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Talos F200X Standard Operating Procedure



These instructions are intended for reference only, and will *not* replace the thorough training required for proper system operation. Contact electron microscopy staff with questions or to report a problem.





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Enable the tool in **BADGER** 1 **CNI Membership** Reservations FEI Talos F20 Columbia Nano Initiative Multiple D CLEANROOM SMCL 8:00 8:30 Electron Microscopy 9:00 ♠ FEI Talos F200X S-TEM* 9:30 Enable 00 ASTAR Analysis PC <u>D</u>isable ZEISS SEM* (sc4698@ bo Make Reservation 30 A EBSD Analysis PC Request Work APIPS II* **S**hutdown bo 30 Light Zeiss Microscope* Report Problem 00 Make Comment A Dimple Grinder* Search PCS 00 A Plasma Cleaner* 30 **Q**ualify User 🃤 Grinder-Polisher* 00 Browse Manual 30 Diamond Saw* 00 Training Calendar **Vacuum:** Look at the pressure **AVAILABLE Pressure Readouts** values for the system. Accelerator 1 Log Column 1 Log The **Column** reading should be **Detection Unit** 14 Log below 20, and the system must be **Nitrogen Level** V_{ac} Status 86 % Available before starting. **Stage Position** V_{ci} Nitrogen level should be above $1.41 \, \mu m$ α 0.00° -0.10 μm β 0.00° 10%. 0.01 µm Otherwise contact EM staff.



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3 Loading the TEM grid to the holder: Wear gloves!

Turn on the stereoscope light by pressing the blue M button on the right side (pressing it 3 times, turn it off).

Place the single tilt holder (located on the second drawer of desiccator) on the table. Remove the plastic cap.

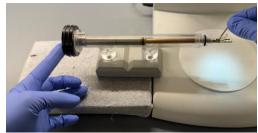
Use the pin to lift the sample clamp. Put your left-hand finger on the back to prevent the holder from moving.

Place the sample in the middle of the ring.

Shown below:











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Cont... The TEM grid should lay flat. Video on holder loading: youtu.be/qDFljfuem3I **AVAILABLE Pressure Readouts** 1 Log Accelerator Load the Holder: Column 1 Log **Detection Unit** 14 Log On the microscope touch screen **Nitrogen Level** press "Load Sample". Wait a few V_{ac} Status 86 % seconds till it stabilizes. **Stage Position** V_{ci} $1.41 \, \mu m$ α 0.00° -0.10 μm β 0.00° 0.01 µm Load Sample





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5 Insert holder:

Carefully insert the holder, with the holder pin aligned at the 5 o'clock position, as far as it will go into the CompuStage.

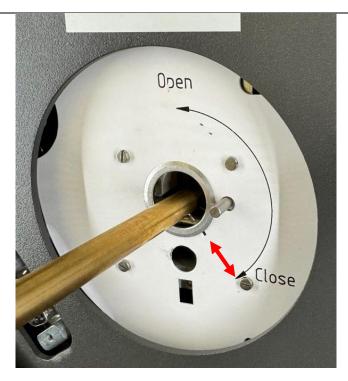
Be careful not to scrape the tip.

You will feel a slight pressure when the black O-ring hits the mating seal. Push firmly but carefully until the holder goes in about 1 cm further and stops at the final position of the mating seal.

The black line marker on the holder will be aligned with the white panel.

If the holder is misaligned, and can't go till the black line, take it out and try again.

Waiting more than 5s while the holder is misaligned will cause vacuum failure.









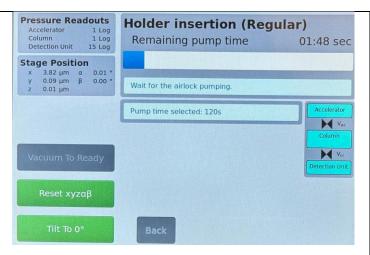
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6 Pumping load lock:

The

remaining pumping time for the specimen exchange is shown on the TEM screen.

If you are using the double-tilt holder, you need to connect the cable and select double-tilt from the "Select Holder Type" menu.



