FEI Themis Z S/TEM: EFTEM
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ANALYSIS OF RADIOACTIVE SPECIMENS IS <u>STRICTLY</u> PROHIBITED

This document assumes the user is familiar with and competent in operation of the Themis Z S/TEM in TEM mode.

1. Instrument voltage

    1.1. It is recommended to perform EFTEM at the default voltage setting of 300 kV unless your specimen cannot tolerate it for some reason. If you need to perform EFTEM at a lower voltage (60 or 200 kV), please contact staff for assistance setting up the instrument and GIF.

2. Instrument alignment/settings for EFTEM

    2.1. Perform the same basic alignment in TEM mode before switching to EFTEM mode noting the following:

        2.1.1. Use the 100 µm C2 aperture

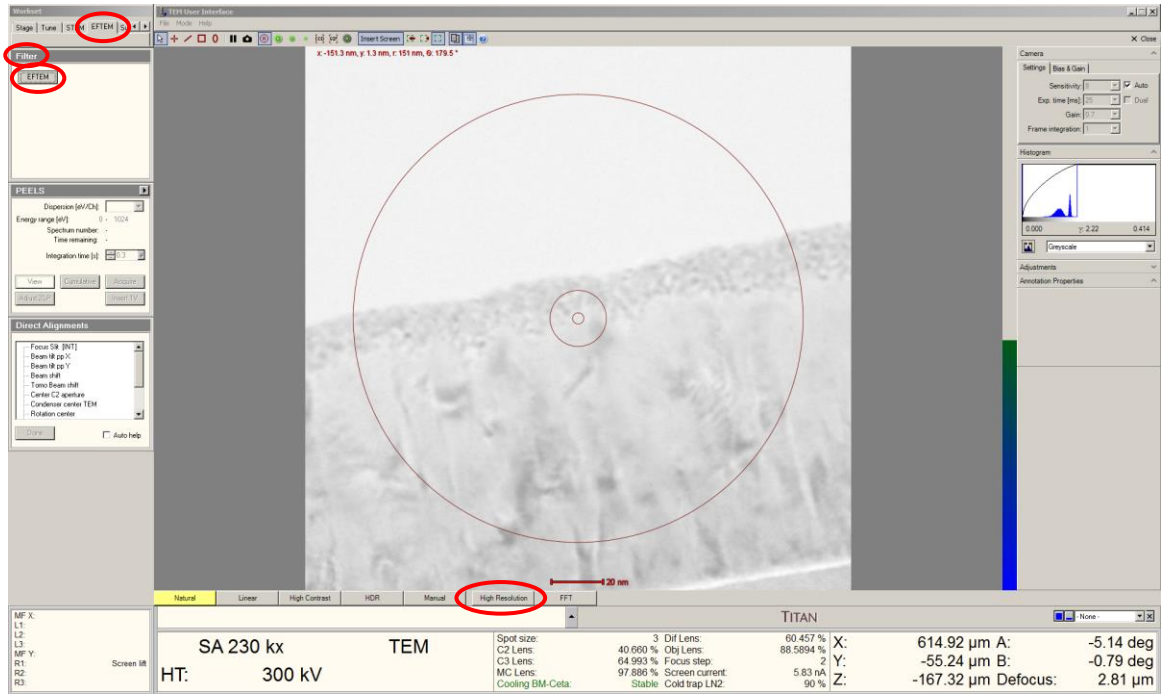
        2.1.2. Insert/center the 40 µm objective aperture; this will define the collection angle for the GIF.

    2.2. Verify the Ceta camera and all microscope STEM detectors are retracted.

3. Switching to EFTEM mode

3.1. In Microscope Control, select the “EFTEM” tab, navigate to the “Filter” control panel, and select “EFTEM”.

3.2. Activate the “High Resolution” display mode for the FluCam.

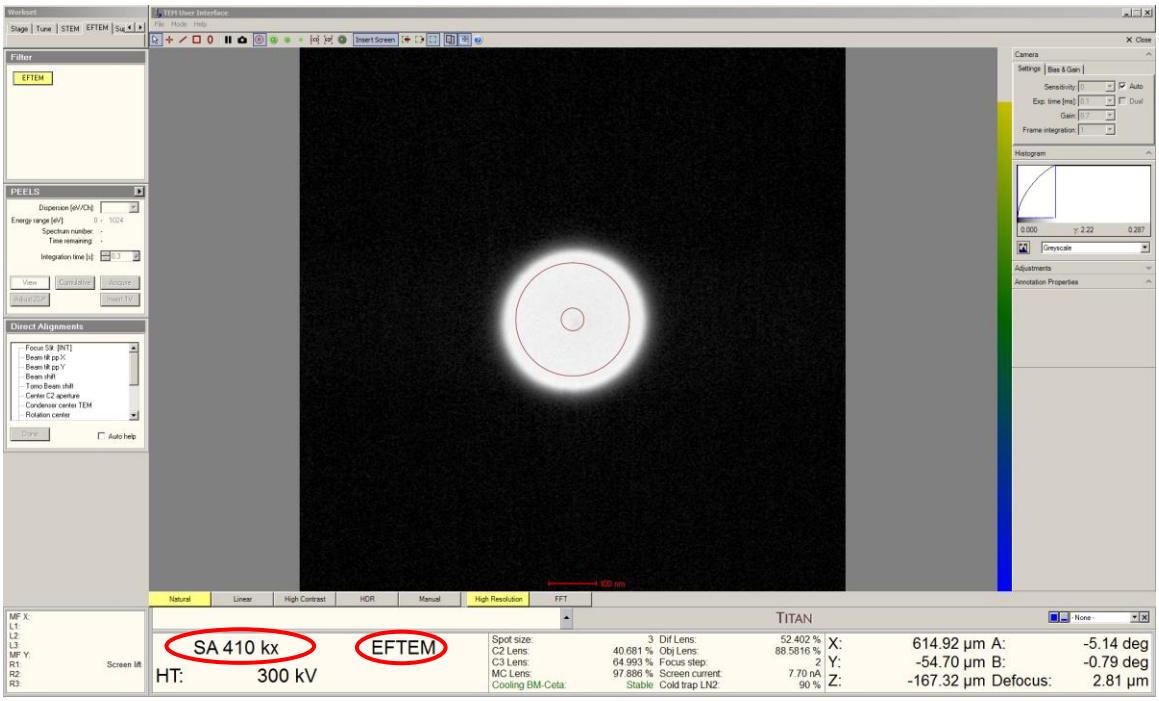


4. Working in EFTEM mode

4.1. SA and Mh modes are available in EFTEM just like regular TEM mode; if the mode is switched from SA to Mh, the same considerations with regards to alignment adjustments for optimal performance will still apply.

