

# Titan Operation Manual



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# Put Rod to first position and open the vacuum overview page

The screenshot displays the TEM User Interface with several control panels on the left and a large central vacuum overview image. The panels include:

- Status: Col. Valves**: A table showing the status of various valves.
- FEG Control (Expert)**: Controls for the Field Emission Gun, including gun lens, extractor, and emission current.
- High Tension**: Controls for the high voltage, currently set to 80 kV.
- FEG Registers**: A table for managing FEG registers.
- MF X: Beam shift X MF Y: Beam shift Y**: Controls for beam shifting.

The central image shows a detailed view of the vacuum chamber components. A menu is open in the bottom right corner, listing various system functions. The 'Vacuum Overview' option is highlighted with a red circle.

**Status: Col. Valves**

Component	Status	Log
Gun	1	Log
Liner	17	Log
Octagon	9	Log
Projection	30	Log
Buffer tank	47	Log
Cold trap LN2	40 %	

**FEG Control (Expert)**

Operate: Gun lens: 3 Fine  
Extractor: 3700  
Extraction (Standard mode): 3750 V  
FEG Emission: 185  $\mu$ A  
Status: 0. 200.

**High Tension**

High Tension: 80 kV  
80 kV  
Free high tension: ☐

**FEG Registers**

Lbl	EV	GL	Mode	Spot
80kVTEM	3850	3.2	Par SA	3
80kVST...	3850	3.2	Probe...	5

**MF X: Beam shift X MF Y: Beam shift Y**

L1:	ScreenDim	R1:	Reset Defocus
L1:	Normalize all <td>R3:</td> <td>Spotsize +</td>	R3:	Spotsize +
L3:	Spotsize -	L1:	Normalize all

**Error on the LensMiniCondenser board; please consult the logging**

**TITAN**

Parameter	Value	Parameter	Value
80000 x STEM		HT:	80 kV
Screen current:	0.000 nA	Spot size:	5
		Focus step:	5
		C2:	17.618 %
		Defoc.:	-42.13 nm
		C3:	20.376 %
		A:	0.00 deg
		X:	0.00 $\mu$ m
		B:	0.00 deg
		Y:	0.00 $\mu$ m
		Obj Lens:	77.5327 %
		Z:	0.00 $\mu$ m
		MC Lens:	-97.886 %
		Conv.:	9.85 mrad

**Vacuum Overview**

- None -
- Alignments
- Apertures
- Application Preferences
- Application Selection
- Beam Settings
- CCD/TV Camera
- Control Pads
- Dark Field
- Direct Alignments
- Experiments
- Filter
- Filter Tuning (User)
- Image Settings
- Low Dose
- Magnification Calibration
- Normalizations
- PEELS
- Stage
- STEM Detector (Service)
- STEM Detector (User)
- STEM Imaging (Expert)
- Stigmator
- Super X EDX (Service)
- Super X EDX (User)
- System Status
- Vacuum Overview**
- Workspace Layout

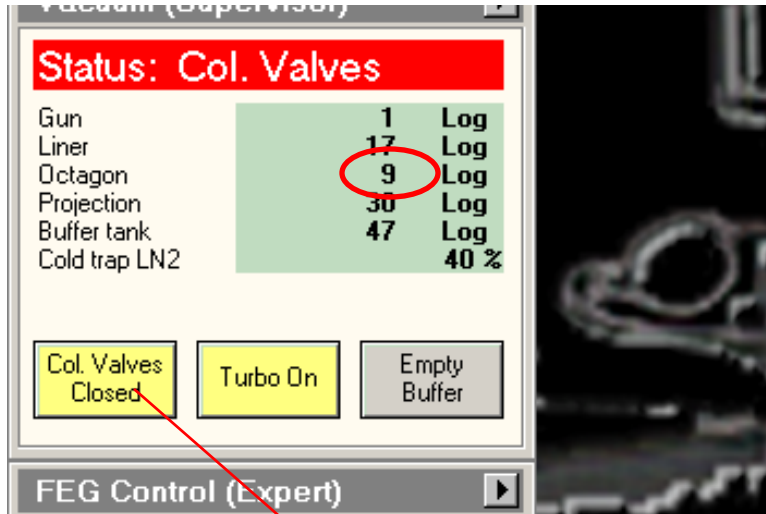
**Start** **TEM User Interface** **STEM mode 1st step.bm...** **11:57 AM**

delay!



When time reaches 0,  
insert rod by turning  
Counter Clockwise and  
letting it gently into  
the Titan.

# After inserting Octagon Pressure should be less than 25.

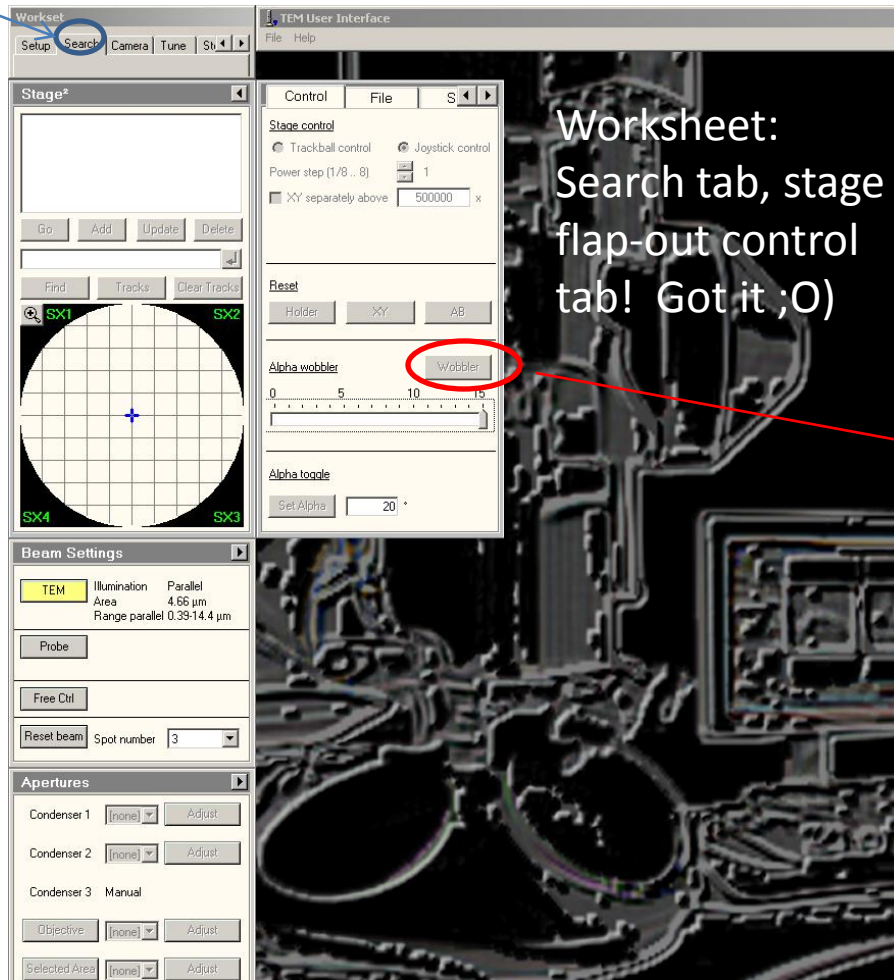


Now click on Col Valves  
Closed to turn on Beam!



# Set Eucentric Height

Search



Activate  
wobber and  
use Z controls  
on MUI to  
minimize  
motion  
induced by  
alpha wobble

Adjust Z  
height



Right MUI,  
manual user  
interface

# Now that Eucentric Height is set we are ready to do basic alignments

Tune worksheet click on Gun shift in Direct Alignments

The screenshot shows the TEM User Interface with the 'Tune' worksheet selected. The 'Direct Alignments' section has 'Gun Shift' selected. The 'Beam Settings' section shows 'TEM' illumination. The 'Image Settings' section shows 'Reset shift' and 'Scherzer' buttons. The bottom status bar displays various parameters including SA 8500 x TEM, HT: 80 kV, Spot size: 3, and Screen current: 0.000 nA.

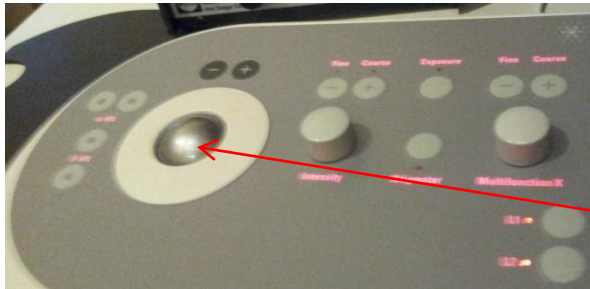
SA 8500 x  
TEM

HT: 80 kV Spot size: 3  
Focus step: 5 C2: 50.300 %  
Defoc.: 109.93  $\mu$ m C3: 35.822 %

Screen current: 0.000 nA

A: 0.00 deg X: 0.00  $\mu$ m  
B: 0.00 deg Y: 0.00  $\mu$ m  
Obj Lens: 77.2689 % Z: 0.00  $\mu$ m  
MC Lens: 97.886 % Conv.:

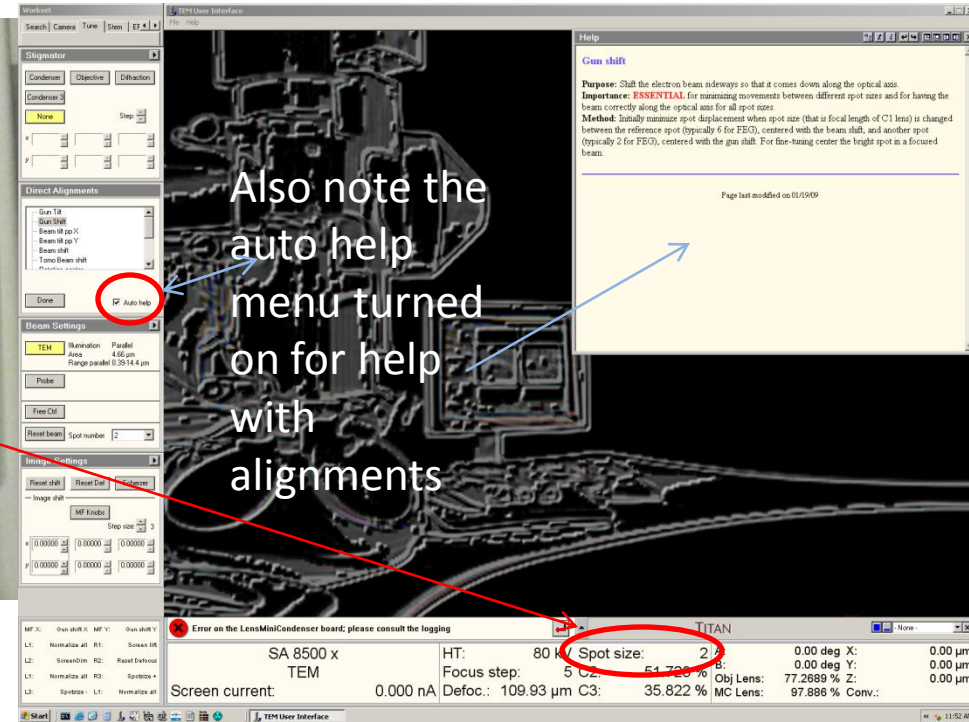
# Beam and Gun Shift align\*, we flip between spot 6 and spot 2



\*Gun Shift alignment is probably the most important as it sets the beam to go through center of apertures at top of column.



# Gun Shift at spot 2

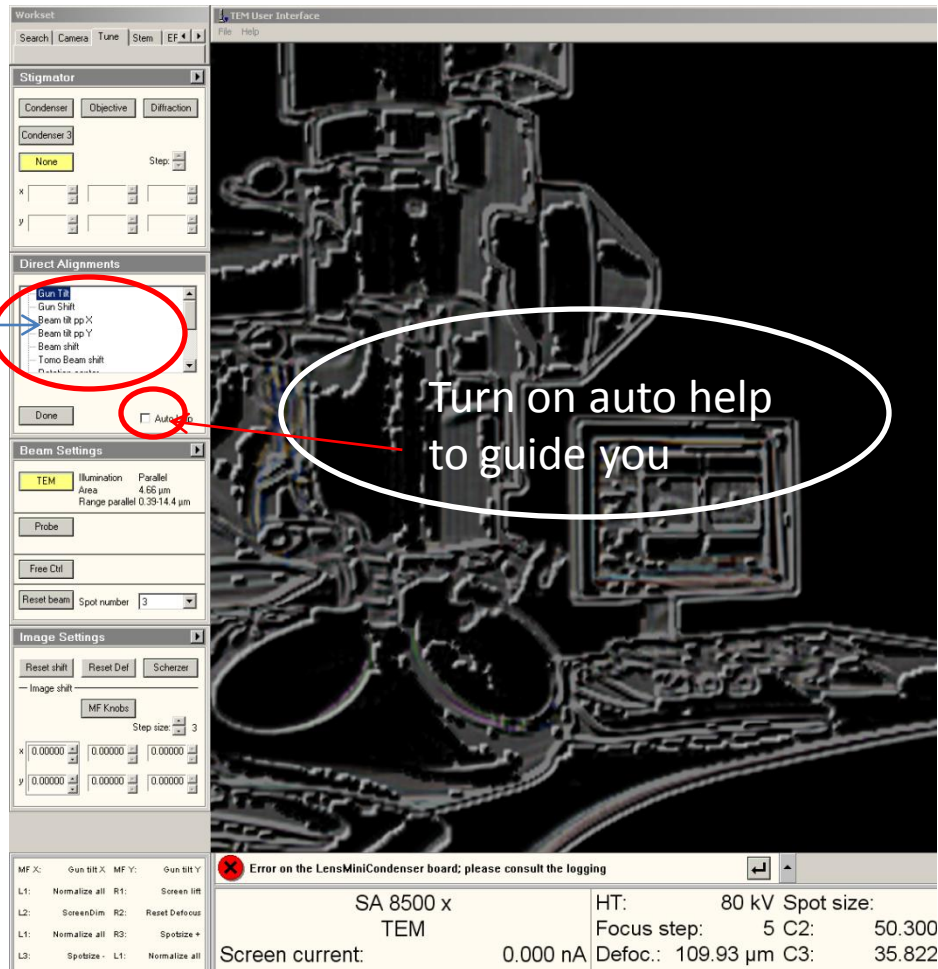


Computer auto assigns gun shift to Multifunction x and Multifunction y. Use MFx and Mfy to center beam at spot 2!

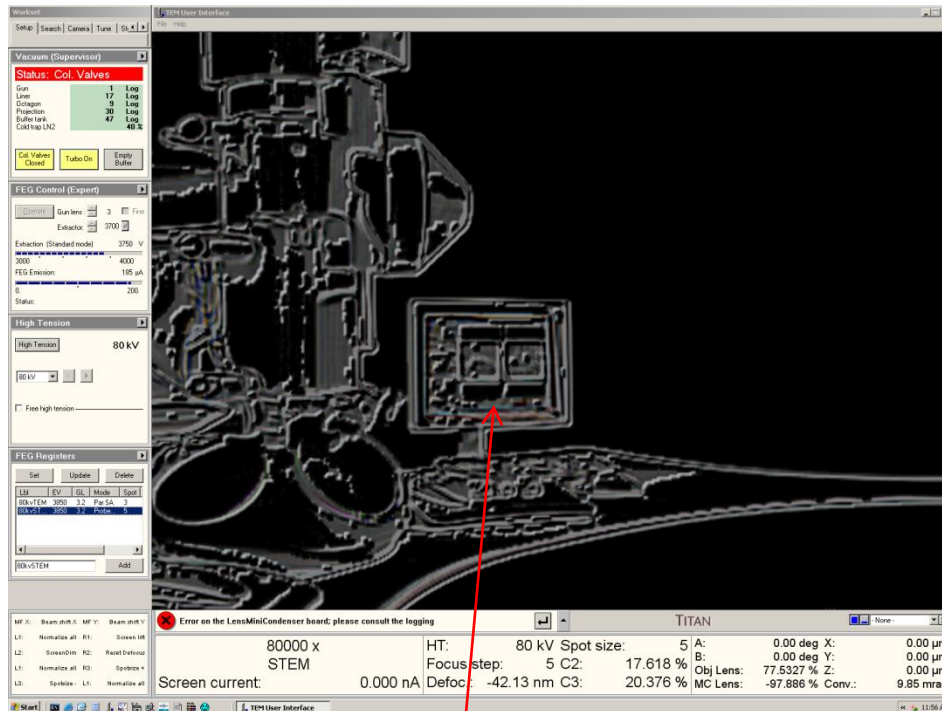


# Now that we have the spot 2,6 (gun shift and beam shift) TEM alignments the others go quickly.

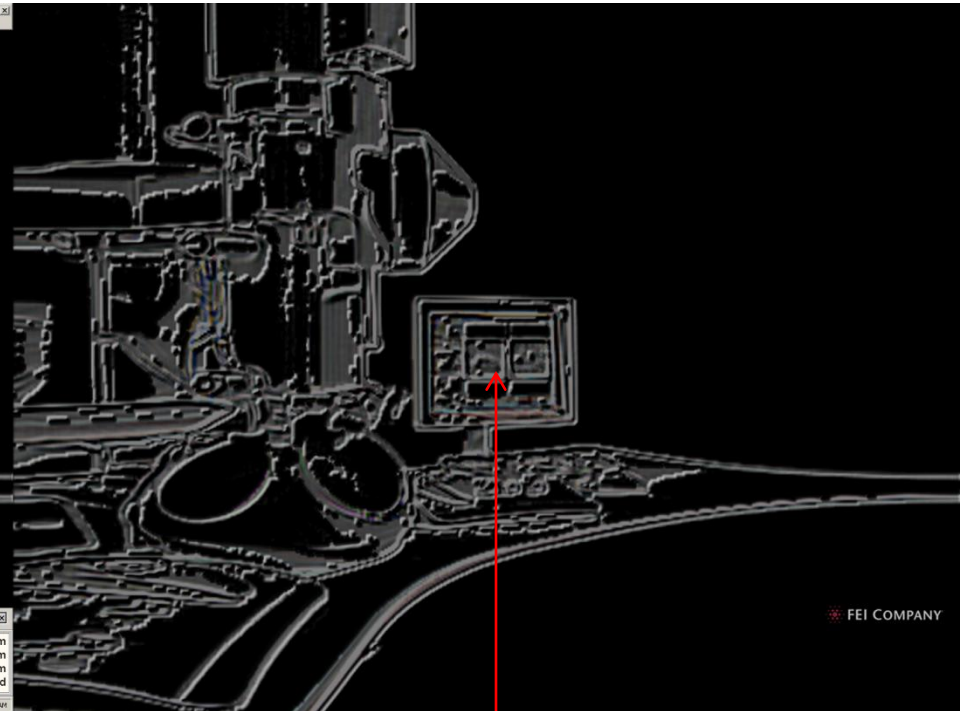
Step through all the alignments



# STEM (Scanning Transmission Electron Microscopy) Overview



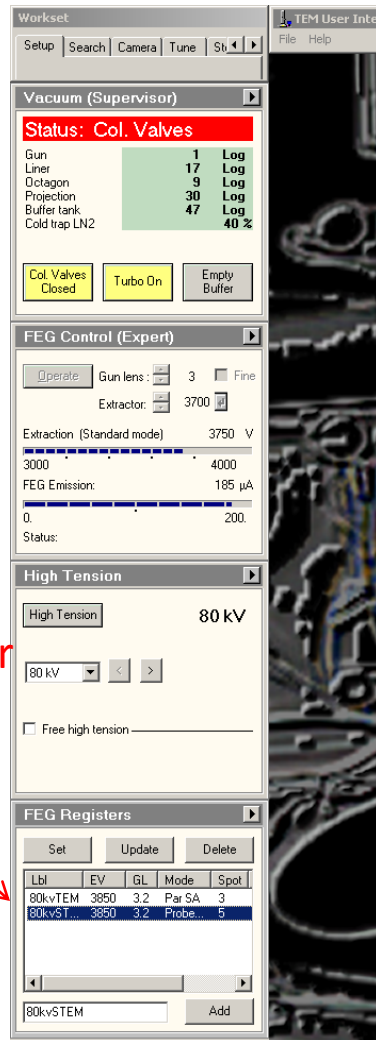
STEM images using TIA software will go here!



