the brightest points of the two probes. If needed, reduce the magnification to view both spots clearly.
11. Click “Rotation Center (Intensity)” to align the beam tilt and then make the beam opening concentric by adjusting the “Multifunction x/y” knobs. Alternatively, stop the wobbling by turning the lower wheel of the focus knob counterclockwise, then defocus the probe to reveal the “caustic spot” (as shown in the figure below). Adjust the probe to center the “caustic spot” as accurately as possible.



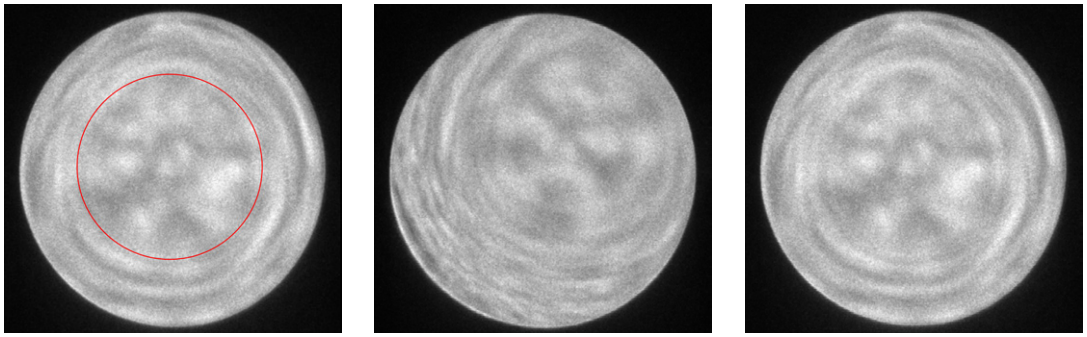
Left) Temperature color-coded image of the probe shape at eucentric focus, taken at the highest SA magnification. Right) A well-aligned beam tilt obtained using “Rotation Center (Intensity).”

12. Recheck the pivot point. Focus the probe using “Intensity List [FOCUS]” and center it using the “Beam shift” control.
13. Assign a hand panel key (e.g., L2) to “Normalize all” and press it to normalize the lenses.
14. Repeat steps 9-13 multiple times until minimal adjustments are needed after normalizing all lenses.
15. Focus the probe using “Intensity List [FOCUS]” and center it with the “Beam shift” control.
16. Switch to diffraction mode by pressing the “Diffraction” button on the right control panel.
17. In the “Direct Alignments” OCX, click the “Diffraction alignment” option and center the diffraction pattern in the selected detector.
18. Save an FEG register file.

Alignments in diffraction mode and data collection

19. Start STEM imaging. Adjust the stage height to achieve a sharp image in focus, then center a fresh amorphous area or create an amorphous spot using the electron probe.

20. Pause the scan and ensure the probe position marker is centered. (Optionally, mark the current C2 aperture position on the FluCam by drawing a circle, then insert a larger C2 aperture, such as 150 µm.) Use the focus knob to adjust the Ronchigram (see figure below). Adjust the condenser stigmators to correct the condenser stigmatism and make the aberration-free area (which should resemble a shimmering pool) in the Ronchigram as round as possible. Mark the aberration-free area, insert the desired C2 aperture, and correct any aperture misalignment as needed.



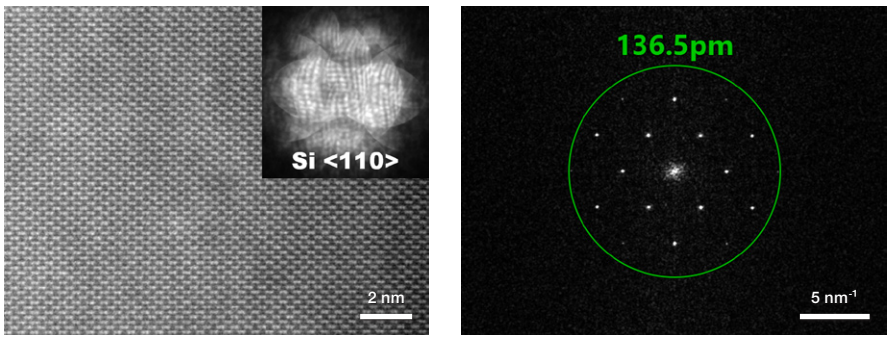
Three Ronchigrams collected with the 150 µm C2 aperture. Left) Stigmated. Center) Aperture misaligned. Right) Optimized; the red circle denotes the desired C2 aperture.

21. Start scanning and adjust the gain and offset of the STEM detectors to maximize the dynamic range. Save or update an FEG register file.

22. Restore or move the stage to the position of your thin crystalline sample. Fine-tune the tilts for the zone axis and adjust the stage height as needed.

23. Increase the magnification to approximately 2.5Mx. Fine-tune the focus and adjust the condenser stigmatism. Optionally, use “reduced area” in Velox or TIA Software for finer adjustments.

24. Acquire a STEM image to verify the resolution using fast Fourier transform (FFT). Alternatively, capture 10–20 frames and then generate a drift-corrected frame-integrated (DCFI) image, which is available in Velox Software under Processing à DCFI ➔ Optimized for periodic images.



