The image shows the cover of a spiral-bound notebook. The cover is a light tan or beige color with a subtle, mottled texture. On the left side, there is a silver metal spiral binding. The notebook is set against a light yellow background. The text on the cover is centered and written in a bold, italicized, black serif font.

***Scanning Electron Microscope
(FEI Sirion)
Training Notebook***

***NanoTech User Facility (NTUF)
Center for Nanotechnology
University of Washington
April 2008 (rev. 8)***



SEM Operation Procedure

- I. Initiate Software
- II. Load Sample
- III. Beam Settings
- IV. Calibrate Stage Height
- V. Beam Optimization
- VI. Image Acquisition & Transfer
- VII. UHR Mode
- VIII. Shutdown
- IX. Default Settings

I. Initiate Software

📄 First open **XL Microscope Control** Program.



📄 Next open **Scandium** Database.

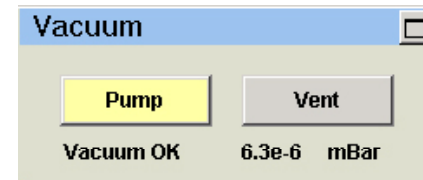


***Note:** Shutdown the computer and reboot if any software freeze occurs. Sirion SEM will not be affected.*

***WARNING:** Never touch any buttons on the front panel of the Sirion table.*

II. Load Sample

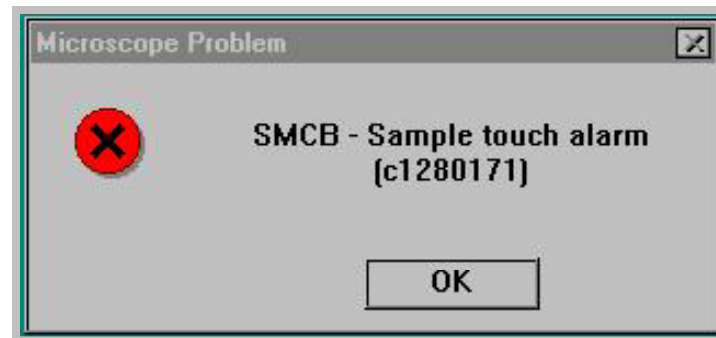
Handle samples and stubs with gloves and blow them with the nitrogen gun before putting samples in SEM chamber.



- Select **Vent** in the **Vacuum** submenu.
- With gloved hands, insert sample stub and tighten using set screw (finger tight).
- **Set the Height Adjuster on the stage base to make sure the sample has at least 5mm of detector clearance.**
- The stage switch should be set to “A” for imaging or EDAX.
- Close the chamber door and pump down by selecting **Pump** in the **Vacuum** submenu.

II. Load Sample

*If 'Microscope Problem: Sample Touch Alarm' box appears **while loading samples**, do not be alarmed. Click **OK** and continue.*

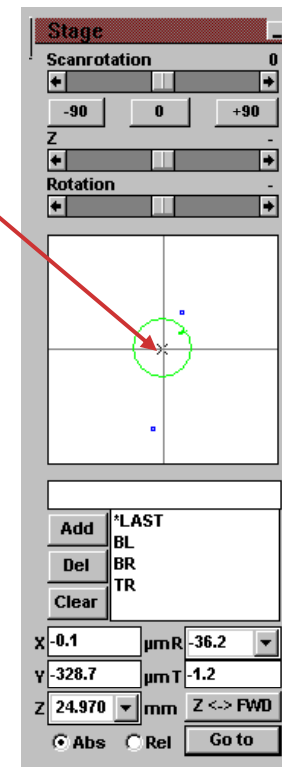
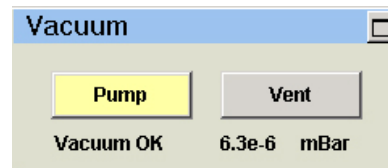


WARNING: If this box appears after pump-down, during SEM operation, consult with NTUF staff immediately!

II. Load Sample

Double click center of the crosshair to center the sample stage using the **Stage** submenu reference map.

Wait for vacuum status message to read **Vacuum OK**.



