FEI Titan (S)TEM Operation Manual

Before and after starting a session, check:

Good column vacuum (< 20 log, <10^{-7} Torr)
Good nitrogen level (> 10%)
Close the column valve before sample holder insertion/removal
Reset the stage before sample holder insertion/removal

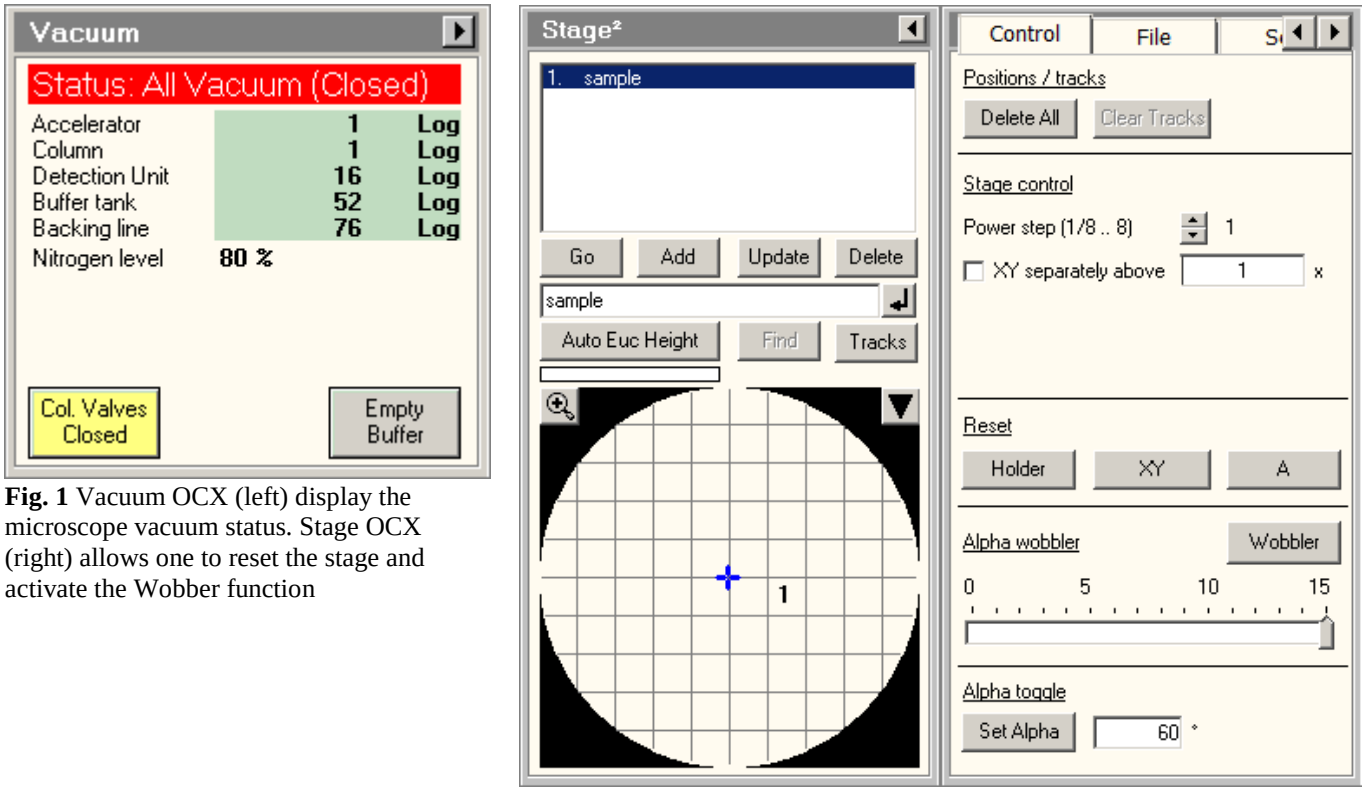

Fig. 1 Vacuum OCX (left) display the microscope vacuum status. Stage OCX (right) allows one to reset the stage and activate the Wobber function

