

Microprobe Notes

Coating.

- Sharpen carbon rod in Polaron coater and flatten face of the other rod with a file.
- Brush off any loose carbon.
- Clean the base plate and brass plate with ethanol on a paper towel.
- Close the valve in front of glass dome.
- Fit the dome and safety shield.
- Turn the **Power** switch on.
- Turn the **Diffusion Pump** switch on.
- Press the **Pumpdown Start** button. Vacuum should be below 10-4 torr after about 10 min.
- Coat for about 20 min - leave longer if possible.
- Move the **Pulse** switch up 6 times to get 25nm coating. The brass plate should go a royal blue colour. Let the vacuum pressure fall between pulses if it rises above 10-4 torr.
- Press **Pumpdown Stop**.
- Turn **Diffusion Pump** off.
- Release the valve in front of the Dome.
- Remove the safety shield and lift the dome and place it down at the back.
- Warning - the carbon rods and their holder will be hot.

Cleaning.

- The dome, base plate, brass plate will need periodic cleaning.
- The dome has a thin layer of hand cleaner on it. Carbon deposits on this layer.
- Lay the dome on it's side on some foam rubber sheet.
- Remove the rubber seal.
- Use wet paper towels and then dry towels to finish. There are a few patches of resistant carbon on the glass. I have been trying to remove them since 1996. Don't spend any time on these as they are probably an exotic form of carbon.
- Wipe a thin layer of hand cleaner, from the toilet, evenly over the inner glass surface.
- Refit the rubber seal
- Pumpdown and turn system off after 30min. The static vacuum will keep water away from surfaces and shorten future Pumpdown times.

Probe Restart.

- The JEOL manual will help.
- The probe starts like a car. Use the key and pumping will start.
- Operating pressure is reached when the Load/Filament gauge lights up.
- Wait a further 10 minutes and slowly start warming the filament up.
- Look for emission at the expected filament settings. If not, wait a further 10 minutes to allow the column to be fully evacuated.

Computers.

The Imageslave computer runs Win98 using an older chassis as compatible ISA slots are needed for the interface card. The password is "dave".

The iMac is configured like the lab computers upstairs. The petrology folder is there and printing works. Student logins should work.

Sample changing

This is documented. Be bold when opening the gate valve. Hesitation causes the microswitch to reset. So when the valve is finally opened air is admitted to the chamber and the sample holder

locks on. Close the gate valve. Turn the filament down and press the reset button on the pumpdown panel and wait for the system to recover. Once the Load/Filament gauge lights up the sample holder can be removed.

Filament changing

This is documented. Centring is important so spend some time on it using a stereo microscope. Basic tools are in a bag on top of the microprobe. Spare filaments are in the left hand top draw. Take care when handling and tighten well when fitting.

Faults and adjustments

- **Imaging.** The electron microprobe is 29 years old so naturally some parts do not perform as well as new. The scan generator buttons can stick sometimes. Pull out on all the buttons except the one that is pressed. Do this if a line rather than a picture appears on the viewing screen.
- **Sample rotation.** If the standards are in an unexpected position check the stage rotation is fully complete. Move by hand if necessary. If the standards are still out of position rotate to **position 1 x=16000 y=16000** and reseal the standards holder.
- **Spot Centering-1.** Use benetoite to observe the beam. Centre at high and low magnification as documented. Set the spot diameter to 20 microns. Centre with the **objective aperture adjust knobs**. Take care not to select another aperture - there are 4 and position 2 is standard.
- **Spot Centering-2** Use the **coarse condenser control to raise the beam current. The spot size increase should be concentric. If not, adjust the shift X,Y knobs to suit.**
- PHA software adjustments should not be needed in the short term.
- The **flow gas bubbler** should show one bubble every second or so. Cylinder replacement should not be required for several years. If sensitivity drops in spectrometer 1 try increasing the flow rate slightly using the fine valve on the cylinder regulator.
- **Vacuum.** Settings are marked on various gauges. Large deviations need checking.
- **Interference.** Things seem to have settled - there was a faulty fluorescent light fitting nearby. There is still an unknown event that reliably occurs at midnight which may stop automated analyses. It may be a Microsoft problem or strong electrical interference from power system switching.
- Most **usb error messages** can be overridden or at worst a computer restart may be needed. Check stage xyz and spectrometer settings are OK. Use ctrl T to set the stage to x=16000, y=16000, z=11000 and ctrl P to set the spectrometers to 10000. You can move the stage and spectrometers
- If the stage or spectrometers go out of range an alarm will sound. To stop the alarm look for any counter reading which is very small or very large. Move the appropriate control so the small numbers get larger or the large numbers get smaller. In the case of the z control head towards 11000. Once the alarm stops read the physical numbers into the software using the appropriate **x,y,z,Rotate, SP1, SP2 or SP3** panels. Multiply the spectrometer numbers by 10 and the stage numbers by 100 before you do so. eg 1000 -> 10000 on spectrometer 1 and 160->16000 for stage x axis for example. The JEOL manual defines the stage and spectrometer limits.
- The results folder is here: C:\mseds\result\
- When an analysis program is changed, automatic beam measurement is disabled. Re enable it by going to Options, Analysis Options and re check the box.

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