III. Beam Settings

- Select appropriate accelerating voltage (kV) and spot size from **Beam** menu:

Accelerating Voltage

Conductive samples ≥ 5 kV

(Silicon, metal, ITO)

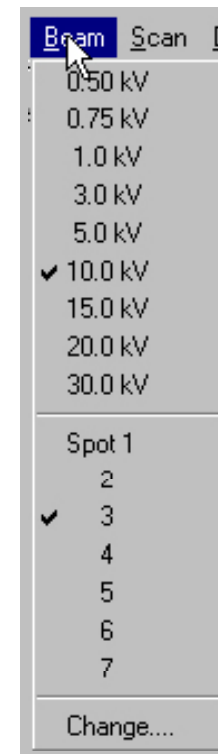
Nonconductive samples ≤ 5 kV

(glass, polymer, epoxy, some metal oxides)

Spot Size

Spot size 3 is best for nano-scale

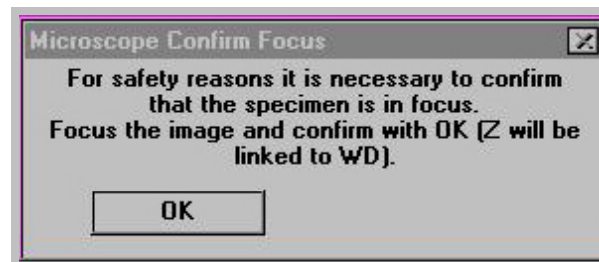
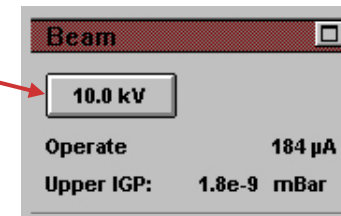
Larger spot size for micro-scale



IV. Calibration of Stage Height

- Turn on the accelerating voltage from **Beam** submenu by clicking the box (kV).

*Microscope dialog box will appear,
DO NOT CLOSE this window!!*

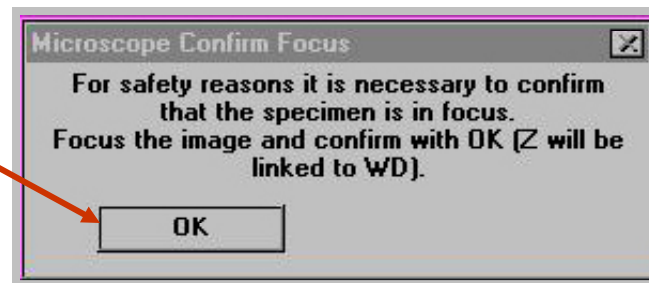


- Auto adjust brightness and contrast (F9).



IV. Calibration of Stage Height

- Use '+' and '-' buttons on number pad to increase/decrease magnification respectively.
- Holding the right mouse button, move the mouse left-right to focus on the sample with magnification $\geq 2000X$.

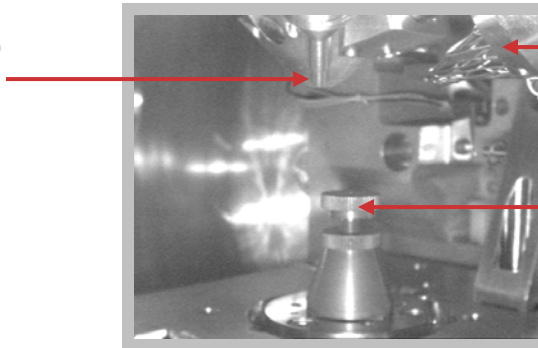


- With the sample in focus, click **OK** on **microscope confirm focus** to update Stage Height (Z) with the accurate Forward Working Distance (FWD).

IV. Calibration of Stage Height

- View inside chamber on external TV monitor (make sure both monitor and CCD camera are on).

**Pole Piece/TLD
Detector**



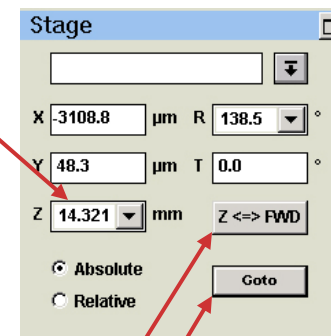
SE Detector

Sample Stage

Always watch the sample on the TV monitor when you raise, lower, or tilt the stage.

IV. Calibration of Stage Height

- In the **Stage** submenu select **Z = 10 mm** and click **Goto**, raising the sample to ~10 mm.
- Refocus the sample at this new stage height with magnification $\geq 2000X$.



↓

Acc.V	Spot	Magn	Det	WD	Exp	500 μm
5.00 kV	1.0	43x	SE	9.6	1	

The WD after refocusing will be 9.5-10.5 mm if the sample was well focused initially.

