# PROCEDURES FOR OPERATION OF THE JEOL 6330F FIELD EMISSION SEM

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**Getting Started**

When you approach the microscope, the pumps and water supply should be running smoothly.

Turn on the electronics using the button marked I under the bench.

Turn on the monitor. If the ‘states’ page is shown with an orange highlighted box, there has been a failure, call the Technician.

This microscope is kept at ultra high vacuum status so it is vital that the holders are kept clean, sample stubs are **ALWAYS** handled wearing gloves, and that samples are completely dry before they are put into the machine.

GLOVES SHOULD ALWAYS BE USED WHEN LOADING THE SAMPLES INTO THE MICROSCOPE.

Please be very gentle with the microscope – do not force the airlock door or sample rod. Also do not continually change kV or WD as the microscope takes a little time to settle after any big changes.

**Sample Exchange**

ALWAYS WEAR GLOVES.
Check WD is 39mm, X displays 25 and Y displays 35.

1. Load either the single holder or the 5600LV multiple holder with your sample(s). If you use the multiple holder load a plain 32mm stub in the FE SEM specimen holder underneath your sample holder so that your samples are flush with the FE SEM holder surface.
2. Screw the holder onto the specimen exchange arm.
3. Place the plastic shield snugly onto the O-ring of the airlock (do not damage the plastic – any nicks or cracks will prevent the airlock from getting down to vacuum).
4. Whilst holding this in place, press the green button. The airlock will start pumping, when this is done the green light will go out.
5. CAREFULLY open the door by turning the wheel one quarter turn towards you, pulling the door out and turning the wheel one quarter turn away from you. This is now in the locked position.
6. Push the sample holder into the chamber and onto the stage carefully. Check through the viewing window that the holder is sitting correctly on the stage.
7. Unscrew the holder and pull it out.

8. Close the door by reversing the process described above.
9. Once the door is closed, push the green light and hold the arm near the plastic shield because the vacuum lets go quite quickly.

**Getting a beam**

1. Decrease the working distance (Z height) to 15, 8 or other suitable height using the stage Z height control.
2. Open the Control menu under Column and highlight this WD.
3. Check the accelerating voltage – this is usually 2 kV.
4. The HT button in the top left corner will remain black for two minutes after the airlock is used. It will then turn blue (HT ready) and then when you click on this to turn the HT on the button turns green. The current will ramp up slowly to about 12 – 12.5μA.
5. During the first hour or so this will gradually fall off, so remember to reset the current. This can be done automatically by highlighting the Auto Reset - CONSTANT command in the Control menu. REMEMBER TO CANCEL THIS BEFORE YOU TAKE A PHOTO.

**NOTES:**

If you can't find an image:
1. Check current is at about 12μA.
2. Reduce magnification to the lowest possible.
3. Check correct WD is highlighted in Column – Control menu.
4. Check focus.
5. If screen is black – increase and balance contrast and brightness
   white – decrease and balance contrast and brightness
   black, with noise – check gun alignment

   If the image disappears/changes significantly when swapping from TV rate to slow scan, check the contrast and brightness.

**The Console Controls**

Focus, Magnification, Contrast, Brightness – all fairly obvious.

X and Y deflectors - when using the alignment menu these control the highlighted parameter. When not in the alignment menu these control the X and Y objective lens stigmatism correction.

Photo, Freeze – buttons used when recording an image

ACB – auto contrast and brightness: a personal choice!!!

Wobb, Align, Stig – wobbler and alignment menu are used during the alignment procedure. Stig is highlighted when the alignment menu is not in use.

Quickview – TV rate, normal working speed.  There are two speeds so press the button twice and toggle between the speeds.  The "slow" quick view is the most useful (less noise).

Fineview – slower scan, again with two speeds.  The faster is useful to see how a photo would appear, the slower slow scan is used for the photograph.

RDC image – reduced image for focusing and stigmatism correction.

Consult the manual regarding all the menus along the top button bar – there are some useful ones, but too much to go into here!