7 | Final insertion:

When the pumping cycle ends (red light on the CompuStage turns off), gently rotate the holder pin counter-clockwise from 5 o'clock (close) to 12 o'clock (open) position.

Gently guide the holder into the column.

Do not let go of the holder at this point!

Tap the end of the holder gently to ensure stabilization.

(*Note*: For high-resolution work, you may have to wait 30 minutes or more for the O-ring seal to stop drifting.)







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vacuum contact EM staff member immediately. They will recover the vacuum.

In the event of dumping the Vacuum recovery may take from half an hour up to several hours.

> If you need more practice on sample loading, please ask EM staff or an experienced user to be with you.

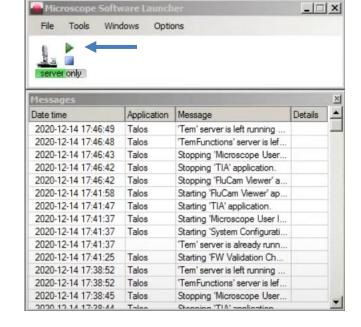
Video on operating the TEM software: youtu.be/NkHjQKrpX2U

> Click on the 'Start' or triangle button to open the TEM software package:

- -TEM User Interface
- -TEM imaging and analysis (TIA)
- -Flucam Viewer

Drag the TIA program to the right monitor and maximize it.







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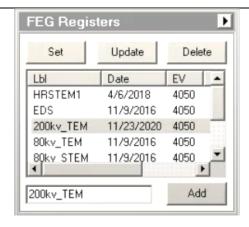
10 Column valve: Ensure the Column pressure is below 20 log Setup | Search | Camera | Tune | Sti ◀ ▶ and press "Col. Valves closed" button to open it. It becomes Vacuum (Supervisor) grey when it is open. Status: All Vacuum (Closed) Accelerator Log Column Log Detection Unit Log Buffer tank Log Backing line Nitrogen level 41 % Col. Valves Empty Buffer Closed **11** | Flucam viewer: A live TEM image R + / □ 0 III **b** ◎ ◎ ◎ will automatically appear in the Flucam Viewer. If image does not Column Valves Closed Screen Inserted appear, check that Flucam viewer is in an un-paused position and screen is inserted ("insert screen" is blue). It's easier to find image at low magnification. If the image is too dark (looking at the grid or thick sample) use joystick to move the sample to the carbon film window

or to a thinner area.



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Registers window, select the 200kv_TEM then click "Set" to load optimized working conditions.



13 Finding the correct Z height of your sample (eucentric height):

Bring some feature/particle to the center of the FluCam. Then go to Search tab, then click on the small triangle to get to the stage control panel.

Click on the "Wobbler" button.

You see the feature on the screen goes left to right when the holder tilts.

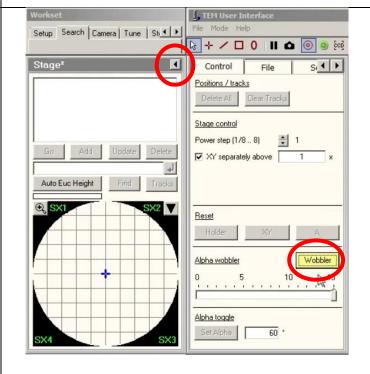
You can also activate Wobbler by pressing L2 on the panel in front of you.

14 Start by pressing the lower Z button and watch if the movement of the sample gets less and less.

Currently the focal Z height on our TEM is in the range of -40 μ m to - 100 μ m.

You can also observe the contrast of your sample is getting less and less, becoming whiter.

Stop the Wobbler.











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Video on adjusting mag, intensity 15 and imaging:

https://youtu.be/d-F jV0AFSE

Magnification and Intensity:

Move the stage with the joystick to the area of interest then increase the "Magnification" to a desired value (clockwise).

You will see the screen is getting darker.



Use the intensity knob to adjust the Screen current between 1-2 nA. Rotate it clockwise to reduce the intensity.



Focus step: 1.60 nA Screen current: Obj Lens: 94.4317 % Cooling BM-Ceta: Stable





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16 Taking Image:

Go to the Camera tab. Then click "Insert" to bring the high-resolution camera in. The button becomes yellow.