Chemical maps using the four Bruker EDS detectors will pop up on the second monitor over here!

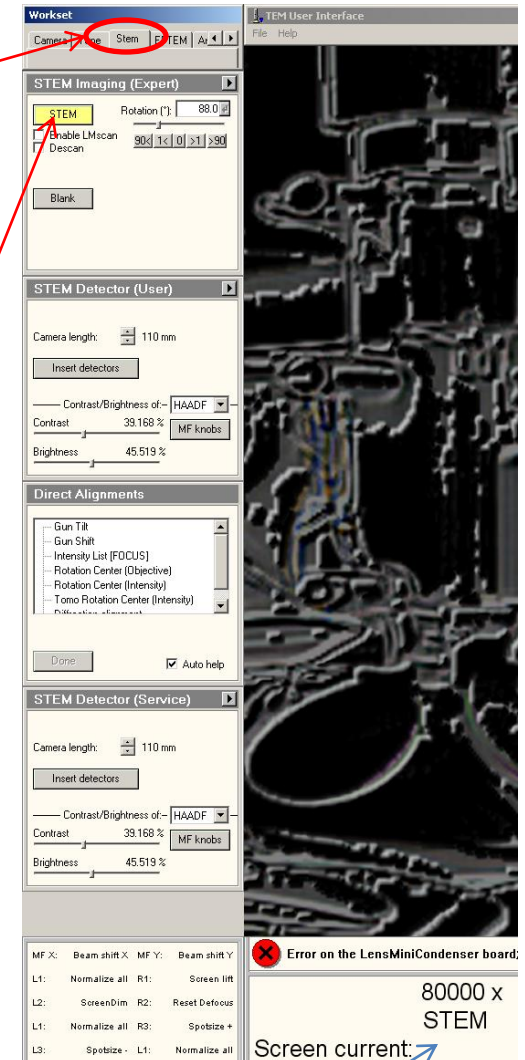
Two monitors, same CPU and hard drive!

# TEM to STEM steps 1, 2, 3



2) Go to STEM worksheet

3) Click on STEM



Mode at bottom of screen now says STEM!



# Stem Step 4 Launch TIA

**Vacuum (Supervisor)**

Status: Col. Valves

Gun	1	Log
Line	17	Log
Octagon	9	Log
Projection	30	Log
Buffer tank	47	Log
Cold trap LN2	40 %	

Col. Valves Closed Turbo On Empty Buffer

**FEG Control (Expert)**

Gun lens: 3 Fine  
Extractor: 3700  
Extraction (Standard mode): 3750 V  
FEG Emission: 185  $\mu$ A  
Status: 0 200

**High Tension**

High Tension: 80 kV  
80 kV  
Free high tension

**FEG Registers**

Set	Update	Delete		
Lbl	EV	GL	Mode	Spot
80kVTEM	3850	3.2	Par SA	3
80kVST...	3850	3.2	Probe...	5

80kVSTEM Add

**Filter Control**

GIF Tridien Control

Primary Energy	80.0	keV
Energy Shift	0.0	eV
Adjust	0.0	eV
Spectrum Offset	0.0	eV
Slit In Width	1.0	eV

Mode: Imaging  
Reduction: None  
Aperture: 5.0 mm

**Adjustments**

ACCCompA	5.12
ACCCompB	1.54
FOCUS X	48.3
FOCUS Y	11.12
SX	-72.81
SY	-2.22
ACHROMATICITY	32.14
SQUARENESS	61.69
IMAGE SHIFT X	3.35
IMAGE SHIFT Y	6.95

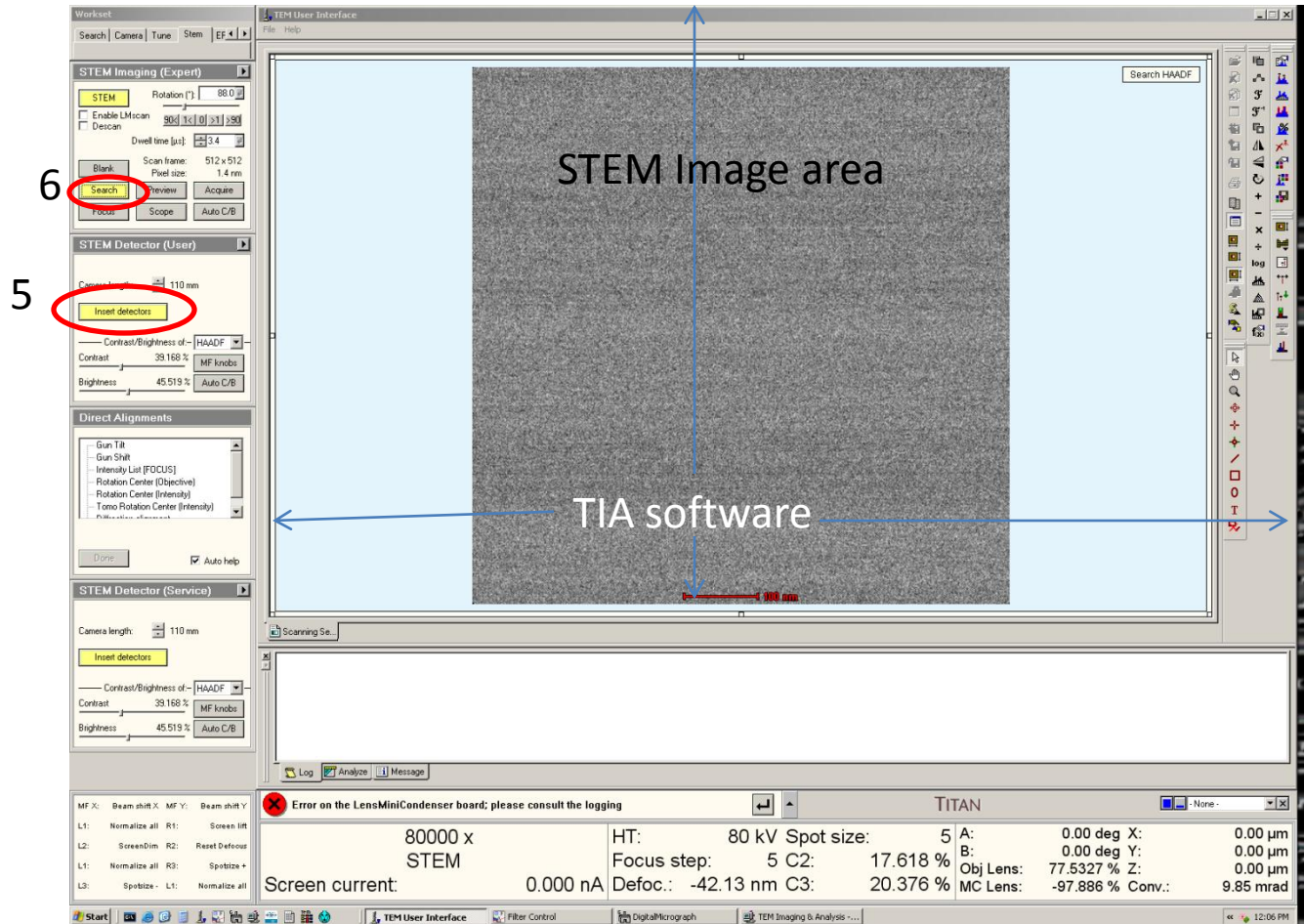
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TOOLS FOR NANOTECH  
Software for Winning Performance  
Copyright © 1994-2010 FEI Company  
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Initializing cameras  
FEI Software  
TEM Imaging & Analysis  
version 4.2 build 813

**Error on the LensMiniCondenser board: please consult the logging**

80000 x STEM  
Screen current: 0.000 nA  
HT: 80 kV Spot size: 5  
Focus step: 5 C2: 17.618 %  
Defoc.: -42.13 nm C3: 20.376 %  
A: 0.00 deg X: 0.00  $\mu$ m  
B: 0.00 deg Y: 0.00  $\mu$ m  
Obj Lens: 77.5327 % Z: 0.00  $\mu$ m  
MC Lens: -97.886 % Conv.: 9.85 mrad

Launch TIA software

# STEM Step 5 Insert detectors, 6 click search



# Now to Collect EDS Map in STEM

Workset

Setup Search Camera Tune St

Vacuum (Supervisor)

Status: Col. Valves

Gun	1	Log
Liner	17	Log
Octagon	9	Log
Projection	30	Log
Buffer tank	47	Log
Cold trap LN2	40 %	

Col. Valves Closed Turbo On Empty Buffer

FEG Control (Expert)

Operate Gun lens: 3 Fine

Extractor: 3700

Extraction (Standard mode) 3750 V

3000 4000

FEG Emission: 185  $\mu$ A

0 200

Status:

High Tension

High Tension 80 kV

80 kV < >

☐ Free high tension

FEG Registers

Set Update Delete

Lbl	EV	GL	Mode	Spot
80kVTEM	3850	3.2	Par SA	3
80kVST...	3850	3.2	Probe...	5

80kVTEM Add

Log Analyze Message

Scanning Se...

