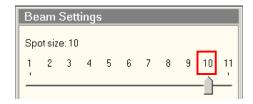
FEI Tecnai F20 S/TEM: atomic resolution STEM imaging Nicholas G. Rudawski ngr@ufl.edu (805) 252-4916 (352) 392-3077

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This document assumes the user is already familiar with operation of the instrument in STEM mode (alignment, imaging, use of TIA, etc) and is working with a single-crystal (or very coarsely grained polycrystalline) specimen.

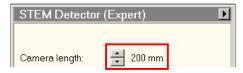
All CBED patterns in this document were simulated using 200 keV electrons,  $\alpha$  = 9.6 mrad (when using the #1 C2 aperture,  $\alpha$  = 9.6 mrad for the actual CBED patterns, too), and a damage-free 20 nm-thick SrTiO<sub>3</sub> specimen oriented along/near B = [001]. The actual CBED patterns you will observe will depend on your material, specimen thickness, presence of any damage layers, and crystallographic orientation; those shown here are for demonstrative purposes only.

- A clean specimen is critical for atomic-resolution STEM imaging, so be sure to plasma clean your specimen before loading into the TEM; you must also use the double tilt holder to ensure you can accurately align your specimen along the necessary zone axis.
- 2. While still in TEM mode, identify your area of interest and align it along the necessary zone axis as best as possible (fine adjustments will be made in STEM mode); make sure the specimen is at eucentric height when finished.
- 3. Enter STEM mode, set spot size = 10, and perform STEM alignment (rotation centering and condenser stigmation); select and center the #1 C2 aperture; for atomic-resolution STEM imaging, it is critical that the instrument is aligned as well as possible at these specific instrument settings.

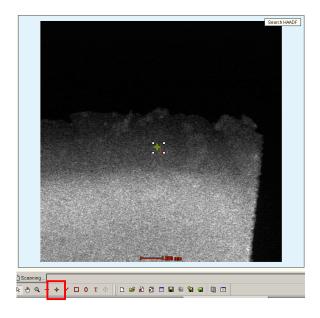


## 4. Crystallographic alignment

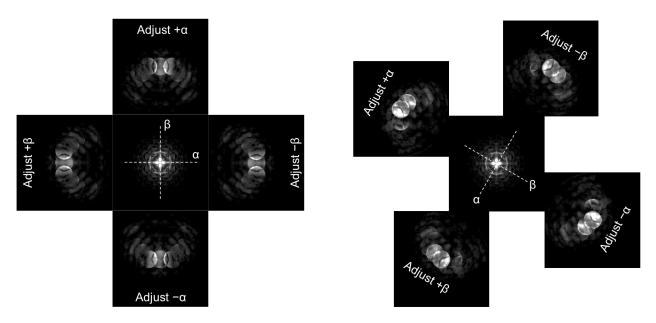
4.1. A camera length of L = 100 – 200 mm is usually sufficient for crystallographic alignment purposes; L = 200 mm will orient the CBED pattern in a way that makes specimen tilting more intuitive; L < 200 mm will show more of the CBED pattern, but at the expense of orienting the CBED pattern in a way that makes specimen tilting less intuitive.



- 4.2. The highest quality atomic-resolution STEM images will be obtained when the sample is as closely aligned along a major crystallographic zone axis as possible. To check the crystallographic alignment, you must look at the CBED pattern on the focusing screen. To complicate matters, you must also achieve this alignment at your desired area of interest (e.g. at a film/substrate interface).
- 4.3. Acquire a live STEM image in TIA using "Search" mode, center your region of interest in the image, and then freeze the image. In TIA, insert a beam position marker onto the image; when you click and drag on the marker, the CBED pattern observed on the viewing screen will correspond to the beam position marker position.