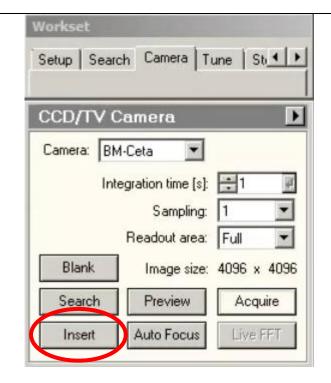
Click on the "Insert Screen" to remove the Fluoresce screen. The beam will be able to reach the high-res camera.

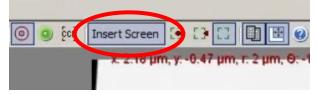
Click on the "Search" button to get an overview of your sample on the right monitor.

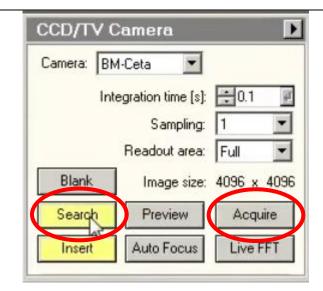
Click "Acquire" to capture that image.

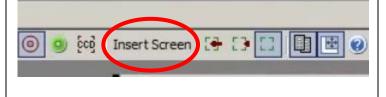
When the image appears, click on the "Insert Screen" to bring back the Flu-screen.

This also prevents the camera to be exposed to the beam for too long.















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18 If you need to change the magnification, do it over the Flu-Screen. Not on the high-res Camera. X Cut 19 Save Image: С Сору Right click on the image that you X Delete have acquired. Then click "Export Data". 🔓 Lock <u>A</u>utoscale Vertical Autoscale Horizontal Autoscale Save Selection. Arrange Panes Split Pane Horizontally Split Pane Vertically Info... Properties. Statistics... Print Image Preview Print Image Save in: Desktop · 🕝 🗷 📂 🖽 · Go to Transfer folder, find your folder with your name (if you don't have one, create a new folder with your name and UNI). update_9922697_23.10.2... Talos_1.15.1 Acquire CCD .tif PC TIFF (full res) (*.tif) TiO2 Anatase 36kX 01.tif File name: Save as type: PC TIFF (full res) (*.tif) Make sure click on the file type PC TIFF (full res) (*.tif) "with scale maker" PC TIFF w/scale marker (full res) (*.tif) PC TIFF w/o arlays (screen res) (*.tif) PC TIFF(16 Bits) (full res) (*.tif) PC TIFF(16 Bits) w/scale marker (full res) (*tif) Bitmap (BMP) (full res) (*.bmp) Bitmap (BMP) w/scale marker (full res) (*.bmp) Bitmap (BMP) w/overlays (screen res) (*.bmp)



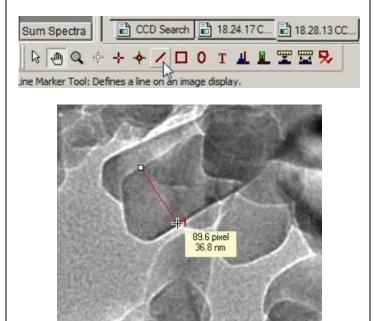


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20 Measurement on your image:

From the tool bar in the TIA program, you can click on the "Line" tool.

Click anywhere on your sample and drag the line to see the particle/feature size.







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21 Closing your Session:

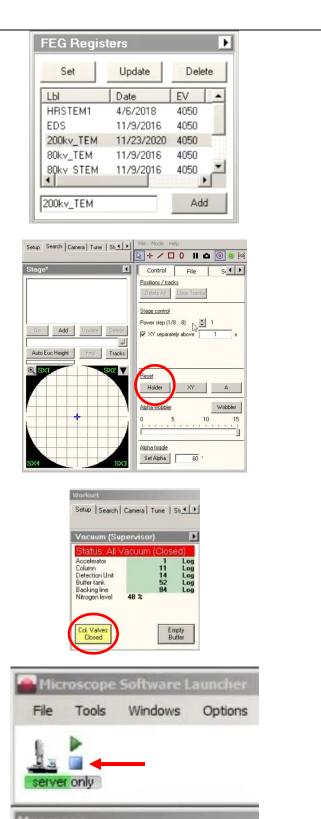
1-From the FEG Registers, click on the "200kv_TEM" and "Set".