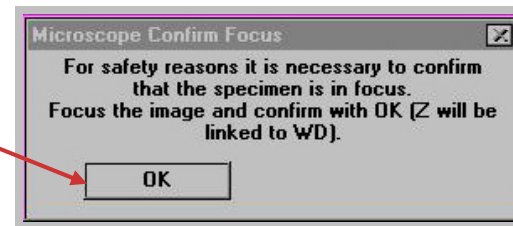
IV. Calibration of Stage Height

Click **Z<=>FWD** to open the **microscope confirm focus** box.

With the sample in focus click **OK** to update the stage height.

Now select the final Z (≥ 5 mm) and click **Goto**, raising your sample up to the final WD.

Move to the area of interest using the stage tracking tool.



Final Stage Height

Micro-scale Features Z ≥ 7.5 mm

Tilted Samples Z = 7.5 mm

E-Beam Lithography = 6.5 mm

Nano-scale Features Z = 5 mm

X-ray Analysis Z = 5 mm

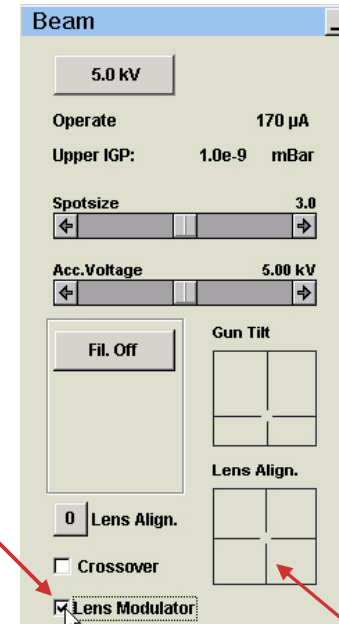
V. Beam Optimization

Lens Alignment:

- Change **Scan** to **TV** mode.



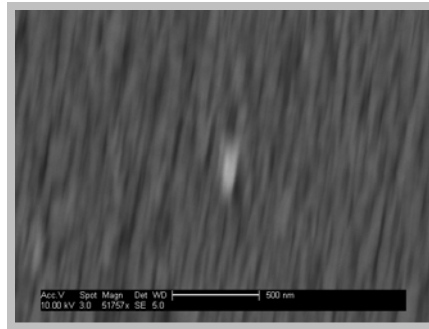
- Increase magnification to $\geq 10,000X$.
- From **Beam** submenu turn **Lens Modulator** on by clicking the box.
- Using **Lens Align.** box, click and hold the left mouse button to correct for X & Y image translation.
- Final image should remain stationary with no X or Y translation. The image should oscillate in/out of focus.



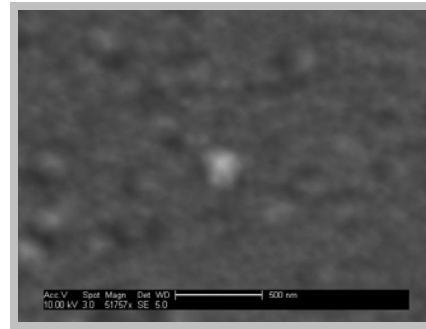
V. Beam Optimization

Stigmation Adjustment:

- Obtain best possible focus on image. (See examples below)
- Image should not exhibit streaking or distortion before adjusting stigmation.
- Hold down shift and right mouse button.
- Drag green cross in X & Y until image resolution is well defined.
- Re-focus image using right mouse button.



Incorrect focus for stigmation adjustment

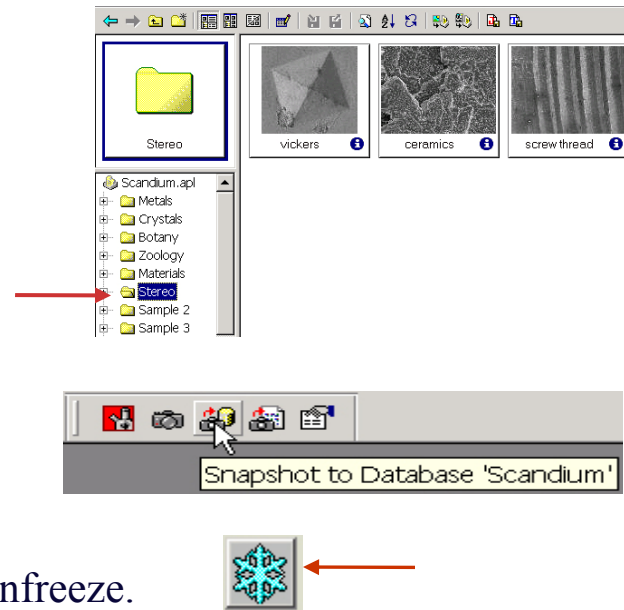


Correct focus before stigmation adjustment

VI. Image Acquisition


F2 Capture (Standard Definition):