4.1.1. If needed, the same alignments performed in TEM mode using the FluCam can also be performed in EFTEM mode; the only difference is that the image will appear much smaller on the FluCam.

4.2. Optimal illumination for EFTEM is with the beam centered on the FluCam and expanded just outside the second circle mark.

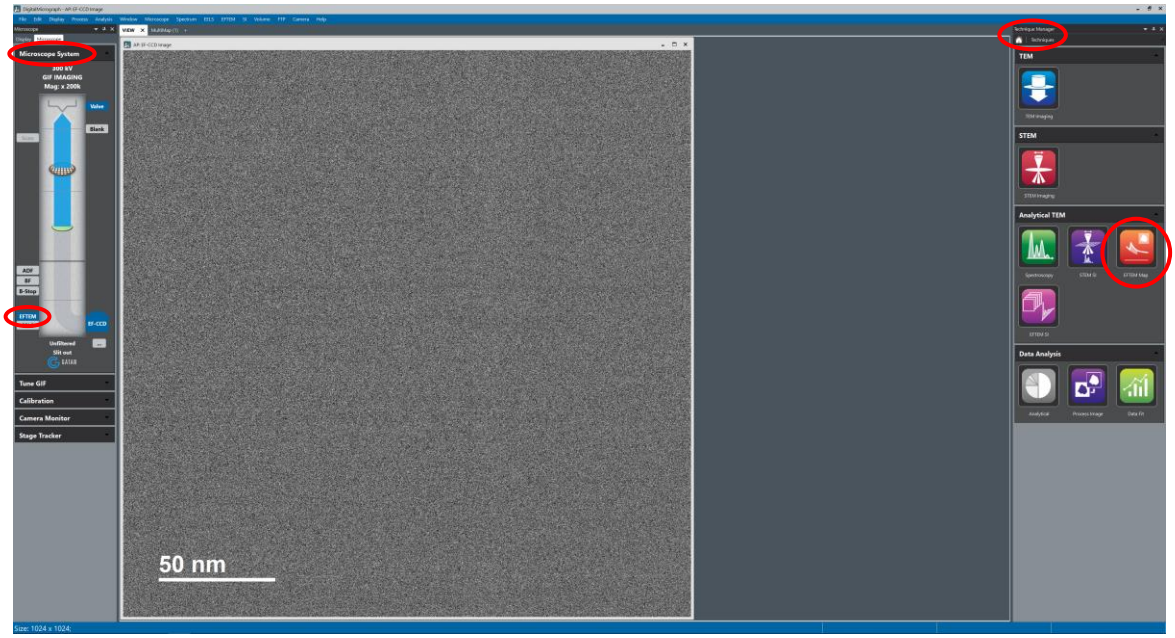


4.3. When performing EFTEM, the indicated magnification and/or illumination should only be adjusted with the viewing screen down using the FluCam; do adjust either of these while live imaging using the GIF camera.

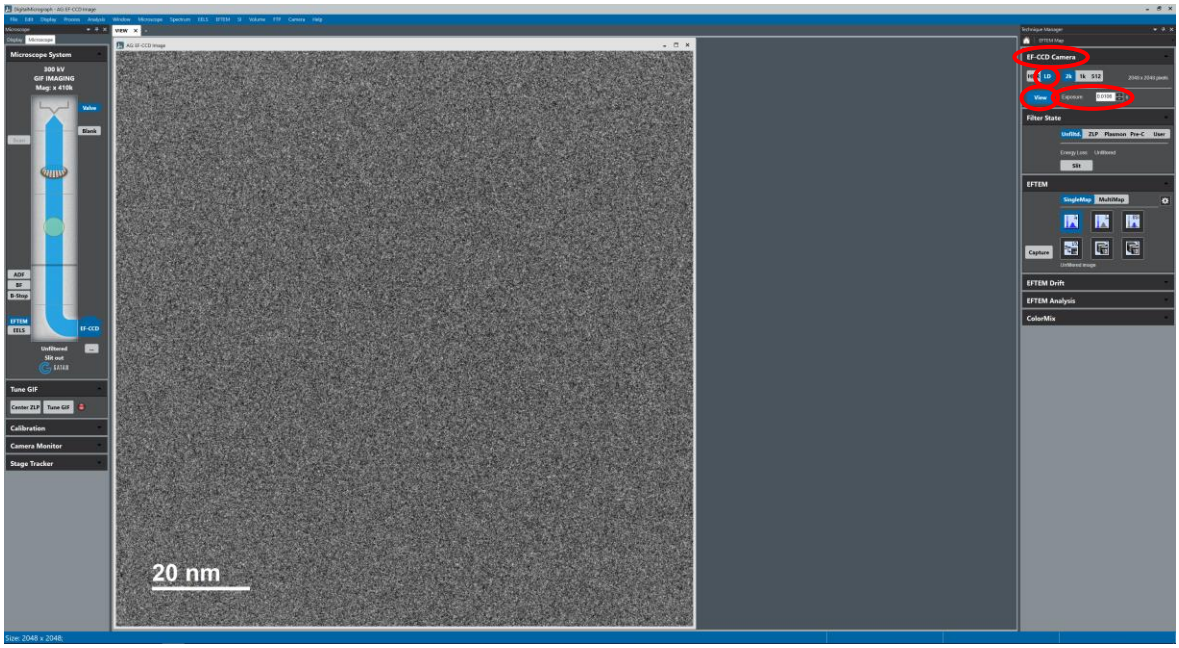
5. GIF imaging and tuning

5.1. In DigitalMicrograph, navigate to the “Technique Manager” window and select the “Home” icon; then select “EFTEM Map”.