2- Click on "Holder" in the search tab to reset the stage to zero coordinates.

3- Close the "Column Valves".

4- Stop the TEM programs by clicking the square/stop button on the TEM software launcher.

TIA software on the right monitor will ask you if you still need to save your data. Click "No to all" and click "Yes".







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22 Removing the Holder:

On the TEM screen click "Remove Sample" then wait for a few seconds.

Follow the following instructions to remove the holder from the TEM:

Video on how to remove the holder

youtu.be/qDFljfuem3I?si=XI 6OF tCyJqZiTPZ&t=84



AVAILABLE Pressure Readouts Accelerator 1 Log Column 24 Log **Detection Unit** 14 Log Nitrogen Level V_{ac} Status 35 % **Stage Position** V_{ci} 0.01° $2.69 \, \mu m$ α 0.08 μm β 0.00° 0.01 µm

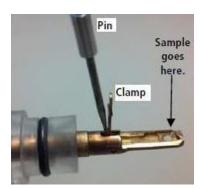
- 1- Pull the holder out, till you feel the resistance.
- 2- Simply rotate the holder (not pull) clock-wise for 120 deg, till it stops.
- 3- Put your fingers on the white panel, and with other hand gently pull the holder out.

23 Take the grid out:

Remove the clamp by the pin

Gently take the holder out of its plastic tube (only 2 mm), then rotate it to drop the grid on the filter paper on the stereoscope.

Close the clamp and place the holder in the desiccator.









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24	Disable the tool in badger.	Window	Equipment Actions	Reservation Action	
	o o	CNI	<u>E</u> nable		
			<u>D</u> isable		
		Columb	Shutdown		
			Report Problem		
		→ SMC	Make Comment		
		9− Elec	Qualify User		
			March March March March	S-TEM* (cg2929@	
		= 4	♠ ZEISS SEM		
			PIPS II*		
			A Light Zeiss Micro	oscope*	
			Dimple Grinder*	1 1€/C	
			- A SECOLO DE POSICIO DE CONTRACIO.		
			Plasma Cleaner		
		= 3	♣ Grinder-Polisher	*	
			A Diamond Saw*		
		1-8	A Microtome		
	Diago make sure to keep the				
25	Please make sure to keep the				
	table and working area clean and				
	organized.				





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HR TEM set-up

STANDARD TEM ALIGNMENT: Direct Alignments follow the steps necessary for Beam tilt pp X TEM alignment described above. Beam tilt pp Y Go to higher mag, e.g. SA 245KX. Beam shift Rotation center Click on "Comma Free Pivot Point Coma-free Alignment X X" in the Direct Alignments Coma-free Alignment Y Coma-free Pivot Point X window. Condense the beam Coma-free Pivot Point Y using intensity knob and use multifunction X & Y knobs to Done Auto help overlap two spot images. Press "Done". Repeat for Pivot Point Y. **TEM ALINGMENT CONT.**: Insert 2 Insert Ceta camera. Set the integration time, lift the screen (R1), press "Search". In the camera tab change stage piezo moves to Picometer steps. FFT: Press "Live FFT" in the 3 HDR Manual High Resolution CCD/TV Camera window. Go out of focus until you see rings. Stigmator Adjust objective stigmators using Condenser Objective Diffraction the multifunction knobs to round and concenter the rings. None Condenser 3



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COMA-FREE ALIGNMENT: Align **Direct Alignments** the beam with the optic axis by Gun Tilt clicking on "Coma-free Gun Shift Alignment X" Beam tilt ppX and use the Beam tilt pp Y multifunction x knob to minimize Beam shift shape change and movement of **Rotation** cente Coma-free Alignment X FFT rings (should be stable). Coma-free Alignment Y "Coma-free for Repeat Done Auto help Alignment Y". 5 **EUCENTRIC FOCUS:** Click "Eucentric focus" and use the Z height to get to optimized focus position at max ring size. Use the focus knob to get to max fine focus (one ring for crystalline material).