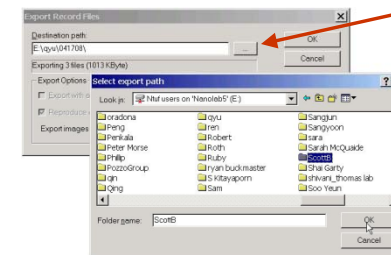
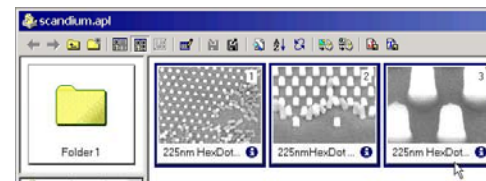
- Push **F2** key to grab an image (single scan: slow scan 4 / 720 x 968 pixels ~ 300 kb).
- Wait for scan to finish.
- Select your folder in the Scandium list.
- Click middle icon to transfer image to database folder.
- To return to live imaging, unfreeze.



VI. Image Acquisition

Transferring F2 Images

- Select the images to transfer.
- Click  to save images on network drive (Z:/NTUF Users).
- Select your folder as the destination.
- Click OK to transfer images.
- Move to Nanolab 5 computer and open the **NTUF Users** folder on the desktop.
- Transfer images into your personal Flash drive from your NTUF Users folder.



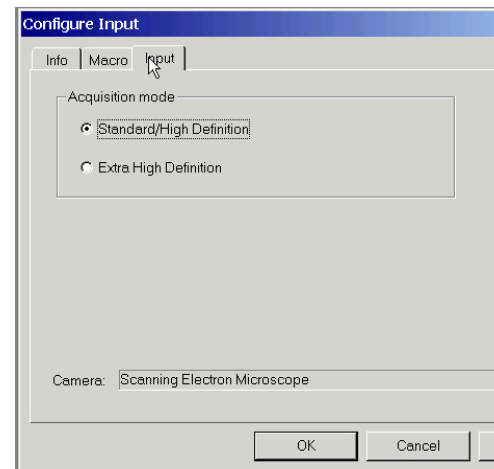
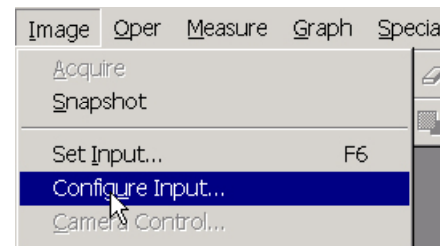
Remember: transfer all of your images to Nanolab5 at the end of each SEM session.

VI. Image Acquisition

F5 Capture (Extra-high Definition)

- From **Image** menu in **Scandium**, select **Configure Input**.
- Select **Input** tab to define Acquisition mode.
 - Standard High Definition = F2
 - Extra High Definition = F5
- In **Microscope Control XL**, push **F5** key to grab an image (XHD: slow scan 4 / 1404 x 968 pixels ~ 1.2 Mb).

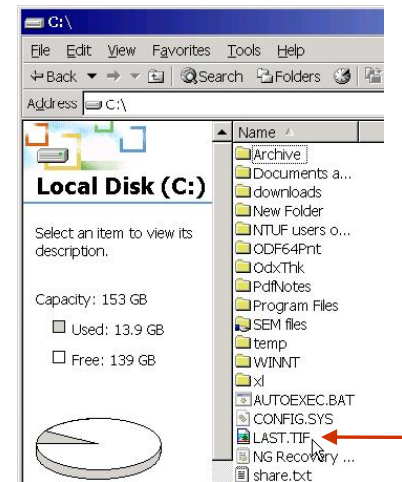
Remember: Always reset to default mode - Standard/High Definition.