**Alignment Procedure**

Unlike the 5600LV this machine has a manual alignment process.  This takes some getting used to before you 'get your eye in'.  When the beam is first turned on it will take about half an hour to settle – during this time you may align the microscope however it will need re-aligning after it has settled.

Always check the alignment before you take a photograph – especially in the first half hour or so.

The alignment has to be checked whenever you turn the kV off and on, change the kV value or change the working distance.

To bring up the alignment menu, press the 'Align' button on the console.  The list on the left contains all the different stages that need to be checked.  The X and Y deflectors on the console control whichever stage is highlighted in green.

Gun Alignment – this can be done at any mag

1. Open Control menu. [COLUMN – CONTROL]
2. At probe current 7 use the X deflector to find the brightest position (if you turn the dial one way the image will become dark, turn it the other way it will become bright then dark – pick the central point where the image is brightest.  This shows that the gun tip is pointing straight down the column and is central).
3. Repeat with the Y deflector.
4. Move to probe current 8, re-focus and repeat the process for both the X and Y deflectors.
5. Continually increase the probe current by 1 until you reach the maximum, 14.  If the screen gets too bright, reduce the contrast.
6. At probe current 14, after checking the gun alignment press Lens Clear three times to remove any residual magnetism from the lenses (historisis). You man need to re-focus after this stage.  If the image disappears, check the gun alignment, contrast, focus, reduce the mag and it should appear.

7. Check the gun alignment at probe current 14.

Condenser Lens Stigmatism
This needs to be checked at probe current 14, at high mag. – about x40K.
1. Focus the image as best you can.
2. Highlight CL Stig
3. Use X and Y deflectors and focus control to fine tune the focusing (correct the astigmatism).
4. Press Lens Clear three times and re-focus.
5. Check the stigmatism correction.

Objective Lens Aperture
This needs to be checked at probe current 7. Increase contrast and re-focus.
We have to check that the aperture is central by wobbling the current in the objective lens and checking the image remains central.
1. Increase the mag to x40K and re-focus.
2. Highlight OL Aperture
3. Press the wobbler button and wait a few seconds for the image to settle.
4. The image should appear to be going in and out of focus. There should be no X or Y movement. If there is, correct it using the X and Y deflectors.
5. Press Lens Clear three times and re-focus.
6. Check OL Aperture using the wobbler.
7. Turn off the wobbler.

Objective Lens Stigmatism
While we are centering the objective lens aperture it seems logical to correct the objective lens astigmatism.
1. Increase mag to x40K and re-focus.
2. Highlight OL Stig.
3. Use X and Y deflectors and focus control to fine tune the focusing (correct the astigmatism).
4. Press Lens Clear three times and re-focus.
5. Check the stigmatism correction.

X Stig Centre
The stigmatism correction coils need to be centred so that their correction factors are accurate.
1. Highlight X Stig Centre
2. Press wobbler button
3. The image should appear to go in and out of the plane of focus, there should be no movement in the X and Y direction. Use the X and Y deflectors to correct any movement.
4. Press Lens Clear three times and re-focus.
5. Check the X stig centre

Y Stig Centre
Repeat the process described above, but highlight the Y Stig Centre.

This whole alignment process may need repeating if the image is still poor – especially if the machine has not been used for a couple of days.

**Record an Image**

1. Select an area
2. Use RDC image to check focusing and stigmatism correction.
3. Use the fast slow scan to check the contrast and brightness.
4. Change to slow slow scan.
5. Press the freeze button. This will flash until one scan has completed then it will stop flashing and the image is frozen.
6. Change the legend by clicking in the text box, re-type your text and press return.
7. Go into Setup – Network Set to check settings. Close.
8. Click on the big N in the top button bar.
9. Re-name your image. Press return. Click on reset so the number returns to 001.
10. Click on Save.

This is saved onto the network. When you save any subsequent images from the same sample it is not necessary to change the text as the number increases by 1 automatically, for example, the first image will be saved 'silica001', subsequent images will be saved 'silica002', 'silica003' etc.