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Diffraction set up

aperture (e.g. 70) and finish your gun shift on a larger spot size (e.g.6). This will protect the ceta camera from over exposure. Focus the beam and press "Diffraction". Use the intensity knob to sharpen the diffraction (larger spot size will increase sharpness).



In the Direct Alignments window press "Diffraction alignment".

Use multifunction knobs to center the diffraction pattern.

When inserting the ceta camera put the search and acquire buttons on the lowest integration time and carefully monitor the histogram intensity chart to determine acceptable exposure.







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Dark-Field set up

1	DIFFRACTION ALIGNMENT: Follow diffraction alignment	
	steps described above. Camera	
	Tab: Select Dark Field.	
2	Click XY button.	Add Delete Delete all Chan. X Y Reset 0.0 1: 0.000° 0.000° None C XY C Conical Dynamic Circle 0.5 s
3	CENTER REFLECTED BEAM: Use multifunction knobs to move the desired reflected beam to center.	Stigmator Fine Coarsa Multifunction X Multifunction Y All 12 • R2
4	OBJECTIVE APERTURE: Insert small objective aperture (e.g. 20 μm) and select the desired diffraction spot. Press and unpress "dark field" to toggle between BF and DF image.	Condenser 1 2000 Adjust Condenser 2 150 Adjust Objective [none] Adjust Selected Area [none] Adjust



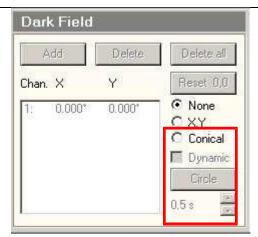
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5 Obtaining Hollow Cone Dark-Field:

In this mode you can select a circular region (like a donut) of diffraction the pattern obtaining the image.

In the diffraction mode, center the transmitted beam, then press "dark field" button on the nub and the "Conical" option on the screen. Insert an objective aperture and center it. Then use the X and Y nubs to tilt the beam and move and select a desired part of the diffraction pattern on the center. Now, press "Dynamic" and "Circle" button. Now, the diffraction pattern will rotate around the center and is obtaining a shape of a donut from your diffraction pattern. Now go to the imaging mode. You will see that some grains in respect to their diffraction condition turn bright and dark.







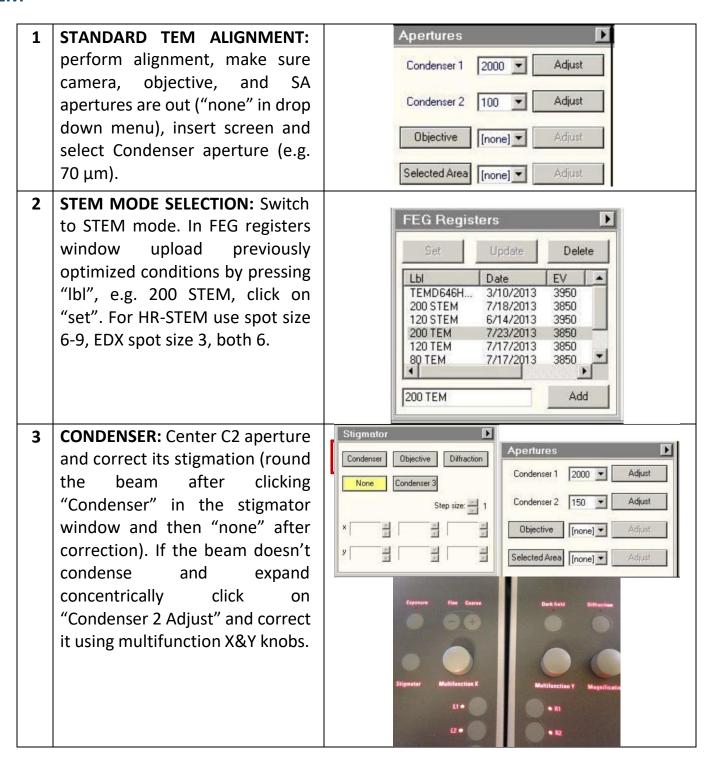


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STEM



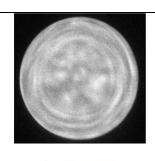


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CENTER BEAM and DETECTOR:

"Diffraction", Click on Ronchigram should be visible in the beam. Move the beam to the center of the HAADF detector (should look like the image to the Click on "Diffraction right). alignment" in the direct alignments window. Click on "HAADF Detector Area" button in the Flu Cam viewer and use the multifunction X&Y knobs move the beam to the center of the detector. Click "Done" to save.