Launch Super X, turn on pulse processors and shutter

None - Alignments Apertures Application Preferences Application Selection Beam Settings CCD/TV Camera Control Pads Dark Field Direct Alignments Experiments Filter Filter Tuning (User) Image Settings Low Dose Magnification Calibration Normalizations PEELS Stage STEM Detector (Service) STEM Detector (User) STEM Imaging (Expert) Stigmator Super X EDX (Service) Super X EDX (User) System Needs Vacuum Overview Workspace Layout

Error on the LensMiniCondenser board; please consult the logging

TITAN

SA 8500 x TEM

Screen current: 0.000 nA

HT: 80 kV Spot size: 2

Focus step: 5 C2: 57.900 %

Defoc.: 109.93  $\mu$ m C3: 33.628 %

A: 0.00 deg X: 0.00  $\mu$ m

B: 0.00 deg Y: 0.00  $\mu$ m

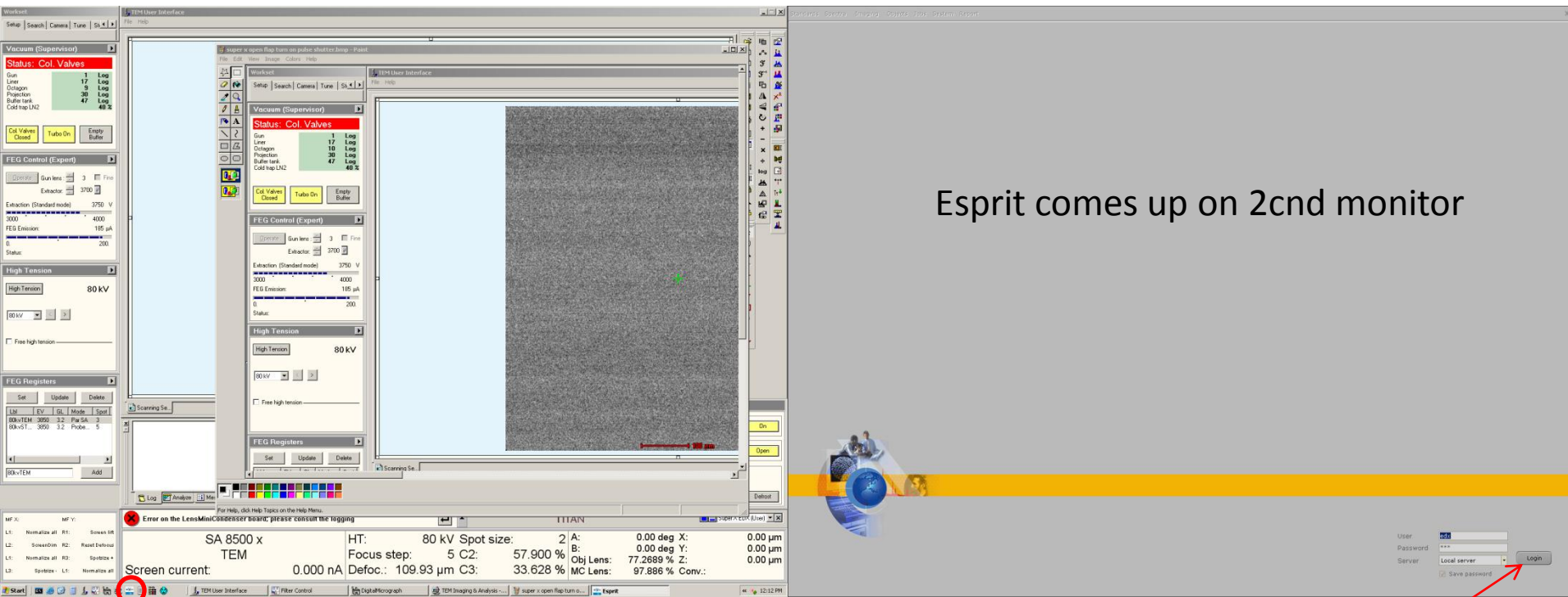
Obj Lens: 77.2689 % Z: 0.00  $\mu$ m

MC Lens: 97.886 % Conv.:

Start TEM User Interface Filter Control DigitalMicrograph TEM Imaging & Analysis - ... untitled - Paint 12:09 PM



# Now Launch Esprit, Bruker Software to Collect EDS map.



Launch Esprit

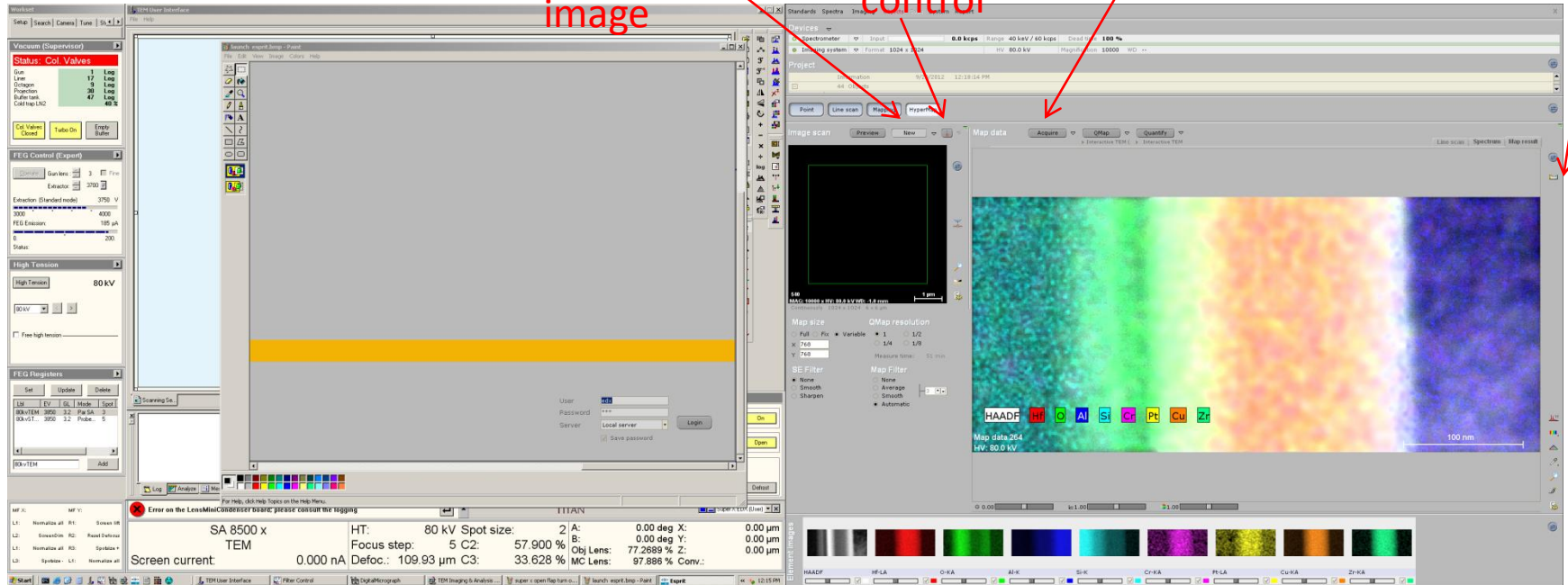
# To Collect Map in Esprit

1) Collect new STEM image

## 2) Hit drift control

#### 4) Click Acquire

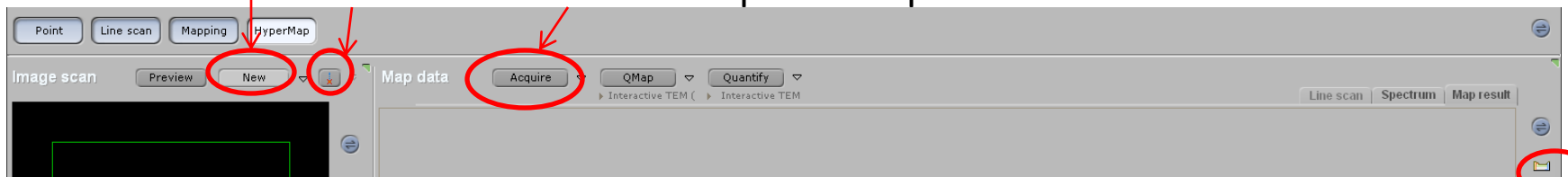
### 3) Select some elements in periodic table