VI. Image Acquisition

Transferring F5 Images

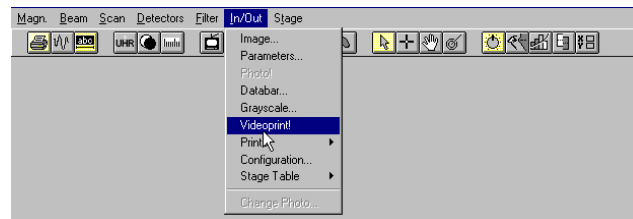
- New F5 images always save as Last.TIF on the C-drive.
- Open the **NTUF Users** folder on the desktop.
- Select your folder from NTUF Users.
- Open **My Computer**, local drive (C:)
- Drag and drop LAST.tif from C: to NTUF User folder.
- Rename with .tiff extension before capturing another F5 image.



VI. Image Acquisition

Print

- From the In/Out menu pick **Videoprint!**

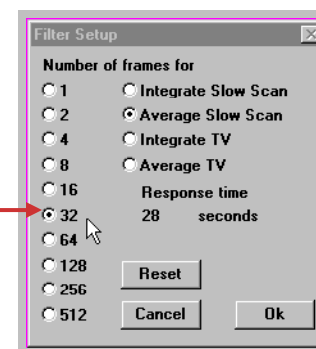



- Pull up to tear off photo paper.
- Report the number of prints when logging out SEM appointment on the NTUF website.

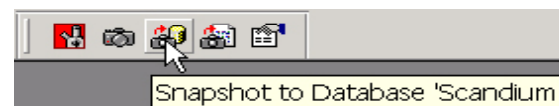
VI. Image Acquisition

Non-conducting samples

- From **Scan Menu**, select a scan rate which does not exhibit charging effects (distortion).
- Change Filter **Average Slow Scan** to achieve a reasonable image (ie. 32 frames = 28 sec).




- Once electron beam has scanned entire screen, click  to freeze image.
- With image frozen, transfer to Scandium as with F2 capture.



VII. UHR Operation

UHR Mode is INVALID for potentially magnetic samples to avoid DAMAGE to the detector!

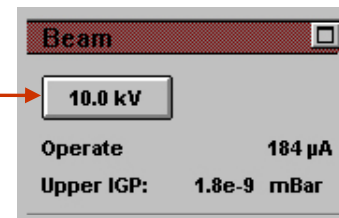
- Before enabling UHR Mode, the sample must meet all of the following conditions:
 - NOT MAGNETIC
 - $WD \leq 5 \text{ mm}$ for accelerating voltage $\leq 20 \text{ kV}$
 - $WD = 6 \text{ mm}$ for 30 kV
 - Magnification $\geq 1000X$

- Find the area of interest on your sample.
- Select the UHR button from toolbar. 

- Always turn off UHR mode before changing samples or venting the chamber.

VIII. Shutdown Procedures

Click **Beam** to turn off accelerating voltage.



Select **Vent** in **Vacuum** submenu - sample will automatically drop to lowest stage position.

Reset Beam to 5kV, spot 3

Reset all changes you made in Microscope Control XL to default settings (next page).

- Wait for specimen chamber to come up to atmospheric pressure (approximately 2 minute).
- With **gloved hands**, loosen set screw and take out sample stub.
- **Close the chamber door and pump down by selecting Pump in Vacuum submenu.**

IX. Default Settings

SOFTWARE

XL Microscope Control

- Magn > Reference (Videoprint)
- Beam > 5kV
Spot Size 3
- Scan > Fullframe
- Filter (TV) > Average 4 frames & Standard def.
- Filter (Slow Scans) > Live, Standard def.
- Stage > ✓ Stage Current
✓ Auto beam shift zero

Scandium

- Setup > Image > Configure Input > ✓ Standard High Def.

HARDWARE

- Stage Switch: "A" (Alarm)
- External Beam Blanker: Off
- Cable Bundle: EDX
- A/B Switch Box: A (EDAX)
- Channel Box: 2
- CCD Camera Box: On
- External (CCD) Monitor: On



Index of Topics

- I. Initiate Software (slide 3)
- II. Load Sample (slide 4-6)
- III. Beam Settings (slide 7)
- IV. Calibrate Stage Height (slide 8-12)
- V. Beam Optimization
 - Lens Alignment (slide 13)
 - Stigmatism (slide 14)
- VI. Image Acquisition & Transfer
 - F2 Image Acquisition (slide 15-16)
 - F5 Image Acquisition (slide 17-18)
 - Videoprint (slide 19)
 - Non-conducting Samples (slide 20)
- VII. UHR Mode (slide 21)
- VIII. Shutdown (slide 22)
- IX. Default Settings (slide 23)