Change the height (Z) of the sample on the nub to make the Ronchigram well aligned and condenser then the use stigmation to make the Ronchigram Astigmated (see images below).

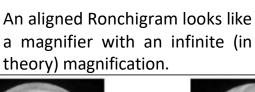


O O O CCC EIF

HAADF detector area

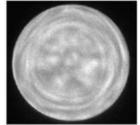












Well aligned and stigmated

ALIGNMENT: If 5 **DIRECT** perform direct necessary alignment as described above.





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LIVE IMAGE: press the Z-axis buttons to bring your sample into rough focus. Click "Search" in the STEM imaging window. A live image will appear in the TIA program. If image does not appear check camera length (should be around 220mm STEM Imaging (Expert) depending on the sample), 78.7 🖟 Rotation (*): STEM contrast, and brightness (Auto Enable LMscan 90< 1< 0 >1 >90 C/B). Fine focus using the Focus Dwell time [µs]: 8 knob. Scan frame: 512 x 512 Blank Pixel size: 73.0 pm Search Preview Acquire Scope Auto C/B Focus 7 **ACQUIRE** An IMAGE: Click STEM Imaging (Expert) "Acquire" in the STEM Imaging 78.7 🗾 Rotation (*): STEM window to obtain HAADF STEM Enable LMscan 90< 1< 0 >1 >90 image. Slow scan rate - higher Dwell time [µs]: 8 quality. Scan frame: 512 x 512 Blank Pixel size Preview Search Acquire Auto C/B Focus Scope **EXIT:** To exit STEM mode click on STEM Imaging (Expert) "STEM" in the STEM Imaging 78.7 🕶 Rotation (*): STEM window and the HAADF detector 90< 1< 0 >1 >90 will be auto retracted. Dwell time [µs]: 8 Scan frame: 512 x 512 Blank Pixel size: 73.0 pm Search Preview Acquire Auto C/B Focus Scope



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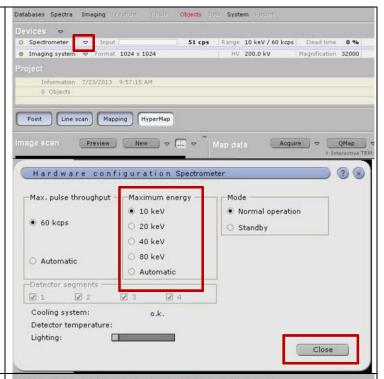
EDX Analysis

2	STEM ALIGNMENT: Perform STEM alignment and HAADF STEM imaging as described above. Press "Search", "Preview",	STEM Imaging (Expert)
	"Acquire", "Focus", "Scope", and "Auto C/B" in the STEM imaging window. They should be inactive when using the Esprit software. To ensure they are inactivated you can press the buttons, so they no longer appear yellow.	STEM Rotation (*): 78.7 Enable LMscan 90< 1< 0 >1 >90 Dwell time [µs]: 8 Scan frame: 512 x 512 Provel size: 73.0 pm Search Preview Acquire Focus Scope Auto C/B
3	DETECTOR ON: Optimum EDX conditions: smallest C1 aperture, 100 µm C2 aperture, Gun Lens 5, and Spot Size 3, giving a probe size of 1-2 nm. Go to the Super X EDX window and click on ▶to show the flap-out window. Under the Super X tab if the Pulse processor Status appears Standby, click on the "On" button to start the EDX detectors. For better resolution it should be kept on for about an hour before collecting X-ray data.	Super X EDX (User) 1
4	HYPERMAP: In the Bruker Esprit program choose the "objects" tab and click on "HyperMap" to activate the HyperMap workspace.	Databases Spectra Imaging Feature EBSD Objects DeVices O Spectrometer Input