## New STEM

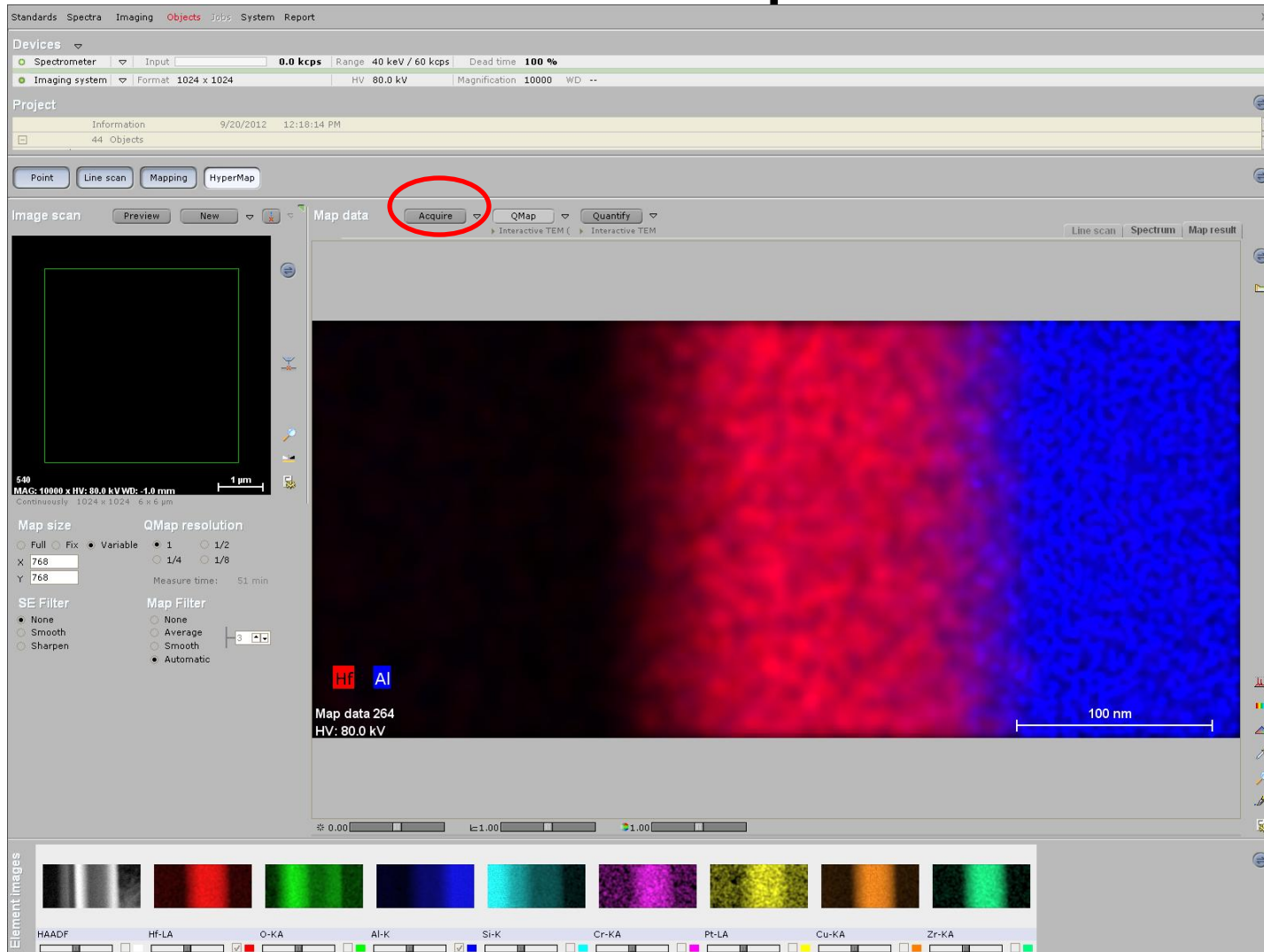
## Drift

## Start and Stop EDS Map



# Periodic Table

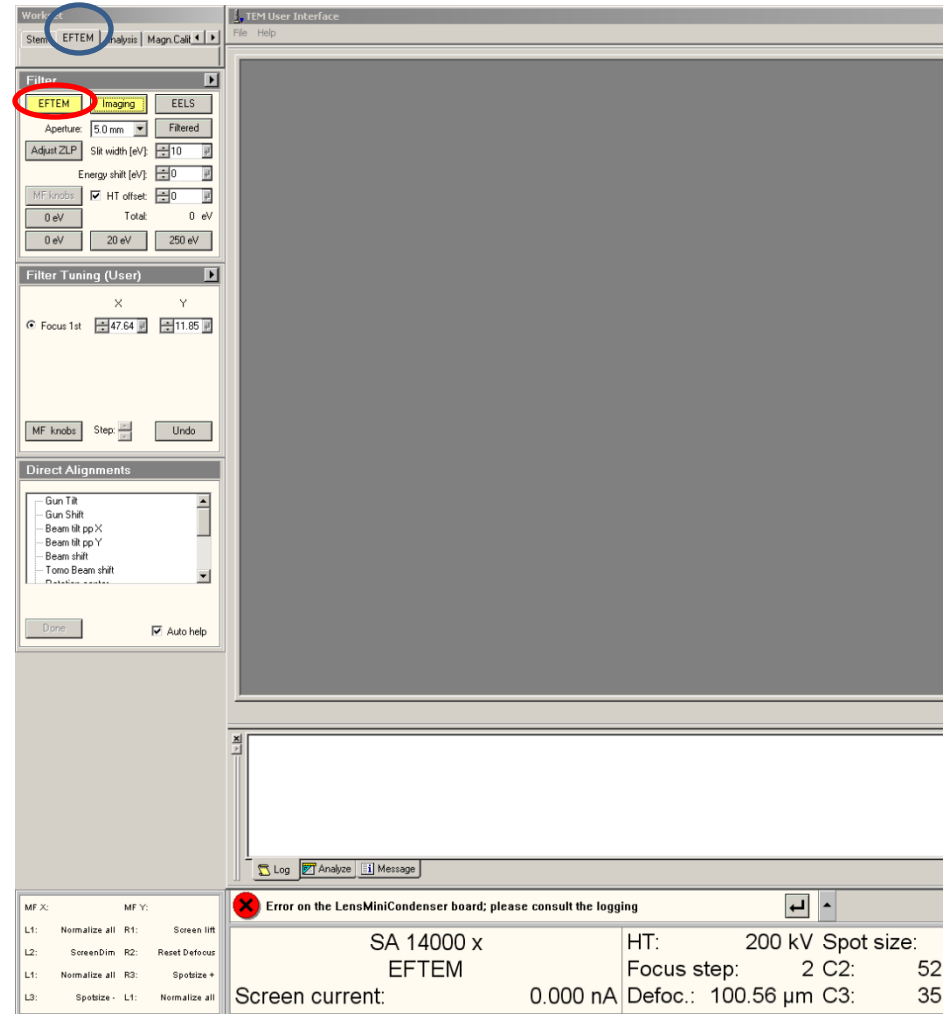
# Now mapping. Click Acquire again to stop





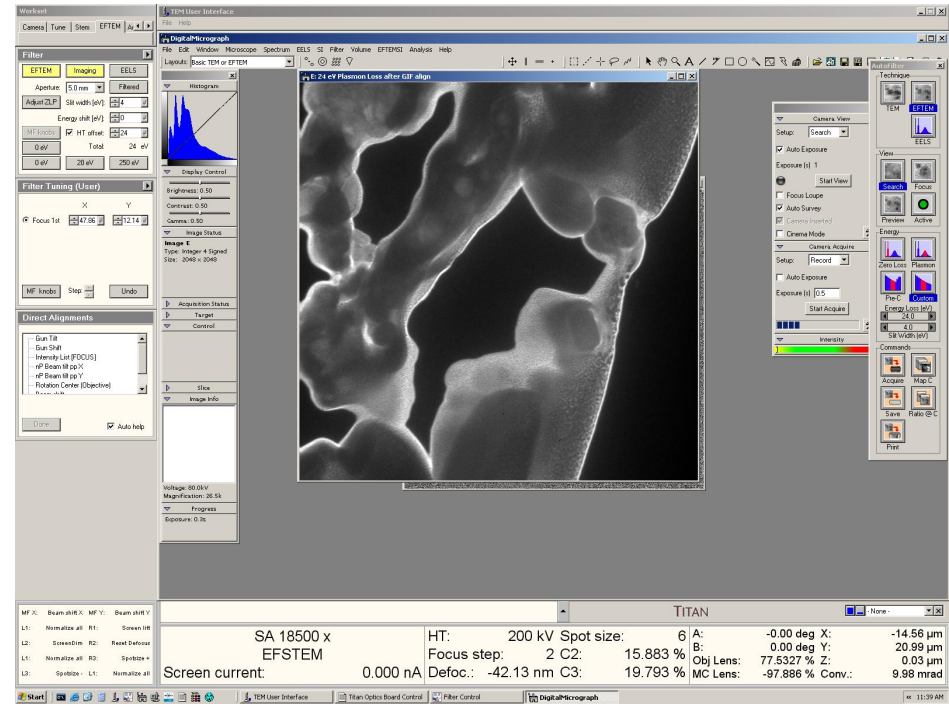
# EFTEM: Go to EFTEM worksheet

- 1) Hit EFTEM button, this will raise phosphor screen and set projector lens strength for lower camera, Energy Filtered CCD – EF CCD.
- 2) Start digital micrograph



# EFTEM Steps

- Select EFTEM basic layout in DM
- Switch to EFTEM mode in Titan control
- Raise screen
- Select TEM in filter
- Hit search and active
- Now adjust mag and focus for EF – CCD with sample.
- Move to a hole, away from sample
- Hit tune GIF, you may have to adjust brightness via dialog, hit enter when done adjusting
- After tune GIF is done move back to your sample.
- Collect Zero loss, make sure focus is good.
- Now begin mapping



# EFTEM basic layout

Workset

Camera Tune Stem EFTEM Ar

Filter

EFTEM Imaging EELS

Aperture: 5.0 mm Filtered

Adjust ZLP Slit width [eV]: 4

Energy shift [eV]: 0

MF knobs: ☒ HT offset: 24

0 eV Total: 24 eV

0 eV 20 eV 250 eV

Filter Tuning (User)

X Y

Focus 1st 47.86 12.14

MF knobs Step: Undo

Direct Alignments

Gun Tilt  
Gun Shift  
Intensity List (FOCUS)  
nP Beam tilt pp X  
nP Beam tilt pp Y  
Rotation Center (Objective)

Done ☒ Auto help

TEM User Interface

File Edit Window Microscope Spectrum EELS SI Filter Volume EFTEM SI Analysis Help

Layers: Basic TEM or EFTEM

Histogram

Display Control

Brightness: 0.50  
Contrast: 0.50  
Gamma: 0.50

Image Status

Image E  
Type: Integer 4 Signed  
Size: 2048 x 2048

Acquisition Status

Target  
Control

Slice

Image Info

Voltage: 80.0 kV  
Magnification: 26.5k

Progress

Exposure: 0.3s

E: 24 eV Plasmon Loss after GIF align

AutoFilter

Technique

TEM EFTEM EELS

View

Search Focus

Preview Active

Energy

Zero Loss Plasmon

Pre-C Custom

Energy Loss [eV]  
24.0

Slit Width [eV]  
4.0

Intensity

Commands

Acquire Map C  
Save Ratio @ C  
Print

SA 18500 x  
EFSTEM  
Screen current: 0.000 nA

HT: 200 kV Spot size: 6  
Focus step: 2 C2: 15.883 %  
Defoc.: -42.13 nm C3: 19.793 %

