SAFE USAGE OF SMIF SEM2
FEI-XL30 ESEM

- Don’t press the buttons on the SEM module (on the front of the chamber)
- Don’t make any changes to the computer settings (including screen resolution) as the current settings are needed for proper SEM functionality
- Loading the sample into the chamber
  - Don’t touch the interior of the chamber.
  - Check the height of the sample using “elephant’s ear” height gauge.
  - Open and close the chamber door using the EXTERNAL door handle.
  - Watch the door closing on the monitor (change the detector setting to ‘CCD’). **Verify the sample/holder is low enough to fit under the lens.**
  - Make sure the door is fully closed before you start pumping.
- Before turning beam on verify the contrast setting isn’t too high (typical:20-50%)
- Set contrast and brightness manually the 1st time before using the ACB button.
- Moving the stage
  - The easiest way to damage the SEM is to crash your sample into the pole piece (a cone just above the sample). Unfortunately, the microscope has no interlocks for this case, so if you move the stage always check using CCD that you are far enough from the pole piece.
  - Have Z distance (pole piece – sample distance) set correctly. When loading the sample, this distance is unknown (the SEM assumes that it’s the same distance as it was for the previously loaded sample). After focusing the beam on the surface of your sample, “working distance” (WD) shows the actual distance between the pole piece and the sample. Have Z set equal to WD at this point! (The SEM asks to confirm that Z<=WD every time you turn the beam on, don’t click “OK” without focusing the sample first). **Stop and ask clarifications if you do not understand the difference between Z, WD and the actual distance between the pole piece and the sample.**
  - Having several samples of different height and moving from one of them to another can also lead to crashing into the pole piece. To avoid it increase Z and check with CCD what is happening inside the chamber as you are moving the stage.
- If using the Peltier stage (**wet mode**), **never rotate or tilt** a sample
- Don’t try to use the SEM2 in a mode for which you have not been trained on.
- Don’t place loose powder samples in SEM2 or use powdered gloves in the chamber.

Any Questions or Problems: **Contact SMIF Staff**
Email: smif@pratt.duke.edu       Phone: 660-5477 / 660-5488
Operating Procedures for SEM2
FEI XL30 ESEM - High Vacuum Mode

Start-Up

1) Sign in usage via the support computer.

2) Click on the **CCD** button in Microscope Control *(if not already yellow)*. This will give you a view of the sample chamber on the screen.

3) Check and if necessary adjust X (0), Y (0), Rotation (0), Tilt (0) and Z (15-20mm).

4) Select the **Vent** button in the Vacuum control box. Click **OK** in the dialog box and wait for the vacuum **Idle** message. It will take about 3mins for the chamber to vent.

5) Put on gloves and carefully open the chamber with handle. Place the specimen stub on the stage.

6) Check the height with the “elephant ear” gauge. The top of the sample should be just below the bottom of the “trunk”. If it is not, lower the stage with the Z knob.

7) Carefully close the door – watch the monitor to be sure the sample does not hit anything.

8) Be sure the lever on the black manual valve behind the microscope column is at **HIVAC** position.

9) Press the **Pump** button.

10) Go to the **Detectors** menu and select **SE**.

11) From the **Beam** menu, select an appropriate **kV** *(e.g. 1-30 kV)* and set the **spot size** to 5

12) From the **Scan** menu, select **Slow Scan 1**.

Imaging samples

13) Once you see **Vacuum OK** (below the Pump button), click the **kV** button in the Beam control group. Wait for the µA reading to stabilize. **Do not remove the Confirm Focus message.**

14) Click the **Autocontrast** button or modify the brightness and contrast using the sliders.

15) Magnification can be adjusted using the +/- keys on the numeric keypad, in the Magnification menu, or by clicking in the inner circle in **Zoom** mode.

16) Focus by pressing the RIGHT mouse button and dragging to left or right. Selecting the **Selected area box** and **Slow Scan 2** may be helpful.

17) Once the sample is in focus, click **OK** on the **Confirm Focus** window. If at any time the **WD** on the databar does not match the **Z** in the Stage control box, click on the Z<=>PFD button to link them.

18) Move the sample using one of the following methods: **(click and hold in the direction you want to move the stage)**, **(double click will bring that point to center)**, or **arrows** on the keyboard. **Rotate** using the **R** knob on chamber door, or adjust **R** field in **Stage** control group.

19) Adjust the working distance of the sample by typing a value in the Z box or by turning the Z knob on the front of the SEM. The optimal working distance for the SE detector is between 10-12mm.
changing the working distance, use the CCD view to ensure the sample does not hit the pole piece. The pole piece is very expensive and not covered under the service contract.

20) Correct the astigmatism if necessary and only if you are above 10,000x. Access the stigmator function by pressing the SHIFT and right mouse button together. Move the cross hair left to right and top to bottom to until the image improves. Try to make the edges in the image as sharp as possible.

21) Adjust Spot Size if necessary (e.g., try 4 @ >10000x). A smaller spot size will give better resolution, but a grainier image.

22) To acquire a digital image, press F2 for standard definition images (338KB).

23) Wait for the button on the tool bar to turn yellow.

24) To save the images, click the In/Out button on the menu bar. Then select Image.
   a) Select the e:\ drive (this is the support computer). Select your folder. The folder name must be only 8 characters. These characters must be numbers or letters only, and no spaces or special characters.
   b) Name your image. The name must be only 8 characters. These characters must be numbers or letters only, and no spaces or special characters.
   c) Press the Save button.
   d) This naming protocol is very important. The images will not save if they not named properly.

25) Click off to revert to a live scan.

26) Tilting the stage must be done manually. Adjust the stage height to at least a 15mm working distance. Then unlock the stage and while holding the stage handle, move the stage to the desired tilt angle. Then relock the stage. Always watch the tilting procedure on the CCD camera to be sure the sample does not hit the pole piece. The pole piece is very expensive and not covered under the service contract.

Leaving the SEM for the Next User

27) Decrease the magnification to the minimum value.

28) In the Beam control group, click the kV button to turn the filament off.

29) Click the CCD button to view the inside of the chamber.

30) Set stage Tilt to 0, Z to >15mm, X, Y, R to 0.

31) In the Vacuum control group, click the Vent button and wait for Idle message.

32) Carefully open chamber, with handle, and remove samples (wear gloves).

33) Close door and hold shut. In the Vacuum control group, select Pump. Wait for Vacuum OK status, then go to the Vacuum control group and select RPM 70%.

34) Remove images from the support computer via e-mail or storage media. Then Close Scandium.

35) Sign out of the usage log via the computer