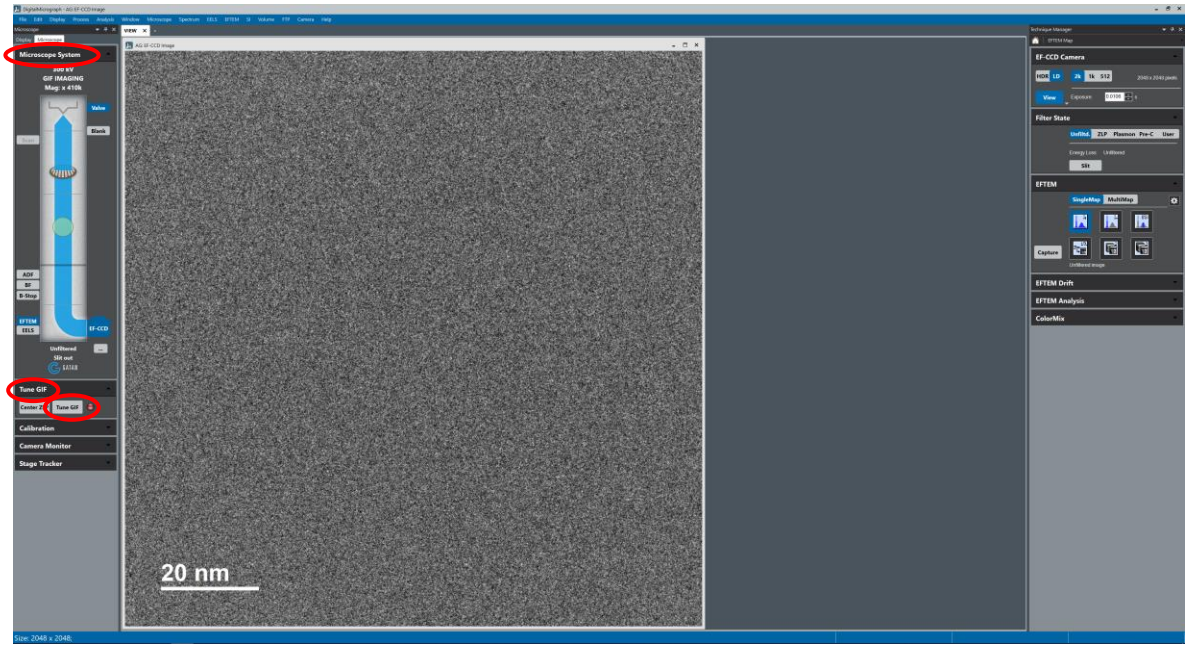
5.1.1. If you are prompted to switch to “EFTEM” mode, select “OK”.



5.2. Navigate to the “EF-CCD Camera” panel; select “LD” and input an exposure time of 0.0106 s; then select “View” and retract the viewing screen to place the beam on the GIF camera.



5.3. Move the sample out of the field of view so the beam is illuminating only vacuum; navigate to the “Microscope System” panel; in the “Tune GIF” panel, select “Tune GIF”.



5.4. If the magnification needs to be changed, it should be done with the viewing screen inserted using the FluCam and the beam size/position adjusted accordingly as per 4.2 and 4.3; do not perform these adjustments while live imaging using the GIF camera.

5.5. If images are to be collected at a new magnification, GIF tuning must be performed in vacuum again at the new magnification as per 5.3 (unless tuning was already performed for that magnification during the session).

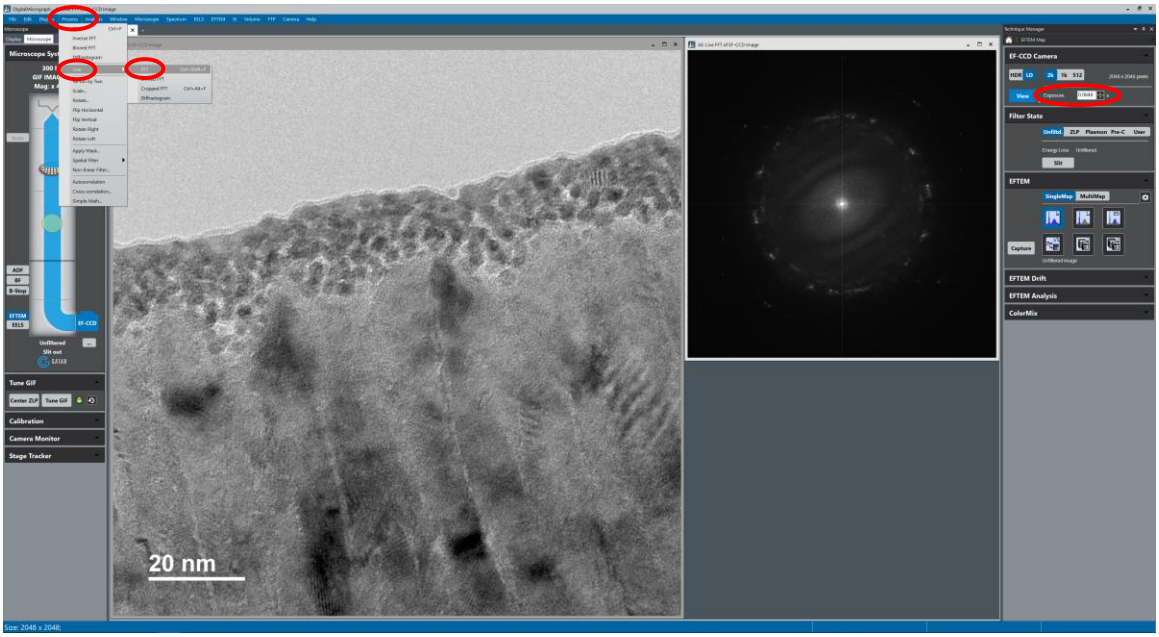
6. Zero-loss high-resolution imaging

6.1. Set the indicated magnification as appropriate for high-resolution imaging and tune the GIF for said magnification (if needed);

6.1.1. NOTE: the indicated magnification needs to be ≥~200000× for effective high-resolution imaging with the GIF camera.

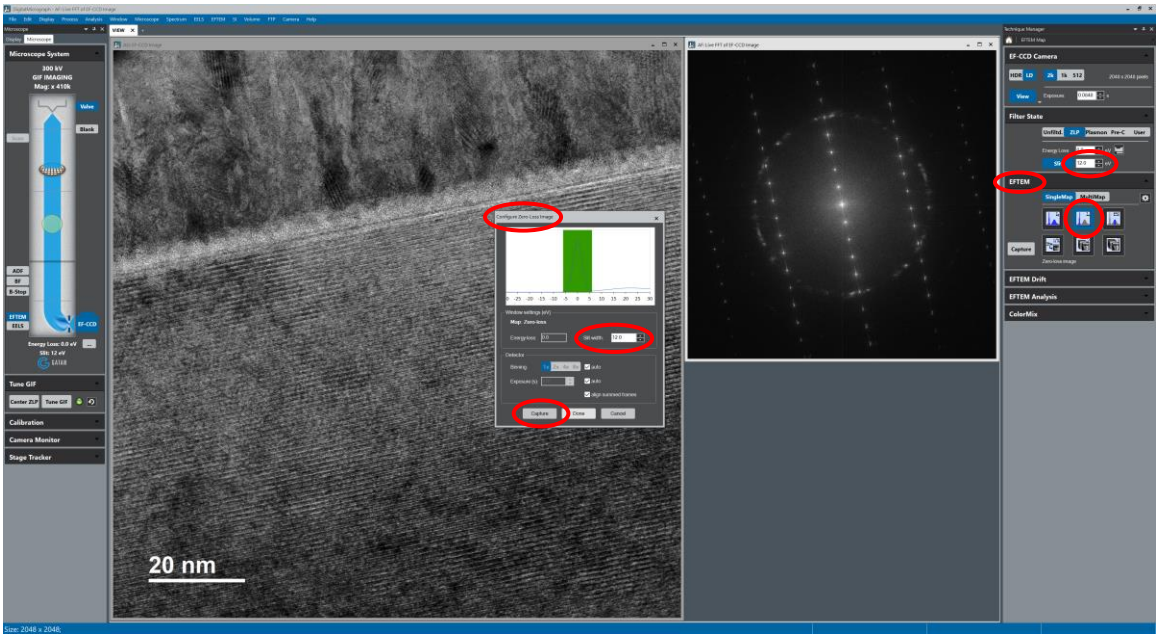
6.2. Start acquiring a live image with the GIF camera; adjust the exposure time as needed.