Sample Holder

Wear gloves when handling a sample holder to prevent contamination. Never touch the brass part of the holder.
There are three types of sample holder available for different application:

1. Single-tilt: morphology observation, EDS acquisition
2. Double-tilt: morphology observation, detailed crystalline material study, EDS acquisition
3. Tomography: similar to single-tilt holder but with lower holder profile allowing high alpha tilt angle

Holder Insertion/Removal

Check the microscope status on the system display located on the right of the access door. Holder insertion/removal can be carried only when the microscope is "Available"

1. Align and insert the holder straight in to the stage load lock area
2. Wait for the load lock to pump down (3 mins)
3. Rotate the holder and fully insert it to the microscope column area
4. Reverse the above steps to remove the holder

Microscope User Interface

The microscope operation interface consists of three programs:

1. TEM User Interface: general microscope operation and the display of live TEM image (Flucam)
2. TEM Imaging & Analysis (TIA): image acquisition and analysis

3. Esprit: EDS acquisition and analysis (Bruker spectrometer system)

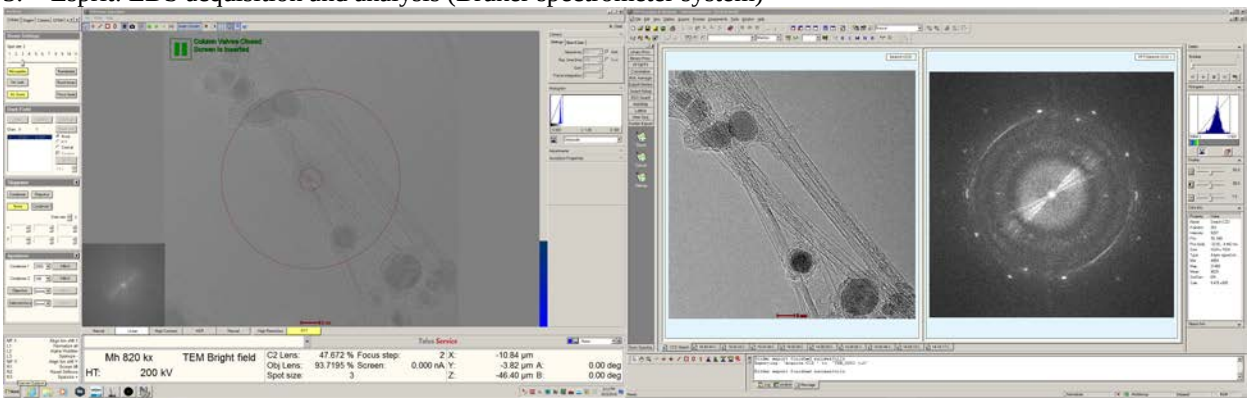

Fig. 2 Overview of the microscope user interface, TEM User Interface on the left and TIA on the right