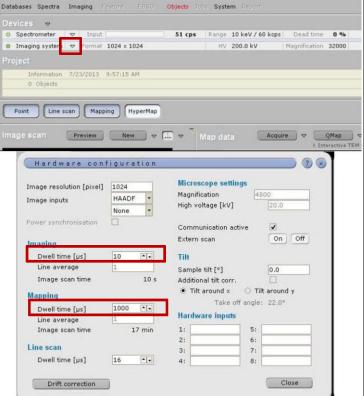
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the Hardware configuration spectrometer window by clicking on the dropdown arrow. Set the maximum energy at a desired value: 10 KeV is recommended for higher elemental resolution and 20 KeV is for better elemental coverage. Click on "Close" after you're done.



6 HARDWARE CONFIGURATION:

Open "Imaging system" (drop down arrow) to open the Hardware configuration window. Set Image resolution [pixel] to a desired value, e.g. 1024. Set for Dwell time **Imaging** (suggested value 20 μm), Mapping (10-50 µm for fast surveys, 1000 µm or more for trace and quantification), and Line scans.







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DRIFT CORRECTION: click on Hardware configuration "Drift correction" from the Image resolution [pixel] 1024 Magnification HAADE Image inputs Hardware configuration window High voltage [kV] Power synchronisation Communication active to open the Drift correction On Off Extern scan Imaging window and adjust the Dwell time [µs] 10 Tilt Line average Sample tilt [°] 0.0 correction interval and Dwell Image scan time Additional tilt corr. Tilt around x
 Tilt around y Mapping time for correction image. Close Take off angle: 22.0° Dwell time [µs] Hardware inputs both windows when you're done Line average Image scan time by clicking on "OK" and "Close". Line scan Dwell time [µs] Close Drift correction Image drift correction Image input Current input O HAADE O None Settings for Objects and LineScan Correction interval: 10 Dwell time: Current value O Special Value ↑ + µs OK Cancel SAVE: The measurement Devices parameters by clicking: under Spectro Measure method "Devices" as a measurement Imaging Load . . . setup that can be loaded (by Project Save ... clicking "Load") next time. **COLLECT STEM IMAGE:** Open the Image scan New Image scan set up windows. Single Continuously Image name and number will appear on image. Click on Image name: "Preview" to view the live STEM Image number: 39 image. Use the joystick to move your sample to an area of interest, adjust brightness and

contrast and click "OK" to close.



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Click "New" to acquire a STEM Preview Map data with predefined image conditions Hardware (in Image configuration and imaging), then Add to project click on export/import button Save . . . in image scan window to open Print . . . Twain export Image menu and save the image. **11** | **DRIFT CORRECTION** (if needed): Preview New

| Instruction | Preview | Previe can be activated by clicking on: +*+ Add to project Print . . . **12** | **MAP SET UP:** In the map setup Map size QMap resolution X 768 window select desired map size O Full 0 1/2 0 1/8 Fix Y 768 • 1/4 (mapping area of interest in Variable Measure time: 13 min pixels as showed by green frame Image filter in Image scan window). Please None None Smooth Average note that there isn't enough 3 --Sharpen Smooth memory for the full Automatic (1024x1024). Image filter is for Preview New

Rew digital image processing. The QMap resolution allows converting up to 8 pixels of spectrum data to 1 pixel in the final EDX maps during quantified mapping to shorten measurement time. Quantified mapping can be perform using HyperMap workspace post data collection. Map filter changes elemental map digitally.