6.3. From the “Process” pull-down menu, select “Live” and then “FFT”; correct the objective astigmatism using the objective stigmators in Microscope Control as per usual.



6.4. Navigate to the “Filter State” panel and select “ZLP”; note the presence of any dark splotches near the edges of the image; increase the slit width in 1 eV increments until the splotches go away (note the slit width).

6.5. Navigate to the “EFTEM” panel and select the icon for “Zero-loss Image”; in the “Configure Zero-Loss Image” dialogue box, specify the slit width just noted and then select “Capture” to acquire the final zero loss image.

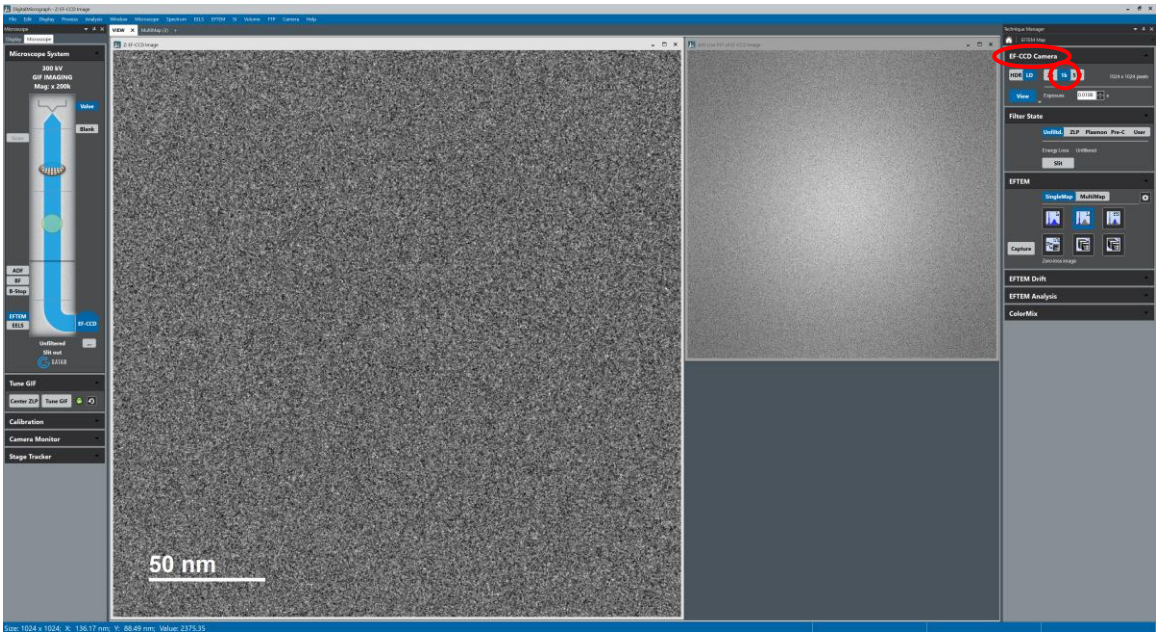


7. Element Mapping

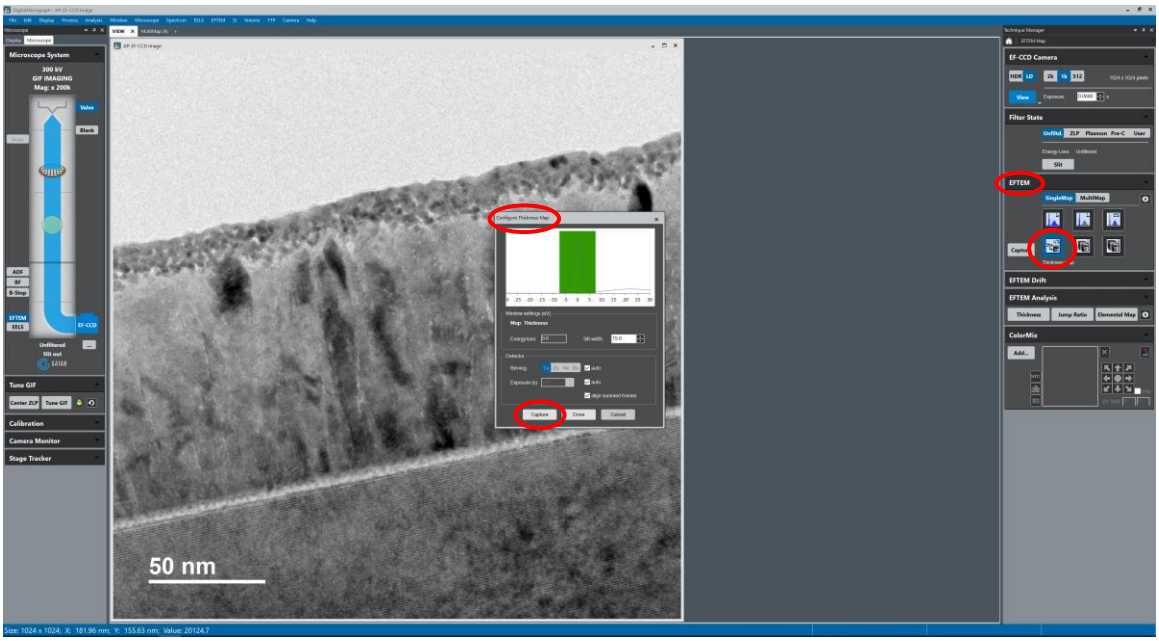
7.1. Set the indicated magnification as appropriate and tune the GIF for said magnification (if needed).