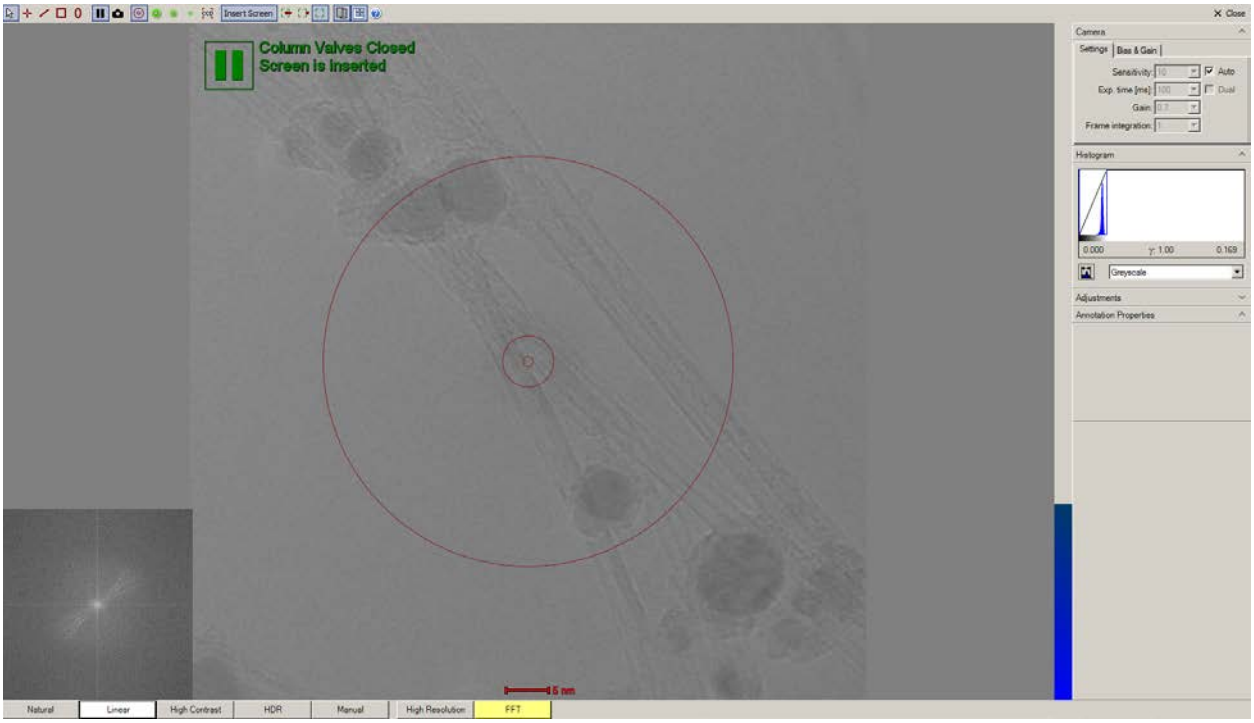

Fig. 3 Flucam displays the live image projected on the phosphor screen

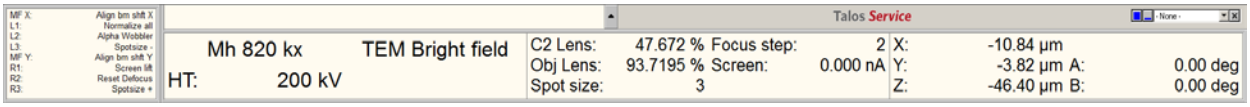

Fig. 4 Microscope status panel and function assignments of the physical buttons on the controller

