Basic Users Manual for Tecnai-F20 TEM

NB: This document contains my personal notes on the operating procedure of the Tecnai F20 and may be used as a rough guide for those new to the microscope. It may contain mistakes and supervisor's instructions should be followed during training and practice on this or other microscopes. It will be gratefully appreciated if you can let me know when you notice any mistakes in this document.

1. Always wear powder-free gloves when you are handling anything that will be put into the TEM (e.g. specimens, holders, negatives, negative magazines) and never touch these with the gloves that you just used in the dark room, as they may be contaminated with chemicals. It costs only a few pounds for a whole box of gloves but a thousand of pounds or even more to clean up a TEM and often the resolution will be never be as good as a clean one.
2. Keep the column valve closed unless you are certain that it is safe to do so.
3. Always check and confirm that the objective aperture has been retrieved before you insert or retrieve a specimen holder, reset the holder (even specimen position motion at large scale).
4. If you don't know about what to do on the TEM, ASK!

Pre-start your session – microscope status check

a. Check the logbook (Check logbook to see if abnormality of the microscope has been noted by the previous user. If yes, contact the staff member of EM centre to clarify before your session starts.)
b. Turn on flat panel monitor if it was off.
c. Login with your username and password allocated.
d. Start Peoui from your desktop
e. Check the microscope status:
    1) Column valve is closed ("Column Valve Closed" should be in yellow)
    2) Vacuum values (normal readings)
       Gun vacuum: 1
       Column vacuum reading: 6
       Camera chamber vacuum:
       Buffer tank:
       Buffer line:…
    3) Compustage is at home – stage position x,y,z, alpha, beta all about zero.
    4) HT is on at 200 kV.
    5) FEG emission operation: Operate is off ("Operate" button appears as gray:
       Gun lens: 3
       Extraction voltage: 2950 V
       Emission: ~35 μA
    6) Objective aperture and SA aperture are out.
    7) Magnification is M 4400

Report to supervisor on any abnormality before you start the session.

Start your own session:

a. Check the level of liquid nitrogen, it should be about ~ ¾ full of the dewar. Put the copper cold finger slowly into the liquid nitrogen and keep the viewing glass on the microscope covered. Normally LN should be refilled after every 2~3 hours and even short during the first session of the day.
b. Open the Vacuum Overview page for reference during operation.

Loading specimen onto the rods: (Wear powder-free gloves)

A few tools that you will need to load the samples onto either a single tilt holder (STH) or a double tilt holder (DTH). These include, a good quality tweezer to handle your specimen, a sharp pin to lift up the clip on the STH, a hex screw and a wrench rod to tighten/release the screw when a specimen is clamping onto (or taken off) the DTH. The hex screw and the wrench rod are normally kept in plastic tubes next to the specimen holders.

Load samples onto STH:

a. Put the sharp pin (it is normally located on loading stage) into a small hole of the STH and lift up the clamp of the holder to vertical position.
b. Load your sample on the holder and clamp it by put down the sharp pin.
c. Rotate the holder to upside down and now tap the other end of the holder to check if the sample has been secured on the holder.

Load samples onto DTH:

a. put your specimen onto the DTH and allow it sit completely inside the "sample bowl" (it is not difficult to do at all) and it is often better to keep the tiny hole on your specimen at the centre of the bowl as possible (you will find it easier to find it once it is in the TEM).
b. Then place the screw on top of your sample (make sure you know the up-down sense of this hex screw) and using the wrench rod to screw it back in. It is important to make sure that screw is all the way in (tightened with finger force though) and does not extend above the sample holder.
c. Make sure your sample is secured in the holder by the hex screw – you may turn the sample upside down and gently tap the back end of the DTH to confirm this (clearly we don't want to see your sample drop into the column).

Insert holder into TEM:

a. Take the STH or DTH out of the plastic loading stage and check carefully under optical microscope (or naked eyes if you feel confident) and make sure that there is no dirt or fibrous debris on the O-ring. Otherwise, clean it with an optical lens tissue.
b. Align small pin on holder with the white line on the Goniometer at 5 o'clock.
c. Insert the holder to the channel until stop (before this firm stop position, you shall be able to sense a gentle friction force caused by the O-ring fitting into the smallest channel).
d. The Turbo pump shall start and the red indicator on the goniometer will be on.
e. Select the right type of holder (either STH or Philips DTH) and enter it. If it is for DTH, follow the instruction to connect the beta tilting cable to the socket on

the right hand side of the goniometer and confirm it by pressing enter on the screen.

f. When the red light becomes off, gently rotate holder counter clockwise until the pin at the end of the holder becomes aligned with the slot/hole on the goniometer, where you will sense the attraction force by the vacuum in the column and gradually allow the holder to slide into the column. (Note: during the holder rotation, it is possible that the column vacuum might increase (worsen). If this is the case, slow down the rotation and often the vacuum will recover quickly and then continue with the rotation).