## USING THE X-RAY ANALYSIS EQUIPMENT

As in the TEM it is possible to use the X ray analysis to get an elemental spectrum. In the SEM, it is also possible to convert this into an elemental map showing areas of different elements. Make sure the sample for analysis is coated appropriately, e.g. when looking for calcium phosphate, for instance, do not coat in platinum because the platinum M line sits very near to the phospour K lines. In that instance you would use a carbon coating. However, if you are wanting to map areas of carbon in your sample, obviously do not coat in carbon, use platinum.

The X ray analysis probe is kept in position at all times in the SEM.

1. Chose the area that you want to map.
2. The kV and spot size will need to be increased to boost the signal and get sufficient readings to produce a spectrum. It is therefore recommended that any secondary electron image needed is recorded before analysis as this may cause damage to the specimen.
3. Select the Link ISIS icon on the microanalysis computer.
4. At the next screen, click on the Labbook. Then click on the icon that looks like a spectrum.
5. Change the kV to 20kV. Reduce the contrast and re-focus.
6. Ensure the working distance is at 15mm. Re-focus.
7. Click on Go.
8. Check the percentage dead time - this should be about 30%. If it is less than this, increase the spot size.
9. During acquisition, you can check the elements present by clicking on the question mark, then click on Auto ID. This will bring up a list of elements it thinks are present.

Click on any of these and a line will appear on the spectrum telling you which peak corresponds to that element. Click on Label Peaks to identify all the peaks. If you want to know what an unidentified peak is (use the ‘magnifying glass’ button to increase the size and scaling of your spectrum) then right click on that peak. The software will tell you what it thinks the peak is, and a list of possibilities as well. If you highlight any of these, a series of blue lines will appear on the spectrum. These are all the keV emission values for that element. If these lines match your series of peaks then that element is present.

10. Spectra can be saved to the hard drive by selecting File, Save a Spectrum. Or they can be saved to the network by selecting File, Export as Tiff, m drive etc.

Using the Mapping function.

To produce an elemental map of an area:

1. Select the elements of a spectrum that you wish to see by right clicking on that peak followed by PAINT WINDOW. You can choose as many elements as you like to map.
2. Then click on the button that looks like a grid.
3. Click on GO and wait for the images to fully appear.
4. These map groups can be saved onto the hard drive. To transfer them across to the network, each elemental map has to be moved and saved individually. So, at the map group, highlight the elemental map you want saved (red diamond in the right corner), go to File, Export as TIFF, choose m drive and save as normal.

Spot Analysis using the Autobeam function.

1. Carry out steps 1 – 6 above.
2. Go back to the Labbook, click on the icon next to the spectrum (looks like a microchip!!).
3. Click on Go (triangle shape). This will lock you out of the microscope controls while the ISIS system is collecting an image.
4. Click on Stop (square shape).
5. Click on the spot on the image that you want t o collect from.
6. Go to the spectrum and click on Go. Correct the spot size to get adequate acquisition.
7. You can save the Autobeam image to the hard drive or to the network.

**BACKSCATTERED ELECTRON IMAGES**

Backscattered electrons are used to produce ‘atomic number contrast’ images. These are not topographical and the detector is positioned above the sample rather than to one side. For this reason, the best BSE images are produced from flat, polished surfaces. If you have a sample with an overall low atomic number, e.g. mostly carbon and you suspect the presence of heavy metal, a BSE image will show any areas containing elements with a high atomic number. Areas of high atomic number will appear brighter than areas of low atomic number. This is not a quantitative method of analysis, and it will not tell you the exact element present.

1. Turn the HT off.
2. Check the tilt is 0°. If using a very flat specimen, the minimum working distance is 8mm. If your specimen has any height, take this into consideration and increase the WD.
3. Insert the detector. Consult the backscattered detector manual for use.
4. When you are finished, turn the HT off and pull the detector out of position.

**Closedown Procedure**

1. Reduce the mag to the lowest possible.
2. Click on the green HT button.
3. Return the Z height to 39, the X value to 25 and the Y value to 35.
4. Put on gloves and remove your samples. Don't leave samples lying around the microscope. They will be thrown away.
5. Turn off the monitor.

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