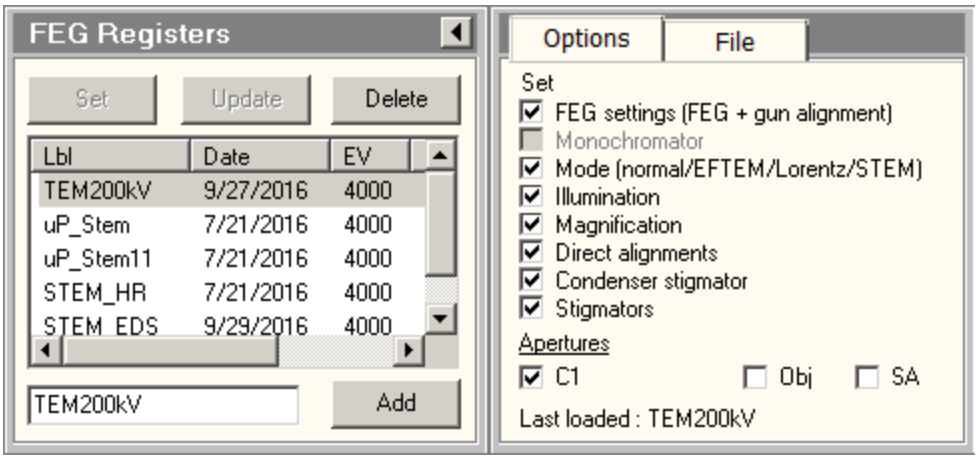

Fig. 5 FEG Register OCX, set the microscope in a desired mode

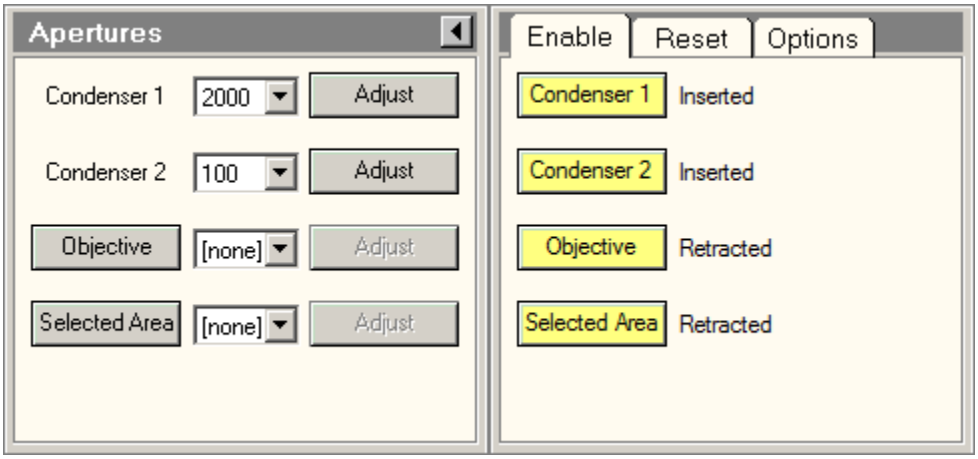

Fig. 6 Aperture OCX, useful for TEM imaging and selective area diffraction work

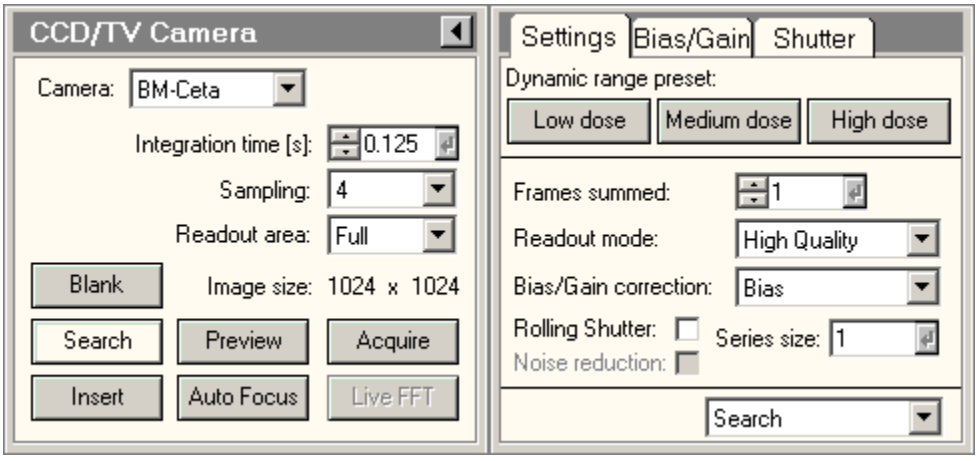

Fig. 7 CCD/TV Camera OCX for image acquisition

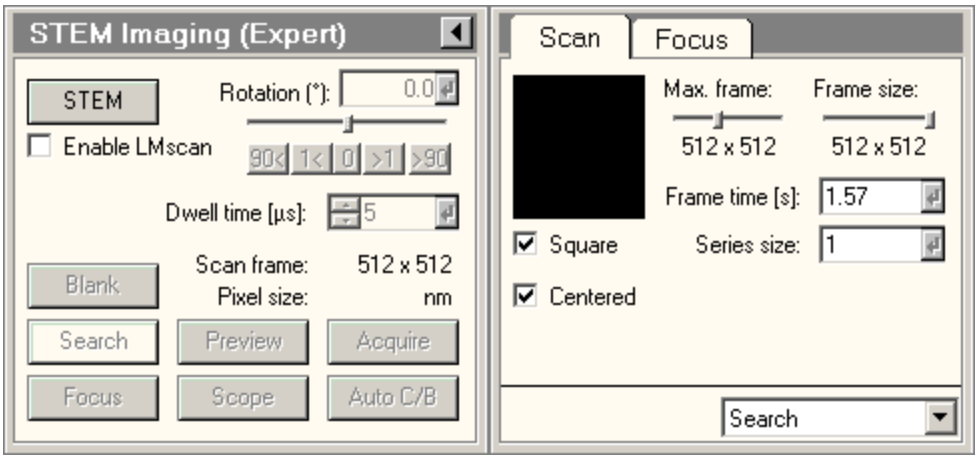

Fig. 8 STEM Imaging OCX, for STEM image acquisition

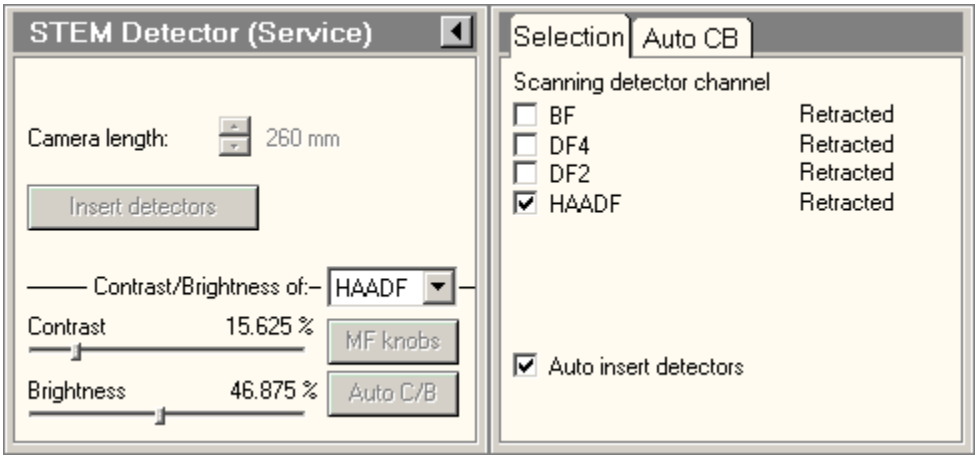

Fig. 9 STEM Detector OCX, for detector selection. Note that BF, DF4, and DF2 detectors work only with the screen is retracted as they are located under the screen.

Eucentric Position

In order to get the optimal microscope performance and reliable diffraction results one needs to set the sample at the eucentric height. To set the eucentric Z height:

1. At some low magnification (~1,000x) and find a reference object
2. Activate the stage "Wobbler" and observe object movement
3. Reduce the movement by moving the Z up or down (for regular grid with single-tile holder the eucentric Z position is around -40 um; with double-tilt holder the eucentric Z is around -120um)
4. Iteration steps 1 – 3 at higher magnification (~50,000x) to fine tune the z position

TEM Operation

Users need to go through a five-minute alignment procedure to optimize the performance while the microscope is routinely maintained by IAC staff. Follow the items on "Direct Alignment" panel:

1. Set the microscope in TEM mode by loading TEM200kV on FEG Registers
2. Make sure the sample is on its eucentric height and click on Eucentric Focus button
3. Beam tilt pp X and Y
   - Set the microscope at some moderate magnification (>50,000x)
   - Converge the beam and use MF-X and -Y knobs to make the beams overlap
4. Rotation Center
   - Set the microscope at some moderate magnification (>50,000x)
   - Expand the beam to illuminate the entire screen area
   - Minimize the image movement by turning MF-X and -Y knobs