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MEASUREMENT SET UP: in Map Interactive TEM (n > Interactive TEM data open the Acquire window (drop down arrow) to adjust measurement set up. If "Manual" Acquire | 🗸 QMap Quantify | PB-ZAF is checked, the measurement will Measuring time (Fast map) require user's second click on the Manual Acquire button to stop. Measuring time [s] Measuring time [s] or Cycles is Switch off microscope checked, the measurement will Options Options be automatically stopped when ☐ Interlaced measurement HV off reaching the set values. Never ✓ Use 1. microscope image only Blank beam check HV off in the Switch off Image average [s] Close column valves microscope drop down menu. Map name Map data 24 Map number: Automatic numbering Object name test OK LOAD METHOD: Open the Load 14 QMap ▽ ▶ Interactive TEM (n ▶ Interactive TEM method window (from either the QMap or Qunatify). Linemarker TEM.mtd is the recommended Load method selection for beginners. edx\Methods\ Volumes → EE - X Pa Automatic Oxides.mtd
Automatic PB-ZAF.mtd 2 Automatic Standards.mtd
Automatic TEM.mtd Interactive Oxides.mtd
Interactive PB-ZAF.mtd
Interactive Standards.mtd Interactive TEM acpc (mod.mtd
Interactive TEM.mtd
Linemarker Halt P8-ZAF.mtd Linemarker Halt TEM.mtd
Linemarker PB-ZAF.mtd
Linemarker TEM.mtd -0 - D: ₩ E: 3 F: File name Information *2: Open Quantify methods Cancel **15** START EDX MAPPING: Click on QMap ▽ "Acquire" to start the mapping. Click again and the Acquire turns





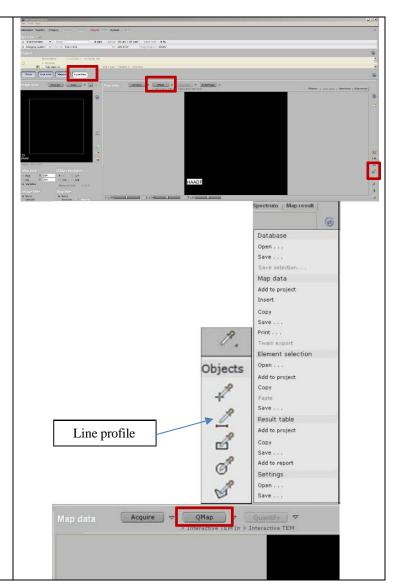
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to "Stop" and the mapping will stop after finishing the current frame. Click on "Stop" to stop immediately. 16 | SELECT ELEMENTS: Click on | to select elements to monitor. Overview Table of elements Finder The "Auto" will eliminate option He F1 F2 F3 F4 F5 F6 F7 F8 that should not be detected. Li Be B C N O F Ne I1 I2 I3 I4 I5 I6 I7 I8 Na Mg K Ca Sc Ti V Cr Mn Fe Co Ni Cu Zn Ga Ge As Se Br Kr Rb Sr Y Zr Nb Mo Tc Ru Rh Pd Ag Cd In Sn Sb Te I Xe Cs Ba La Hf Ta W Re Os Ir Pt Au Hg Tl Pb Bi Po At Rn Fr Ra Ac Ce Pr Nd Pm Sm Eu Gd Tb Dy Ho Er Tm Yb Lu Th Pa U Np Pu AmCm Bk Cf Es Fm Md No Lr C Clear all Auto New element **17 IMAGE OVERLAY:** In the Element images window, click on the check box below each image to 1.00 Pt-LA 1.00 1.00 1.00 O-KA Co-KA overlay/no-overlay the image in the Map data window. Click on the color button to change color for an image. **18** | **SAVE DATA:** Once the acquisition Export/ done click on the import export/import button in the Map result tab of the Map data window to Save the acquisition Super X EDX (User) SuperX Settings result under Database. If this is the last acquisition turn off the On Status: Standby super X detectors by changing Shutter Open Status: Closing their status to: "Standby" (the Temperature control button "On" should be available -cps -% Status: Ready Live time [s]: 600 but not yellow at this status). Acquire Defrost



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pipette button to choose the marker you wish to use and use it to select an area on the map. Analyze the elements as described above and obtain composition by clicking on "Quantify" (composition in % will be described at the bottom of the Map data window). Click on "QMap" for concentration maps.







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o SAVING OPTIONs: Element maps can be Saved under Map data to image files, e.g. .bmp format. Various data and processing results can also be added to project and save all together as one Project package.