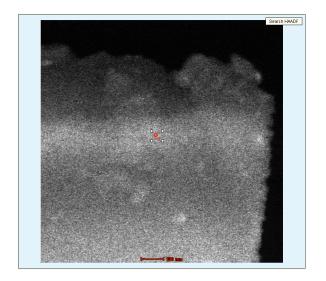


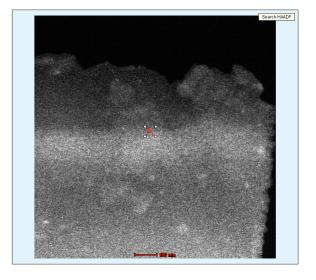
- 4.4. Use the following iterative process to align the sample along the zone axis:
  - 4.4.1. Move the beam position marker to your area of interest and observe the CBED pattern on the focusing screen.
  - 4.4.2. Make an appropriate change in either  $\alpha$  or  $\beta$  tilt (see below for an indication of how to tilt to converge on the zone axis based on L and the appearance of the CBED pattern).



Tilting to the zone axis: when  $L \ge 200$  mm (left) and when  $L \le 150$  mm (right)

4.4.3. Start acquiring a live STEM image again "Search" mode (it will now probably be out of focus) and roughly refocus using the stage Z control (DO NOT refocus using the "Focus" knob at this point).

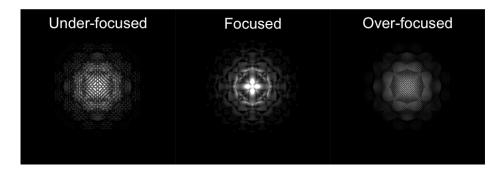




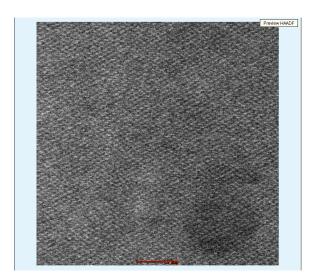
- 4.4.4. Re-center the region of interest in the STEM image and then freeze the STEM image again.
- 4.4.5. Move the beam position marker back over to the area of interest and look at the CBED pattern again (you should be closer to the zone axis than you were before).
- 4.4.6. Repeat this process until alignment with the zone axis for your region of interest has been achieved as indicated by the CBED pattern (which will likely take many iterations). With skill, you should be able to achieve zone axis alignment with <1 mrad precision.
- 4.4.7. Once zone axis alignment is complete, delete the beam position marker from the image.
- 5. The camera length can now be adjusted to produce the desired STEM image (100 mm will produce a HAADF-STEM image); likewise, if the CBED pattern is not centered inside the detector rim, this can be adjusted now using "Diffraction alignment".



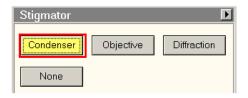
- 6. Fine tuning and image acquisition
  - 6.1. Using "Search" mode, center the region of interest in the image and set the indicated magnification in TIA to 7.2 M× (this can be adjusted later, but is appropriate for fine tuning of the image).
  - 6.2. Freeze the live image and observe the CBED pattern on the viewing screen; adjust "Focus" (focus step = 2) until the probe is focused on the sample (when focused, the CBED pattern will not exhibit any features resembling lattice fringes, unlike the under- and over-focused examples shown below).

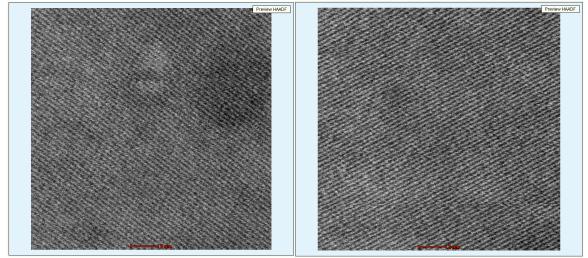


6.3. Start live imaging again using "Preview" mode; set focus step = 1 and adjust "Focus" until the sharpest atomic resolution possible is evident in the image (this can only be done effectively with focus step = 1).