5. Coma-free Pivot Point X and Y
   - Same procedure as in Beam tilt pp X and Y
6. Coma-free Alignment X and Y
   - Turn on FFT and defocus the microscope until ring patterns are visible
   - Use MF-X knob to make the ring patterns concentric
7. Enjoy your TEM session (fine tune Obj Stig and Focus as needed)

STEM Operation

Users need to go through a five-minute alignment procedure to optimize the performance while the microscope is routinely maintained by IAC staff. Follow the items on "Direct Alignment" panel:

1. Set the microscope in STEM mode by loading appropriate STEM setting on FEG Registers
2. Make sure the sample is on its eucentric height and click on Eucentric Focus button
3. On STEM Imaging OCX, select an appropriate camera length (~205mm) and start Search
4. Move the stage to vacuum area and set the probe parking position to the frame center
5. On STEM Imaging OCX, stop Search
6. Escape from the diffraction mode to carry out the probe alignment
7. Beam Shift
   - Use MF-X and -Y knobs to center the probe, decrease the magnification if the probe is not visible
8. Intensity focus
   - At some moderate magnification (390,000x) use the focus knob to focus the probe
9. Beam tilt pp X and Y
   - Use MF-X and -Y knobs to make the probes overlap
10. Rotation Center (Intensity)
    - This step is not required for uP STEM
    - Use the Focus Step knob to freeze the probe motion
    - Use MF-X and -Y to center the hot spot within the halo
11. Enjoy your STEM session (fine tune Condenser Stig and Focus as needed)

Data Saving and Exporting

- Save the data on the support PC (network attached Z drive)
- It is recommended to save the images in FEI's raw format (.emi) as the format contains information about the experimental condition (magnification, camera length, voltage, etc.)
- Use TIA Folder Export function to batch convert the files to .jpg, .tif, etc.
  - Use "Settings" to set up the export parameters (Source and Target folder path, image type, scale bar, etc)
  - Click "Export" to execute