A: -0.00 deg X: -14.56  $\mu$ m  
B: 0.00 deg Y: 20.99  $\mu$ m  
Obj Lens: 77.5327 % Z: 0.03  $\mu$ m  
MC Lens: -97.886 % Conv.: 9.98 mrad

TITAN

MF X: Beam shift X MF Y: Beam shift Y  
L1: Normalize all R1: Screen lift  
L2: ScreenDim R2: Reset Defocus  
L1: Normalize all R3: Spotsize +  
L3: Spotsize - L1: Normalize all

Start TEM User Interface Titan Optics Board Control Filter Control DigitalMicrograph 11:39 AM

This is a custom view, 24 eV loss, of Lanthanum Oxide particle aggregate



# EFTEM Analysis Layout Digital Micrograph

**Filter**

EFTEM Imaging EELS

Aperture: 5.0 nm Filtered

Adjust ZLP Slit width [eV]: 10

Energy shift [eV]: 0

MF knobs ☒ HT offset: 0

0 eV Total: 0 eV

0 eV 20 eV 250 eV

**Filter Tuning (User)**

X Y

Focus 1st 47.64 11.85

MF knobs Step Undo

**Direct Alignments**

- Gun Tilt
- Gun Shift
- Intensity List [FOCUS]
- Rotation Center (Objective)
- Rotation Center (Intensity)
- Tomo Rotation Center (Intensity)

Done ☒ Auto help

**TEM User Interface**

File Help

**DigitalMicrograph**

File Edit Display Process Analysis Window Camera Microscope Spectrum EELS SI Filter Volume EFTEM51 Help

Layouts: EFTEM Analysis

**Histogram**

Brightness: 0.50

Contrast: 0.50

Gamma: 0.50

**Display Control**

**Image Status**

Image E

Type: Integer 2 Signed

Size: 256 x 255

**Acquisition Status**

**Target**

Page

E: Image

**Control**

**Slice**

**Image Info**

Voltage: N/A

Magnification: N/A

**Progress**

5000 x STEM

Screen current: 0.000 nA

**Titanium Map**

**Lanthanum Map**

**Carbon Map**

200 nm

**Color Mix**

Display Align

Component Images:

Color:

Brightness: 0.5

Contrast: 0.5

Select Images...

**Image List**

Select component images:

- E: Carbon map 4 no hw binning
- D: Lanthanum map 4 no hw binning
- C: Titanium map 4 no hw binning

OK Cancel

**AutoFilter**

Technique

TEM EFTEM EELS

View

Search Focus

Preview Idle

Energy

Zero Loss Plasmon

Pre-C Custom

Commands

Acquire Align ZLP

Save Tune GIF

Print

nsMiniCondenser board; please consult the logging

**TITAN**

HT: 200 kV Spot size: 6

Focus step: 2 C2: 15.883 %

Defoc.: -42.13 nm C3: 19.793 %

A: -0.00 deg X: -14.44 μm

B: 0.00 deg Y: 20.94 μm

Obj Lens: 77.5327 % Z: 0.03 μm

MC Lens: -97.886 % Conv.: 9.98 mrad

# EFTEM Analysis II, Color Map and Changing Colors

Workset

EFTEM User Interface

Camera Tune Stem EFTEM

Filter

EFTEM Imaging

Aperture: 5.0 mm

Adjust ZLP Slit width [eV]:

Energy shift [eV]:

MF knobs HT offset:

0 eV Total

0 eV 20 eV

Filter Tuning (User)

X

Focus 1st 47.64

MF knobs Step

Direct Alignments

Gun Tilt

Gun Shift

Intensity List [FOCUS]

Rotation Center (Objective)

Rotation Center (Intensity)

Tomo Rotation Center (Intensity)

Done

For Help, click

DigitalMicrograph

File Edit Display Process Analysis Window Camera Microscope Spectrum EELS SI Filter Volume EFTEM Help

Layouts: EFTEM Analysis

Histogram

Display Control

Image Status

Image A

Type: RGB

Size: 256 x 255

Acquisition Status

Target

Page

A: Image

Control

Slice

Image Info

Voltage: N/A

Magnification: N/A

Progress

5000 x STEM

Screen current: 0.000 nA

HT: 200 kV Spot size: 6

Focus step: 2 C2: 15.883 %

Defoc.: -42.13 nm C3: 19.793 %

A: -0.00 deg X: -14.44 µm

B: 0.00 deg Y: 20.94 µm

Obj Lens: 77.5327 % Z: 0.03 µm

MC Lens: -97.886 % Conv.: 9.98 mrad

Color Overlay

Color Mix

Display Align

Component Images:

E: Carbon map 4 no hw

D: Lanthanum map 4 no hw

C: Titanium map 4 no hw

Color: [Green]

Brightness: 0.5

Contrast: 0.5

Select Images... Display

Lanthanum in Green you can change it

Technique

TEM EFTEM EELS

View

Search Focus

Preview Idle

Energy

Zero Loss Plasmon

Pre-C Custom

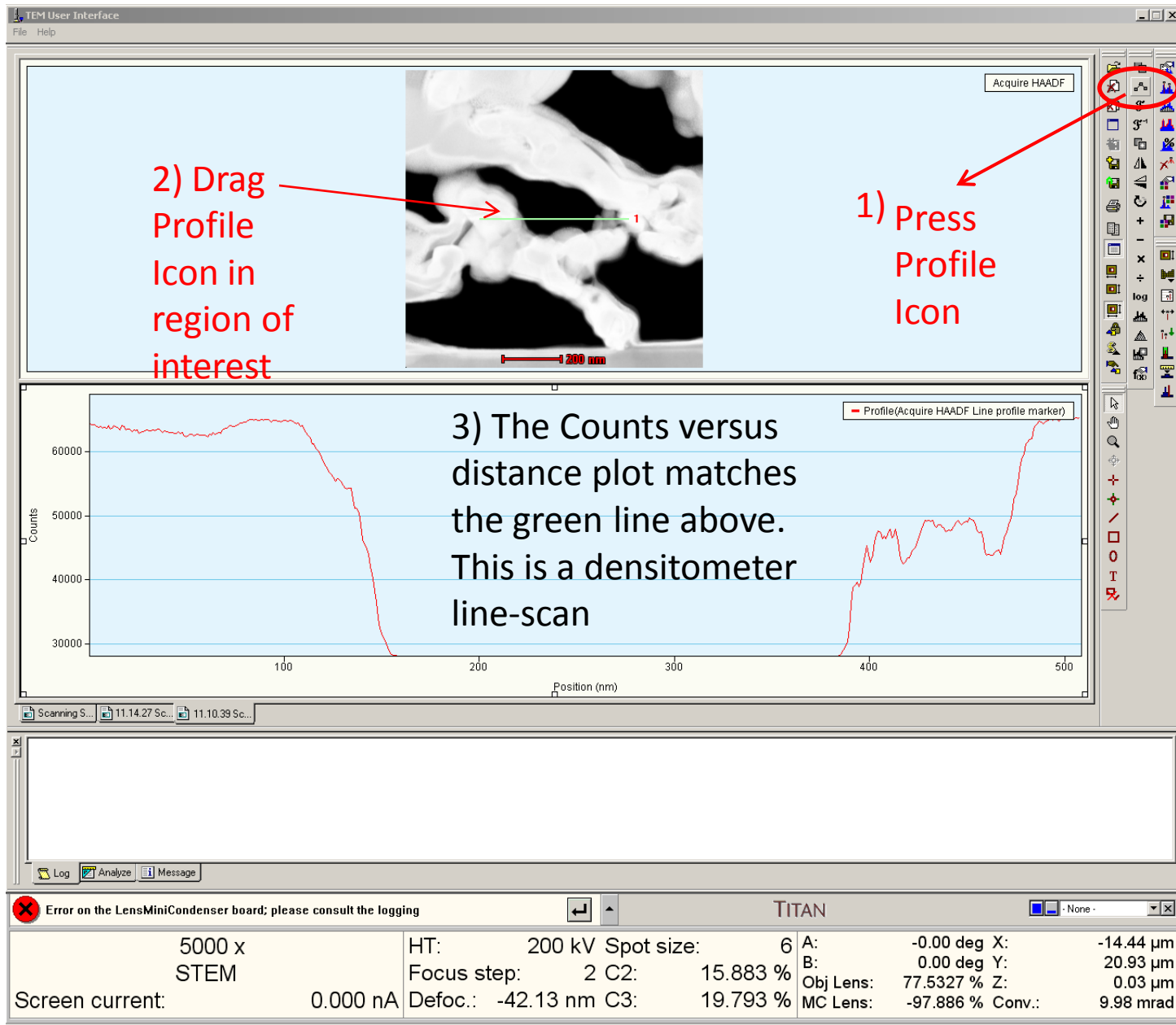
Commands

Acquire Align ZLP

Save Tune GIF

Print

# Advanced TIA Techniques: I. Profile of HAADF or Densitometer Line-Scan!



# Advanced TIA Technique: II

## simultaneous EDX and EELS line-scan

1) Analysis Tab of EM Worksheet

2) Select Spectrum Collection and Spectrum Profile in Experiment Settings dialog

3) Click flap out on the Experiments dialog and set the number of points in the PEELS spectra and the dwell time

The screenshot shows the TEM User Interface software. The 'Analysis' tab is selected in the top menu. The 'Experiment Settings' dialog is open, showing the 'Acquisition settings' and 'Configuration settings' sections. The 'Number PEELS spectra' is set to 10 and the 'Dwell time (ms)' is set to 1000. The 'Spectrum Collection' and 'Spectrum Profile' options are selected in the 'Select component' dropdown. The 'Settings' dialog is also open, showing the 'Acquisition settings' and 'Configuration settings' sections. The 'Number PEELS spectra' is set to 10 and the 'Dwell time (ms)' is set to 1000. The 'Spectrum Collection' and 'Spectrum Profile' options are selected in the 'Select component' dropdown. The 'Settings' dialog is also open, showing the 'Acquisition settings' and 'Configuration settings' sections. The 'Number PEELS spectra' is set to 10 and the 'Dwell time (ms)' is set to 1000. The 'Spectrum Collection' and 'Spectrum Profile' options are selected in the 'Select component' dropdown.

