

## Basic Operating Instructions for JEOL 6320FXV FESEM

**You have to be trained on the instrument before attempting to use it on your own. Always make sure that you understand what you are doing!  
If in doubt don't hesitate to ask Lou or Evgenia.**

### Loading the sample

**Always use gloves when handling the sample holder and the sample that you are going to put in the microscope!**

1. Check that ACCEL VOLTAGE-READY lamp is lit (do not confuse it with the ACCEL VOLTAGE ON/OFF button) and V7G button is depressed and is bright red.
2. Place your sample in the sample holder. Use the screwdriver provided to lock the sample in the holder. Do not tighten the screw too much, it should be just finger tight!
3. Turn on the infrared camera by pressing "Camera" button and turn on the TV monitor.
4. Check the position of the stage: make sure that  $Z = 25$  mm;  $Y = 35.0$  (left handle) and  $X = 25.0$  (right handle), tilt =  $0^\circ$ .
5. Move the glass cap on the exchange rod all the way towards the sample until it locks, insert the holder into the specimen exchange chamber and while holding the glass cap in place, press the vacuum control button and wait for the light to go off (this will evacuate the specimen exchange chamber).
6. Perform the next step immediately after the light goes off. While performing the next step hold the end of the holder rod. Open the specimen exchange isolation valve: rotate the knob towards you by 90 degrees, pull it out until it stops, rotate it away from you by 90 degrees. Push the sample holder in slowly until you position your sample firmly (look on the TV screen and inside the microscope chamber through the glass cap).
7. Unscrew the rod and retract it carefully.
8. Close the specimen exchange isolation valve: rotate the knob towards you, push it in all the way and rotate it away from you.
9. While holding the specimen exchange rod, press the vacuum control button and wait for the specimen exchange chamber to vent, then remove the rod and place it on the stand.
10. Check the ACCEL VOLTAGE-READY lamp is lit, if it is not lit, it means that the vacuum is not sufficient to operate the microscope. You will need to wait until the lamp is lit.
11. For polymers, you will probably be working at 5kV or lower and you will have to raise your sample (lower the working distance). You also need small working distance for high resolution work on any material. If your sample does not extend above the sample holder, change Z to 6 mm, otherwise change it to 15 mm and maybe 8 mm. Be careful and watch what you are doing on the TV monitor.

### Getting a low mag SE image

1. Login using your user name and password (this will be set up soon).
2. Press the ACCEL VOLTAGE ON/OFF button and wait until the EMIS reads 7.5 mA (If during your session EMIS reads a value below 7, press the EMISSION CURRENT RESET button on the right hand side of the microscope).
3. Press V7G button (the lamp will become dim).
4. On the main menu click on the EM MODE button and drag the menu to the convenient location on the screen. Do the same for VIDO and ACQUIRE buttons.
5. Click on the SLM button on the computer screen (Electron Optics Mode menu), go to low mag and find the area of interest or a feature that you can focus on.

It is advisable whenever possible to perform initial focusing, alignment and stigmation OFF the area of interest but somewhere near by, so that you do not irradiate the areas of interest for prolonged period of time.

6. Press FAST button to get a TV image. You can also go to slow scan by pressing SLOW button. By pressing the button again you will get different scanning rates (the same applies to TV mode - FAST button).
7. Adjust brightness and contrast.
8. Focus the image (rule of thumb for final focusing: focus the image at 10x of the magnification you are going to take picture at).
9. For polymers, start working at 5kV accelerating voltage and 4 or 5 probe size. If you can't get good image after focusing and stigmating, you might have to change these conditions. To change the spot size, use PROBE CURRENT button on the microscope control (It will be displayed in the main menu under CL COARSE). To change voltage, use the knob next to the ACCEL VOLTAGE button. Please note that you will have to re-align the microscope if you make these changes. If your sample is charging you can either coat it or lower the voltage and use smaller spot size.

### Alignment and stigmation

1. Aperture alignment (mag 20K - 50K) **Before you do this yourself, please get sufficient training from Lou:**
  - Turn on WOBLER button and observed the image sweep. Adjust (very small adjustments!) objective aperture to minimize the sweep.
  - Turn off WOBLER.
2. Astigmatism correction:
  - Start at relatively low magnification (such as x5K-20K) and go to about 100K for final adjustments. Focus the image, then adjust STIGMATOR X and Y to improve the image (when astigmatism is present you will see the image elongation as you go in and out of focus using the focus button).

### **Getting a high mag SE image**

1. Find the area of interest and focus the image.
2. Click on the SHR button on the computer screen (the image will be rotated compared to the SLM mode).
3. If you would like to rotate the image, use the SCAN ROTATION device. Turn the switch to ON and use the knob directly underneath to rotate the image. When you switch the SCAN ROTATION to OFF it will come back to the setting that corresponds to the actual position of your sample in the microscope.

### **Using digital imaging**

- 1. Make sure you are in slow scan mode! Otherwise the computer will lock up.**
2. Click on OFF and then on ON in the Video Display menu on the computer to get a live image. You can still use all the microscope controls.
3. To acquire an image click on SLOW in the Acquire Frame menu, wait until the image is acquired. Go to File → Save as Tiff. Choose your directory (will be set up soon), give a name to the file and click save.
4. If your sample is charging, it might be better to take an averaged image rather than a SLOW scan. To do this click on INTG in the Acquire Frame menu. Then save the image as above.

SLOW scan in the Acquire Frame menu will produce a better image providing that your sample is not charging.

Make sure you write down the magnification of the image (and other parameters if needed), so that you can put a scale on the image later.

**You are responsible for transferring your images to your computer immediately after your session. The images will be deleted from the server periodically without notice. If you are experiencing a problem, please contact the Lab personnel as soon as possible.**

### **To remove the sample and to shut down**

1. Go to TV mode (FAST button) on the microscope.
2. Press ACCEL VOLATAGE ON/OFF button to turn it off.
3. Close the V7G valve (depress it, it should become bright red).
4. Adjust sample settings: Z = 25 mm; X = 35.0 (left handle) and Y = 25.0 (right handle), tilt = 0 °.
5. Put on gloves.
6. Move the glass cap on the exchange rod all the way to the end until it locks, insert the rod into the specimen exchange chamber and while holding the glass cap in place, press the vacuum control button and wait for the light to go off (this will evacuate the specimen exchange chamber).
7. Perform the next step immediately after the light goes off. While performing the next step hold the end of the holder rod. Open the specimen exchange isolation valve:

rotate the knob towards you by 90 degrees, pull it out until it stops, rotate it away from you by 90 degrees. Push the rod in slowly and attach it to the sample holder (look on the TV screen).

8. Pull the sample gently out of the microscope until it stops.
9. While holding the end of the rod, close the specimen exchange chamber isolation valve: rotate the knob towards you, push it in until it stops, and rotate it away from you.
10. While holding the specimen exchange rod, press the vacuum control button and wait for the small chamber to vent, then remove the holder from the microscope.
11. Check that the ACCEL VOLTAGE-READY lamp is lit.
12. On the computer, go to File → Exit.

**13. Fill in the Log book.**

**Emergency shutdown** (e.g. fire)

1. Press the ACCEL VOLATAGE button to turn it OFF.
2. Turn the POWER key to OFF.
3. Turn the MAIN POWER switch on the SIP/BAKEOUT power supply to OFF.