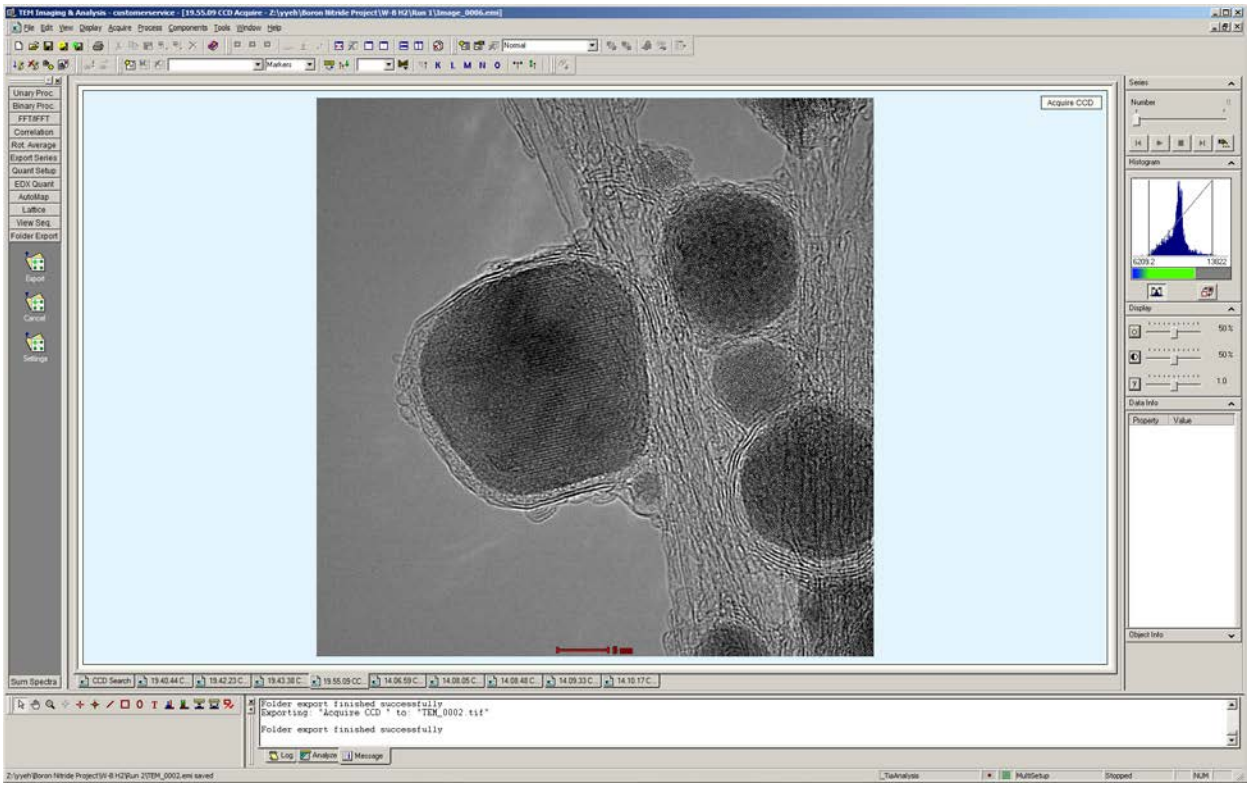

Fig. 10 TIA user interface

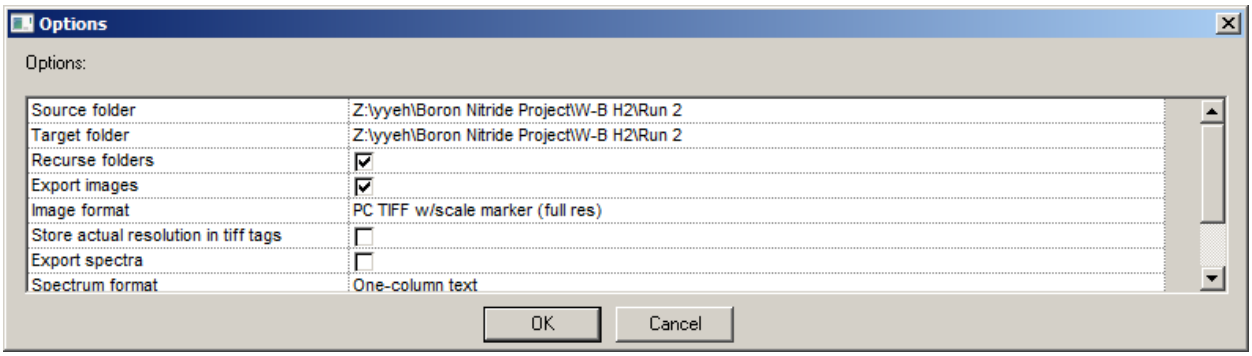

Fig. 11 Setting options under Folder Export

Emergency Information:

Medical Emergencies: Contact 911 and Public Safety (609) 258-1000
Room / facility emergencies: Contact Public Safety (609) 258-1000
Issues related to the instrument:

1. Contact IAC Staff.
2. Leave system as is, Do Not disable vacuum system.
3. Try to shut off the High Tension/Close Vacuum valve.

Audible/Siren Emergency Alerts:
Follow previous steps 2 & 3 and leave the building.

Emergency Contact Information:

Nan Yao: Office (609)258-6394; Cell (908) 922-2236 Email: nyao@princeton.edu
John Schreiber: Office (609)258-0034; Cell (215) 431-4670 Email: js51@princeton.edu
Paul Shao: Office (609)258-3851; Cell (847) 721-086 Email: pshao@princeton.edu