SA 10500 x TEM  
Screen current: 0.000 nA

HT: 200 kV Spot size: 3  
Focus step: 2 C2: 52.984 %  
Defoc.: 100.56 µm C3: 34.190 %

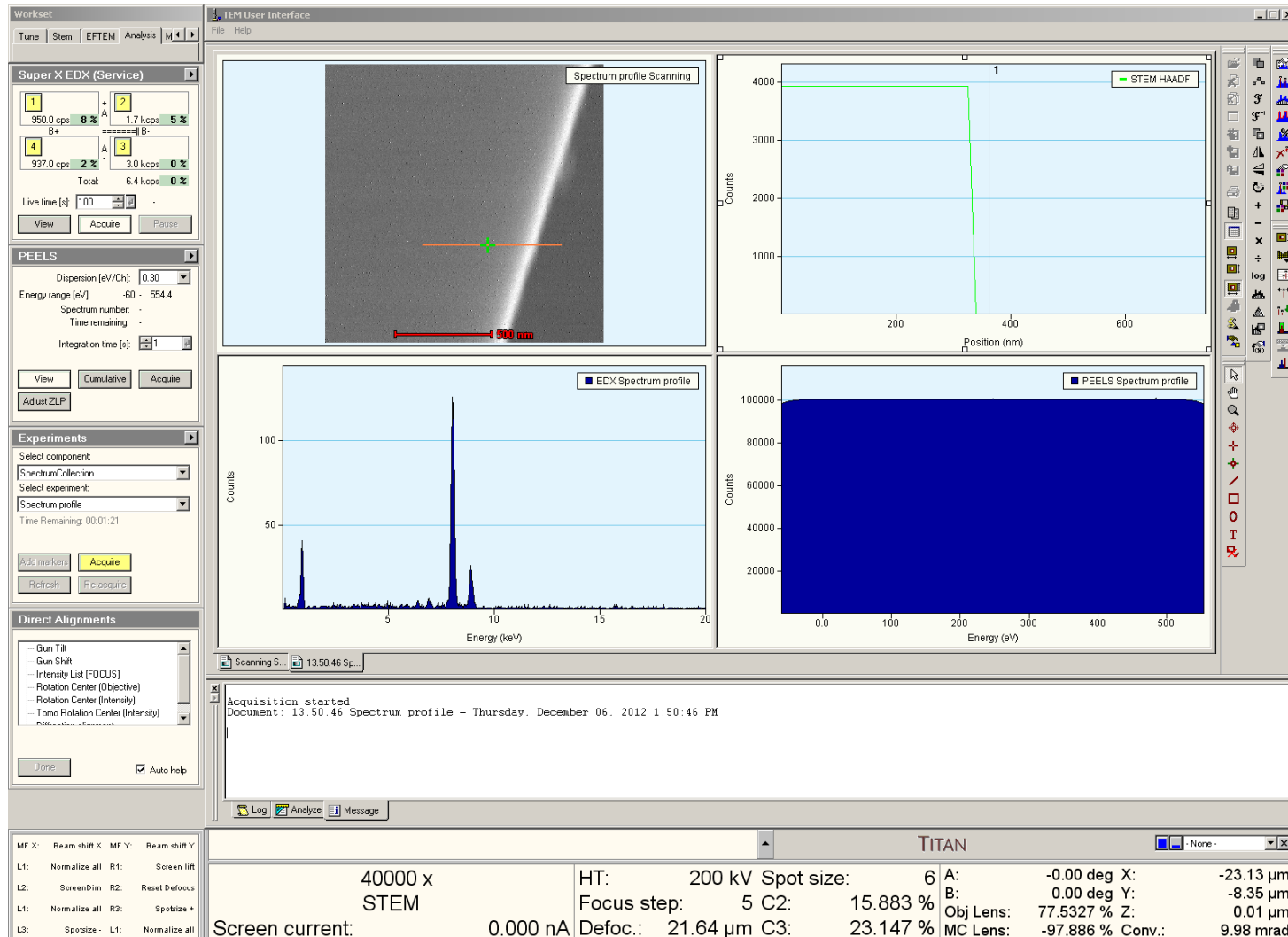
A: -0.00 deg X: -0.18 µm  
B: 0.00 deg Y: 0.08 µm  
Obj Lens: 81.5873 % Z: 0.03 µm  
MC Lens: 97.886 % Conv.:

The HAADF detector has a hole in it and so we collect the energy loss electrons below while the EDX x-rays are collected above near the sample!