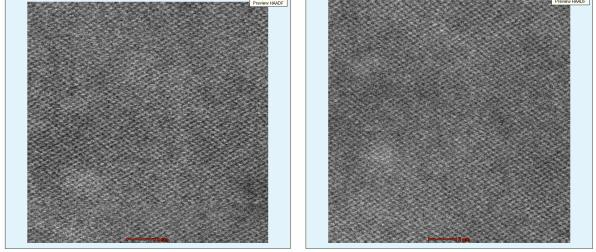


6.4. Activate the condenser stigmators and adjust the "Multifunction" knobs to remove any remnant astigmatism from the probe (use the finest possible setting); astigmatism will show up as preferential streaking that reorients by ~90° as the focal condition goes from under- to over-focus; if no such streaking is evident as the focal condition is changed, then the astigmatism has been properly corrected.





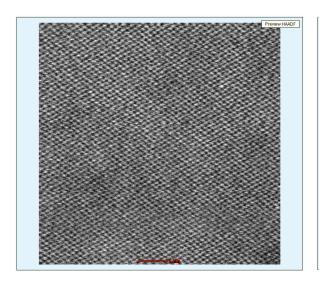
Astigmatic atomic-resolution STEM images: under- (left) and over-focused (right)

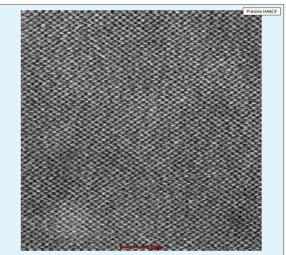


Non-astigmatic atomic-resolution STEM images: under- (left) and over-focused (right)

6.5. The scanning pattern of the STEM image can be rotated to align certain crystallographic directions with the vertical and horizontal image directions.

While acquiring a live image using "Preview" mode, adjust until the STEM image is oriented as desired; keep in mind that a negative rotation is a clockwise rotation.

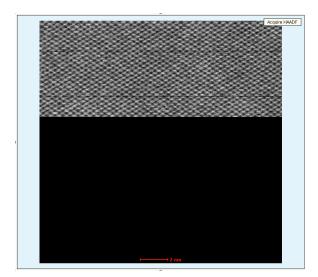




Rotation:

-0.0 deg

6.6. Finely focus the image, set the magnification as needed, perform auto contrast/brightness, and then use "Acquire" mode to acquire the final image. A "Dwell time" of 30 – 60 μs is usually sufficient for this (if adjusted from the initial value, acquisition will automatically restart).



6.7. Due to the long acquisition times needed for STEM imaging, a stable specimen is necessary to obtain images with minimal distortion; it may take 0.5 – 1.0 h (or longer) after loading of a specimen for it stabilize sufficiently such that an atomic-resolution STEM image with minimal drift can be produced (so be patient if the sample doesn't seem to be sitting still).

## Appendix: shorthand procedure for performing atomic-resolution STEM imaging

- 1. Find area of interest in TEM mode
  - a. Set area at eucentric height
  - b. Align area along zone axis as best as possible
- 2. Enter STEM mode
- 3. Set spot size = 10
- 4. Perform STEM alignment
  - a. Rotation centering
  - b. Correct condenser astigmatism
  - c. Align #1 C2 aperture
- 5. Select camera length for crystallographic alignment purposes
- 6. Align region of interest along crystallographic zone axis
  - a. Freeze scanning
  - b. Position beam position marker
  - c. Check CBED pattern
  - d. Adjust tilt
  - e. Resume scanning
  - f. Adjust stage Z to roughly focus image
  - g. Repeat until alignment achieved
- 7. Set camera length as intended for imaging
- 8. Center CBED pattern relative to STEM detector
- 9. Use "Search" mode and center region of interest in image
- 10. Set magnification =  $7.2 \text{ M} \times$
- 11. Freeze scanning
- 12. Observe CBED pattern on focusing screen
- 13. Focus probe on sample (focus step = 2)
- 14. Start live imaging in "Preview" mode
- 15. Adjust focus (focus step =1)
- 16. Use condenser stigmators to stigmate image
- 17. Adjust scan rotation (if needed)
- 18. Finely focus image, set magnification as needed
- 19. Acquire final image