7.1.1. NOTE: the spatial resolution when performing element mapping is 1 – 2 nm; thus, there is little effective benefit in using an indicated magnification > ~200000×.

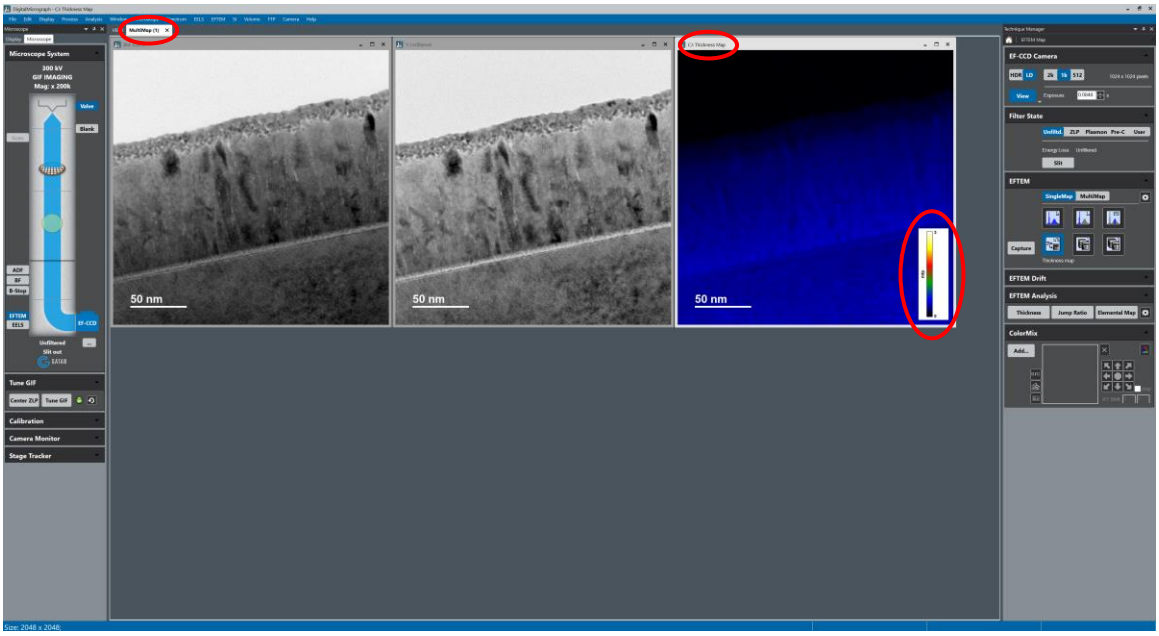
7.2. Navigate to the “EF-CCD Camera” panel; set the resolution to 1k and start acquiring a live image.



7.3. Navigate to the “EFTEM” panel and select the icon for “Thickness Map”; in the “Configure Thickness Map” dialogue box, select “Capture”.

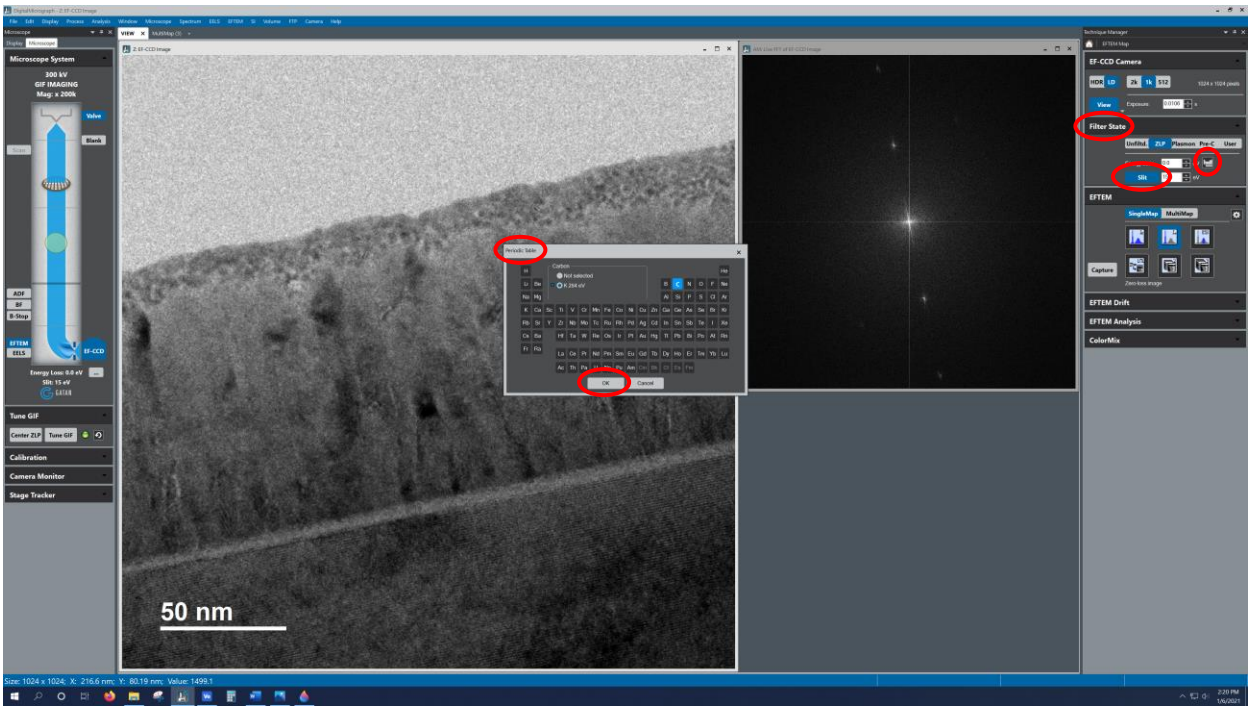


7.4. A new workspace with zero-loss, unfiltered, and thickness map images will be generated; examine the thickness map; the values need to be ≤ ~1 MFP for effective element mapping; if not, the area is probably too thick.



7.5. IMPORTANT: before proceeding to the next step, insert the viewing screen so the beam is blocked from entering the GIF.

7.6. Navigate to the “Filter State” panel; if not already inserted, select “Slit” to insert the slit (“Slit” button will be blue) and then select the periodic table; in the “Period Table” window, select the element and core loss edge of interest and select “OK”.