Left) STEM-HAADF imaging, used to resolve the Si <004> dumbbell along the <110> zone axis; the inset shows the diffraction pattern of Si <110>. Right) The FFT clearly shows the 136.5 pm reflection of a Si dumbbell.

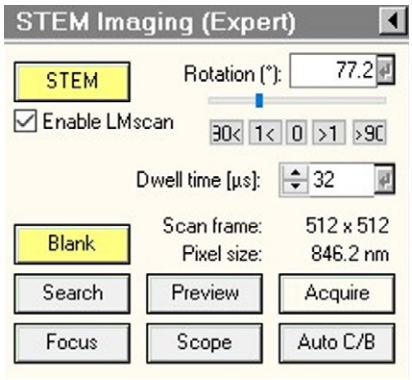
25. Switch to low magnification and save or update an FEG register for future use.

Addendum – Low-magnification STEM

LM STEM is an imaging mode where the objective lens is minimally excited and the C2 lens generates a probe with a very small convergence semi-angle (≤0.5 mrad). This provides a large field of view in an environment with minimal, or even zero, magnetic field, depending on the alignment. This makes LM STEM ideally suited for the study of magnetic materials, as well as thick samples, due to its large depth of focus. Additionally, the wide field of view makes LM STEM useful for examining standard grids or for locating lamellae on half-grids.

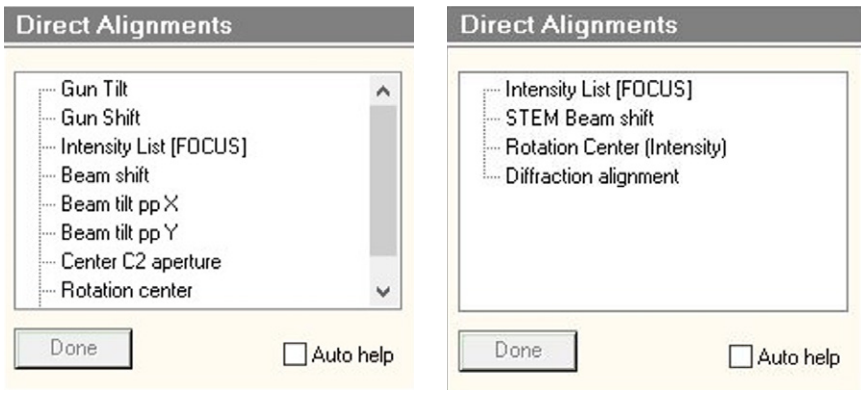
In normal STEM mode, the lowest available magnification is typically 5000x; however, in LM STEM mode, magnifications as low as ~40x can be achieved. The microscope can be adjusted for LM STEM imaging using the following steps.

1. First, check the “Enable LMscan” option in the “STEM Imaging” OCX. Then, turn the magnification knob counterclockwise until you reach the desired low magnification.



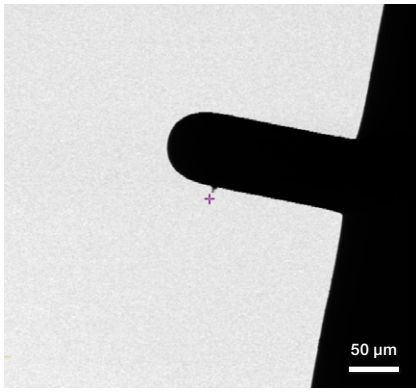
2. Acquire LM STEM images using Velox or TIA Software. Press the “Eucentric focus” button and adjust the sample to the eucentric height using the z buttons. Find a vacuum or amorphous region, stop scanning, unblank the beam if necessary, and ensure that the probe position marker is centered in the image.
3. Switch to probe mode (i.e., diffraction off) by clicking the “Diffraction” button on the right control panel.
4. Find the probe and center it using the “Beam shift” option in the “Direct alignments” OCX.
5. Center the C2 aperture.
6. In the “Direct Alignments” OCX, click on “Intensity List [FOCUS]” and focus the probe using the focus knob.
7. Select “Beam tilt pp x/y” and correct the pivot point by overlapping the spots using the “Multifunction x/y” knobs.
8. Click “Rotation center” to align the beam tilt. The beam will start wobbling; use the “Multifunction x/y” knobs to make it pulse concentrically.
9. Perform a beam shift to center the probe if necessary.
10. Normalize all lenses. Repeat steps 6-9 until there are minimal changes after normalizing all lenses. Then, switch back to diffraction mode by pressing the “Diffraction” button on the right control panel.

11. Click “Diffraction alignment” and center the diffraction pattern on the desired detector. (Note: The camera length in LM STEM mode is typically a few meters.)



Left) Alignment elements in probe mode. Right) Alignment elements in diffraction mode.

12. Start viewing the image in Velox or TIA Software. If the image appears astigmatic, correct it using the condenser stigmator. This can be done by either observing the live LM STEM image directly or parking the beam on an amorphous region to examine the Ronchigram. (Note: Due to the low convergence semi-angle, the spatial resolution in LM STEM mode is typically in the range of a few nanometers.)



Bright-field LM STEM image of a lamella on a half grid.

13. Store or update an FEG register file.

14. To exit LM STEM mode, turn the magnification knob until you return to normal STEM mode. Then, ensure that “Enable LMscan” is unchecked to avoid re-entering LM STEM mode.