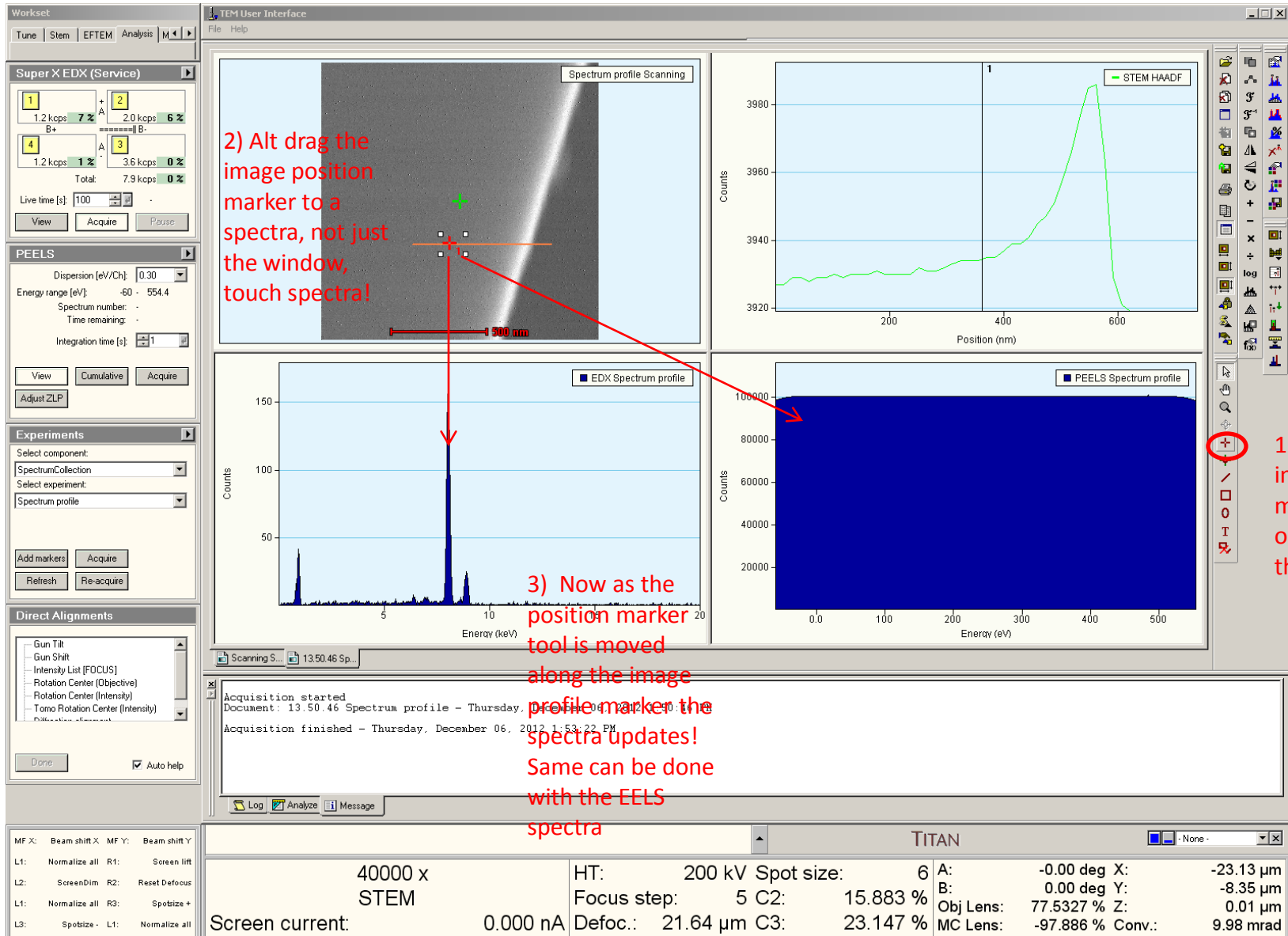


# TIA Advanced Techniques: II continued

## Simultaneous EDX and EELS Linescan



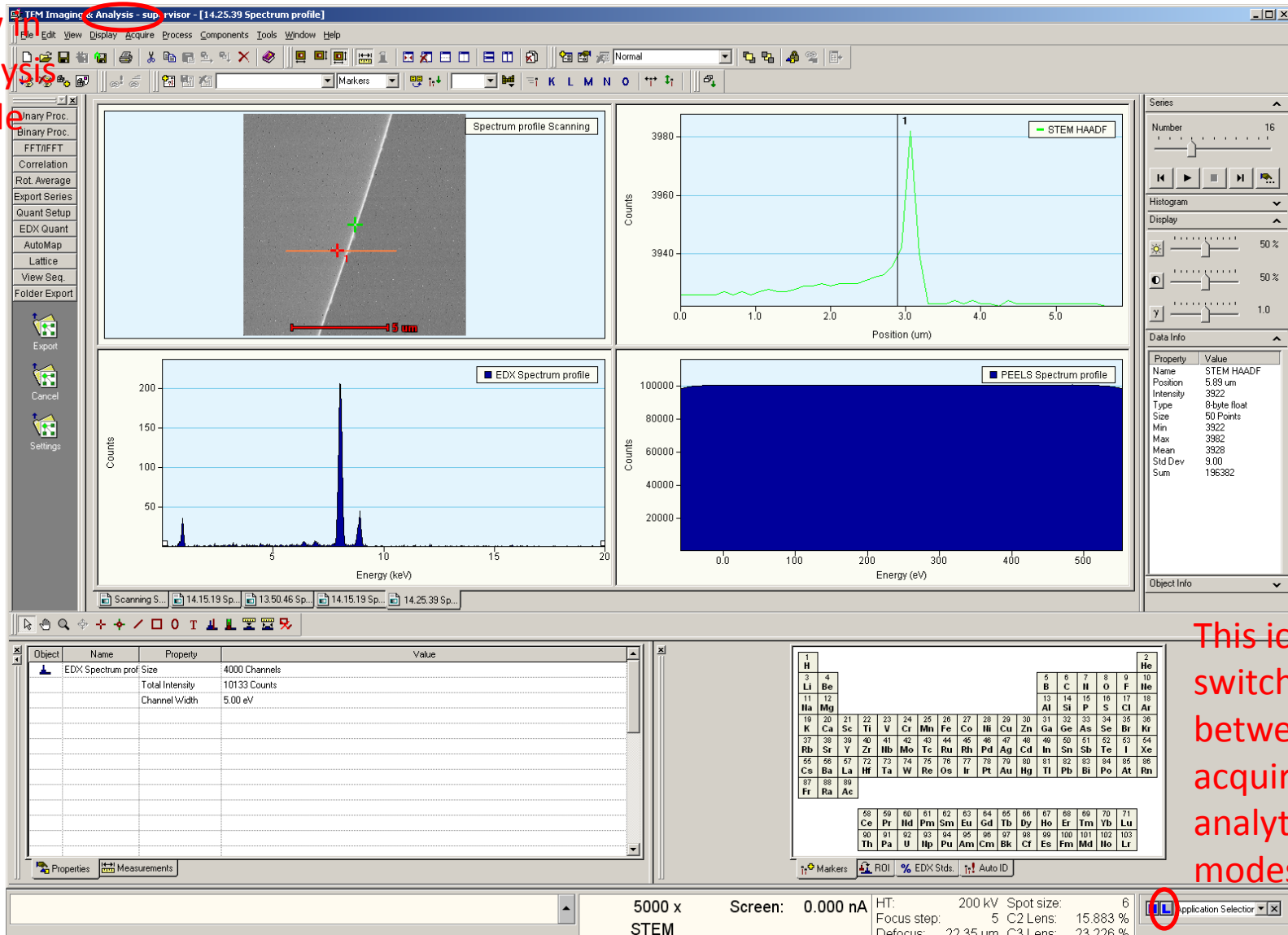
# Advanced TIA Techniques: III linking the position marker on the Image to the EDX and EELS Spectra Perhaps the Best Feature in TIA



# Advanced TIA Techniques: IV Switching

## TIA modes

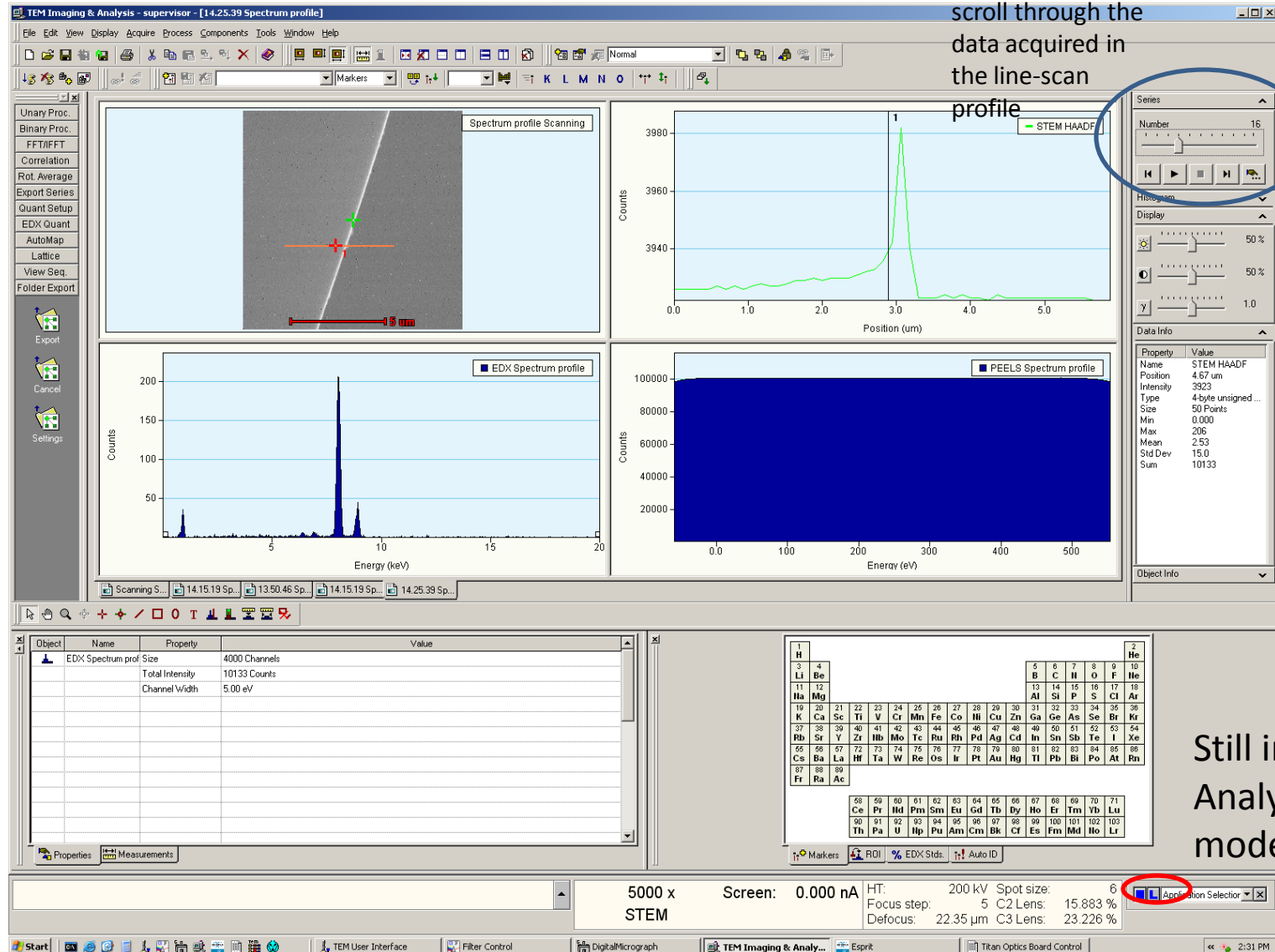
Now in  
Analysis  
Mode



This icon  
switches  
between  
acquire and  
analytical  
modes

# Advanced TIA Techniques: V. Series tool

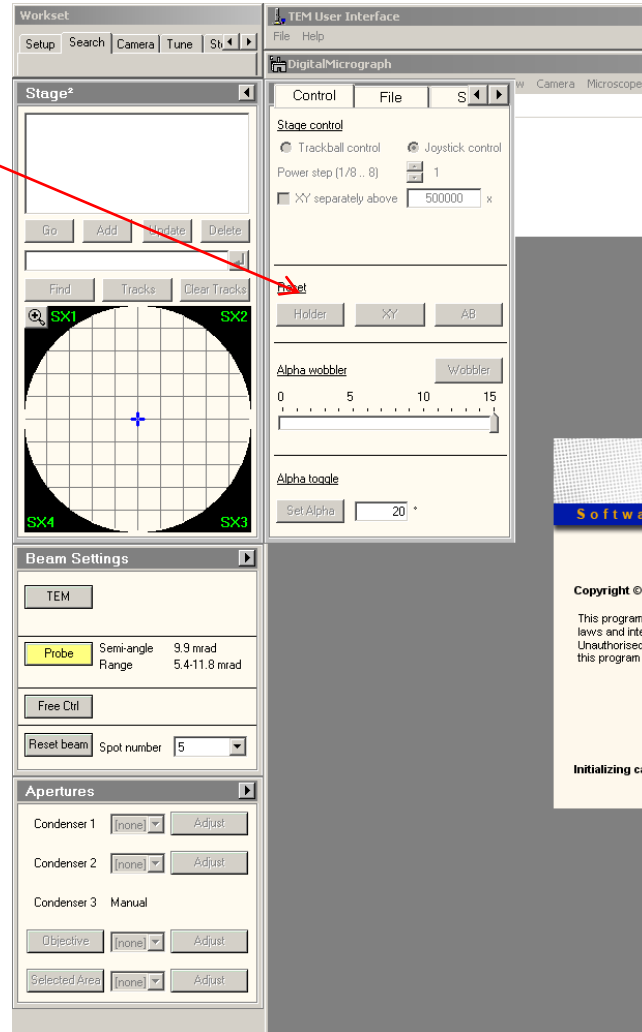
Series tool is yet another way to scroll through the data acquired in the line-scan profile



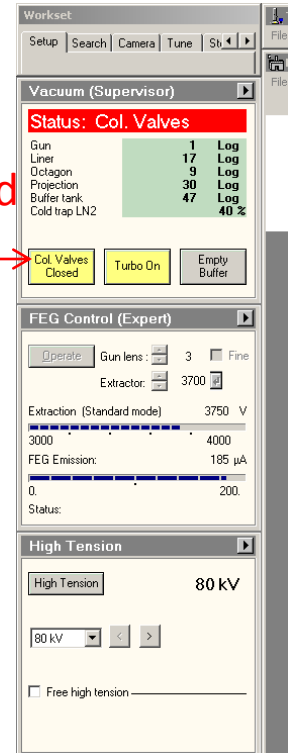


# Resetting holder, so that rod can clear when removed!

1) Click reset holder



2) Click Col Valves till it says Col Valves Closed in yellow



# To shutdown

- Save database if using EDS mapping, this saves the hyperspectral data cube for post processing.
- Close EDS shutters
- Click off STEM Insert
- Set back to TEM with registry, make sure TEM beam comes back
- Reset holder, see previous page
- Shutdown column valves, previous page
- Remove specimen rod

The End