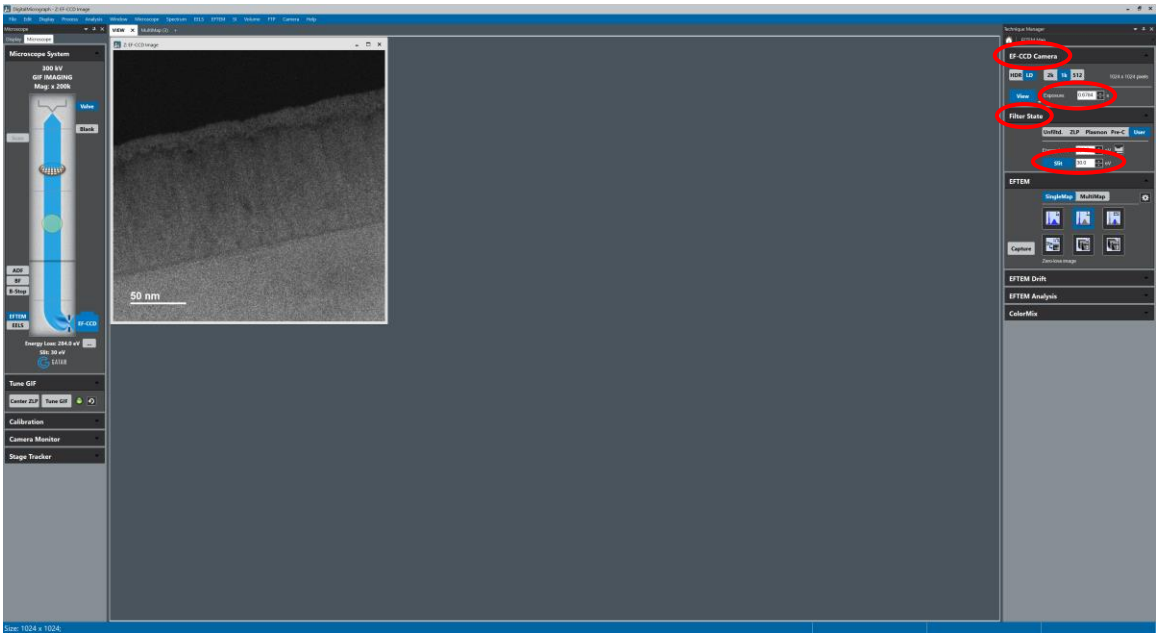


7.7. The beam size/position as observed on the FluCam will change as a result of the new filter configuration; resize/reposition the beam accordingly and then retract the viewing screen to allow the beam to enter the GIF.

7.8. The resulting image will likely show a very weak signal; to increase the signal, try the following:

7.8.1. Navigate to the “EF-CCD Camera” panel and increase the exposure time.

7.8.2. Navigate to the “Filter State” panel and increase the slit width (noting the final value); <u>in general, the slit width should not exceed ~10 % of the value of the core loss edge</u>

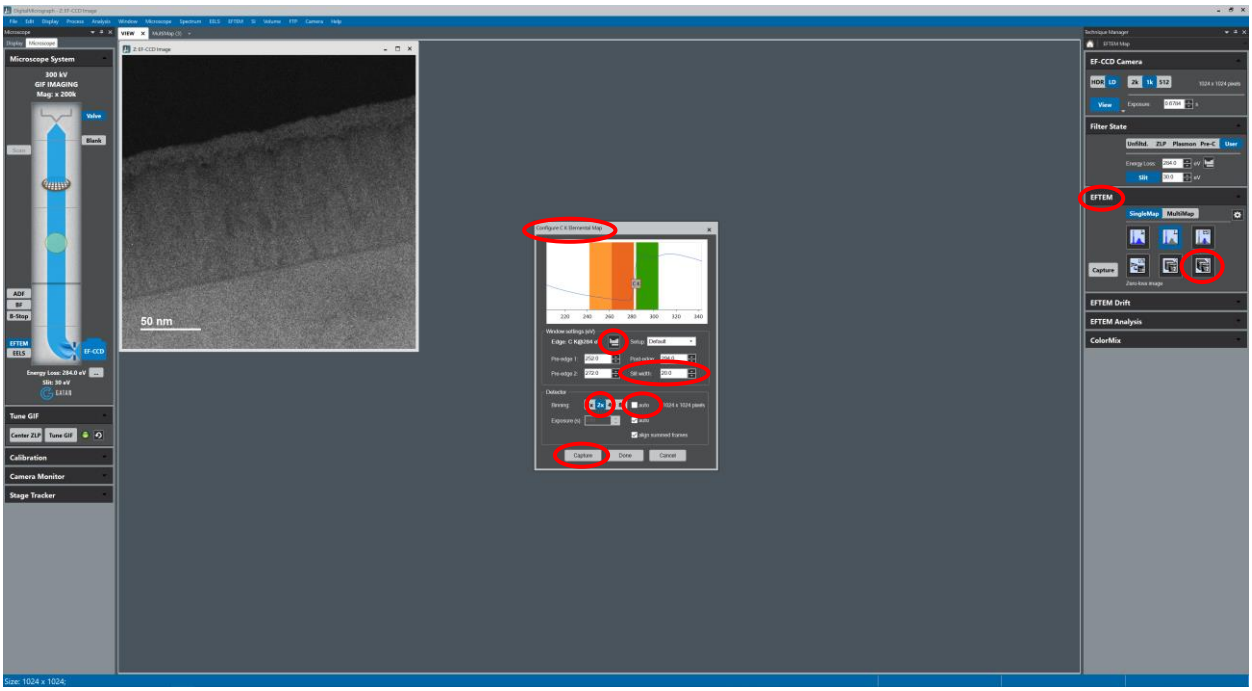


7.9. The optimal focus for the image will depend greatly on the energy of the core loss edge; adjust the “Focus” accordingly until the sharpest possible image is obtained.

7.9.1. This can be difficult to do because the exposure time will usually be rather long and/or the image signal rather weak.

7.10. Navigate to the “EFTEM” panel and select the icon for “Elemental Map”; in the “Configure Elemental Map” dialogue box, select the periodic table icon and select the current core loss edge; next to “Binning”, uncheck “auto” and select “2×”; select “Capture” to start acquiring the map.

7.10.1. NOTE: if desired, the slit width can be increased from the default value to the value obtained in 7.8.2; <u>in general, the slit width should not exceed ~10 % of the value of the core loss edge</u>.