Basic operation:

a. Confirm that the vacuum is < 16 Log
b. Compu-stage position: all zero for x, y, z and alpha, beta.
c. Start the emission by press "Operate" (it becomes yellow) and set the Gun Lens (3), Extraction Voltage (4000 V). This will give the emission about 83~85 μA.
d. Open the column valve (V7) by clicking "Column Valve Closed", it will become gray.
e. Start with magnification at M 4400 and make sure both Obj. aperture and SAD aperture have been taken out.
f. Find the hole (thin area) – using the track ball on the right hand panel (RHP) to move the sample.
g. When the hole has been found, increase the magnification to M 13,500.
h. Focus the electron beam using the Intensity knob on the left hand panel (LHP) onto one spot and move the spot to the centre of the viewing screen using the track ball on the LHP.

Align the C2 aperture:

a. C2 aperture is the second aperture from the top. To align this aperture with the electron beam: when the electron beam is centred at the viewing screen, spread the beam until it almost covers the whole viewing screen and using the two knobs on the C2 aperture to make the illuminated area is a circle concentric with the viewing screen.

Adjust specimen eucentric height:

a. Set magnification at about 13,500.
b. Press the "Eucentric Focus" button on RHS console.
c. Start wobbler (start with a smaller angle).
d. Adjust Z position (+ and – button) until the image movement is minimized. Normally the Z position value should be with in the range -100 μm ~ 100 μm.

Diffraction pattern:

a. Choose the area of interest and move it to the centre of the viewing screen using the track ball on the RHP.
b. Insert the SAD aperture and rotate its outmost knob to choose the right size of the aperture.
c. Centre the SAD aperture.

d. Press the "Diffraction" button to get a diffraction from the area selected by this SAD aperture.
e. Choose the right camera length using the magnification knob.
f. Shift the diffraction pattern using the Multi-function knobs so that the central directly transmitted spot is at the centre of the view screen.
g. Focus the diffraction using the "Focus" knob (use binoculars and the small screen if you find useful).
h. Go to "camera" menu, choose "Manual" for exposure time control and use ~ 1s.
i. Cover the viewing glass.
j. Press the "exposure" button on the LHP to take the diffraction pattern. (Stop any movement/action during the exposure).

Bright Field Imaging:

a. Choose the area of interest and move it to the centre of the viewing screen using the track ball on the RHP.
b. Acquire an SAD aperture as above.
c. Insert the Objective aperture and rotate its outmost knob to choose the right size of the aperture, so that only the central directly transmitted spot is included (you may include a few spots next to it, but the contrast will be lower)
d. Centre the Obj. aperture.
e. Retrieve the SAD aperture.
f. De-select the "Diffraction" button to get an image of the area.
g. Choose the right magnification using the magnification knob.
h. Focus the image using the "Focus" knob (use binoculars and the small screen if you find useful).
i. Go to "camera" menu, choose "auto" for exposure time control.
j. Cover the viewing glass.
k. Press the "exposure" button on the LHP to take the diffraction pattern. (Stop any movement/action during the exposure).

Finish a session or Change sample:

a. Retrieve the Obj. and SAD apertures.
b. Set the magnification to M 4400 and spread the electron beam using the "intensity" knob on the LHP so that the whole view screen is illuminated.
c. Close the column valve by clicking "Column Valve Close" – it will become yellow.
d. Turn off the FEG emission by clicking "Operate" – it will become gray.
e. Reset the holder so that all position values become zero, alpha and beta become zero too.

Retrieve sample holder from TEM:

a. Make sure column valve is closed ("Column Valve Closed" should be in yellow)
b. Pull specimen holder straight out until reaches a stop position.
c. Turn the holder clockwise until reaches a stop position (your right hand may have a break once you've started the rotation).
d. Pull the holder straight out.

e. If you are finishing a session rather than changing specimen, you need to put the STH into the column (following the normal specimen holder insertion procedure) and turn the Turbo pump off once the specimen holder is completely inserted into the column and the vacuum is fine.

Changing films

a. Make sure the column valve is closed ("Column Valve Closed" should be in yellow).
b. Switch off the High Tension ("High tension" should be in gray).
c. Press "camera air" on the vacuum page, and confirm by selecting the "√".
d. Open the blue valve on the N_2 gas attached. (You may also wish to make sure that the gas bottle is not empty and the flow rate is normal)
e. When the camera chamber pressure approaches the ambient pressure, you will be able to life the camera chamber cover which is located at the back of the TEM column (normally you can hear a little sound from the camera chamber if you listen carefully).
f. Lift the negative magazines out and change the negatives in the dark room following instruction.
g. When the refilled magazines are ready, put them back into the camera chamber.
h. Clean the sealing rubber ring on the cover with a piece of optical lens tissue or clean glove and put the cover back (check the position of the cover to make sure it is right).
i. Press "camera air" button to vacuum the camera chamber and selecting the "√" to confirm.
j. Close the N_2 gas valve.
k. Wait until the "Camera Chamber Vacuum" gets down to 58 Log leave the working page.

When you completely finish the session:

a. Clean the bench and make sure everything is exactly the same as when you started the session. (The same for the dark room cleaning).
b. Close the software Peoui and answer "yes" or "No" to choose save the setting or not.
c. Log off your session (NOT shut down!)
d. Turn off microscope monitor