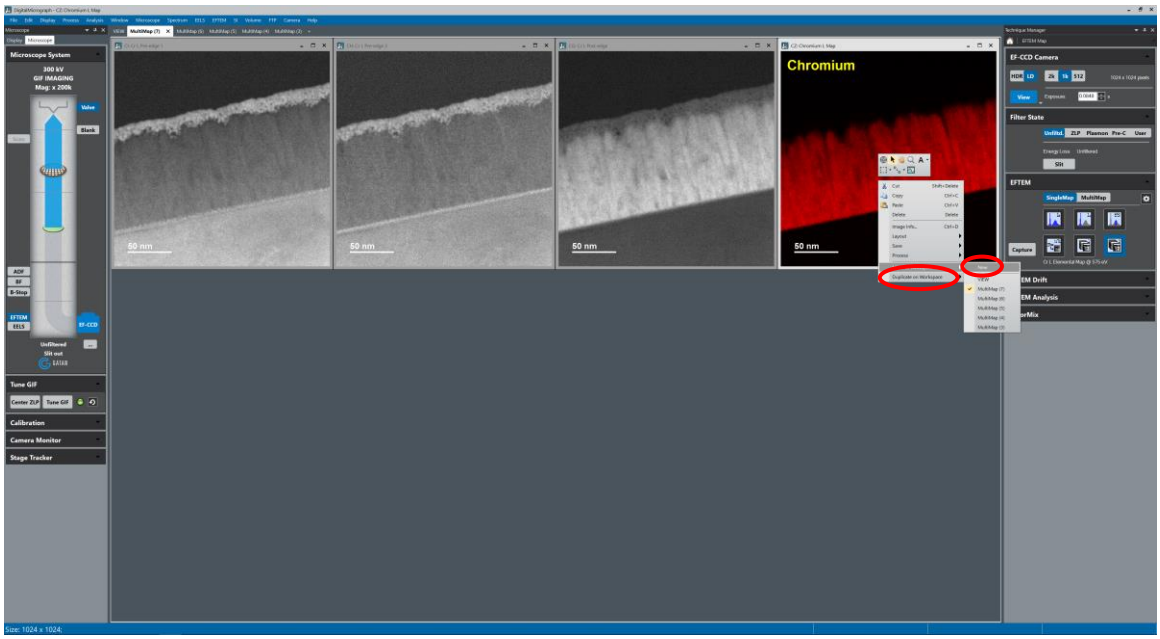


7.11. The element map (along with three other images used to generate the map) will be generated in a new workspace.

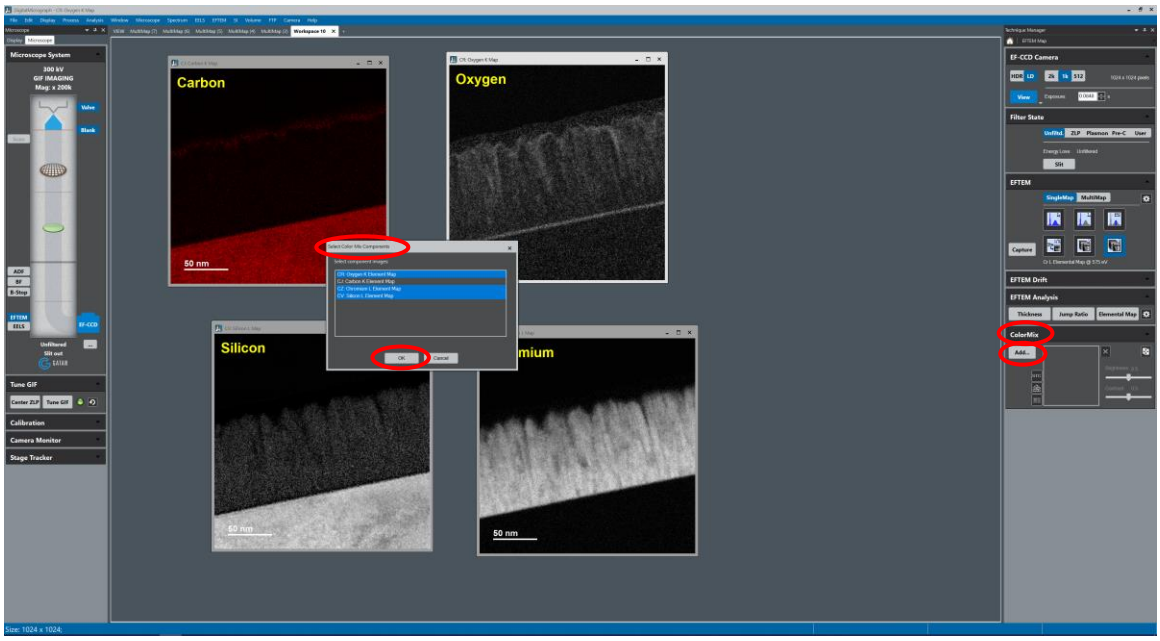
7.12. Repeat 7.5 – 7.10 for each element of interest; a new workspace will be generated for each individual element map (and the three associated images).

8. Composite element maps.

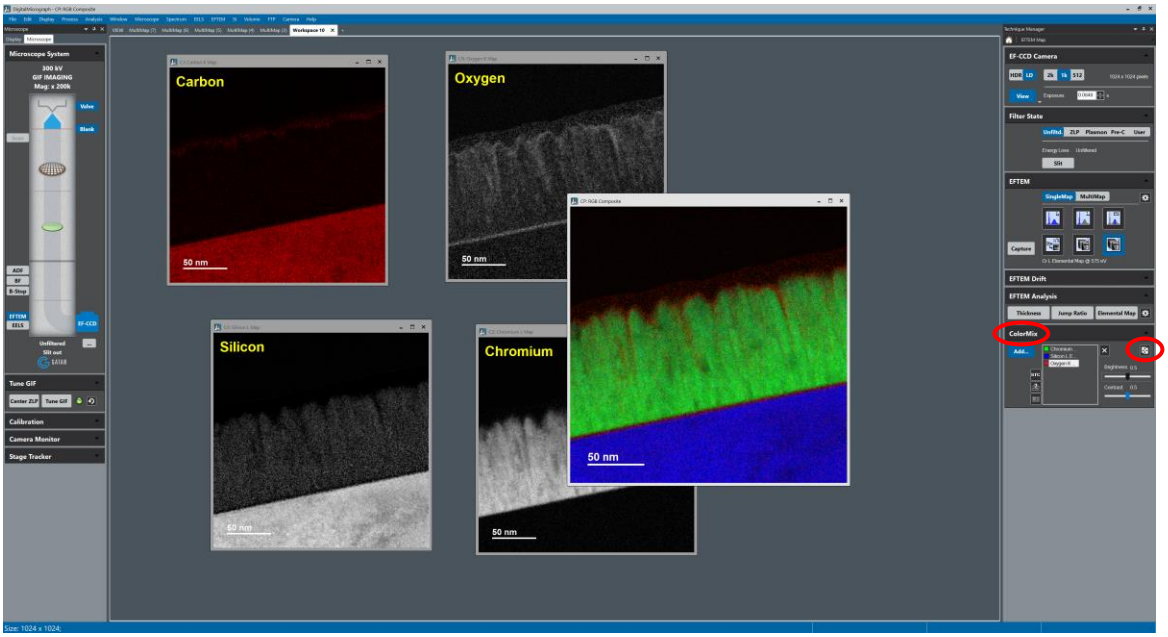
8.1. Right click on an element map; select “Duplicate on Workspace” and then “New” to generate a new workspace; right click on the other element maps and duplicate each one on the same newly created workspace.



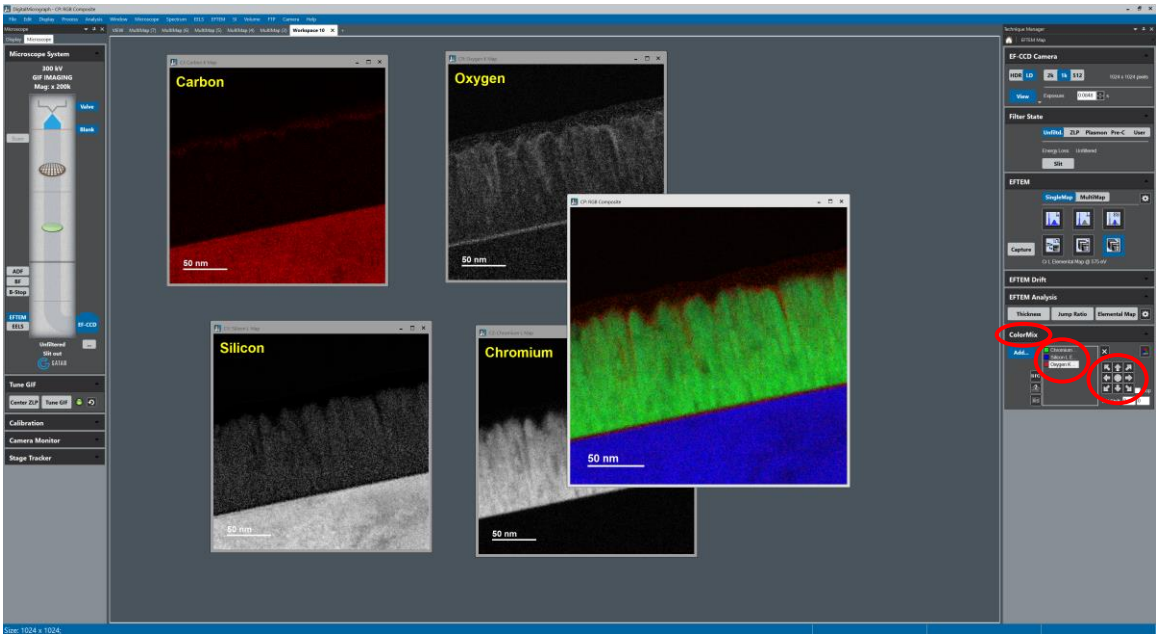
8.2. Navigate to the newly generate workspace with the element maps of interest; navigate to the “ColorMix” panel and select “Add”; in the “Select Color Mix Components” dialogue box, select the maps of interest and select “OK”.



8.3. There may be some slight shifting of the individual element maps relative to each other; to adjust this (if needed), navigate to the “ColorMix” panel and select the positioning icon.



8.4. Select an element map from the list and then adjust the position with the arrows; repeat for the other maps as needed.

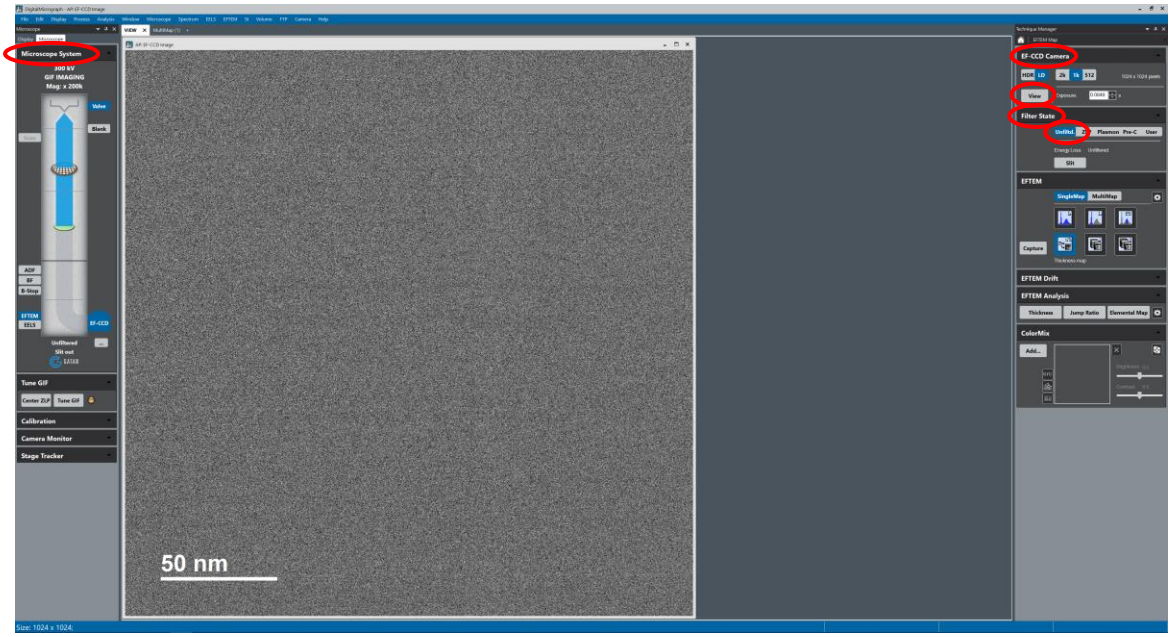


9. Finishing

9.1. Insert the viewing screen to block the beam from entering the GIF.

9.2. Navigate to the “EF-CCD Camera” panel and select “View” to stop acquiring a live image (“View” button will be gray).

9.3. Navigate to the “Filter State” panel and select “Unfiltd.” (“Slit” button will be gray); the “Microscope System” panel should look similar to as shown below.



9.4. In Microscope Control, select the “EFTEM” tab; navigate to the “Filter” control panel and select “EFTEM”

9.5. Deactivate “High Resolution” mode for the FluCam.