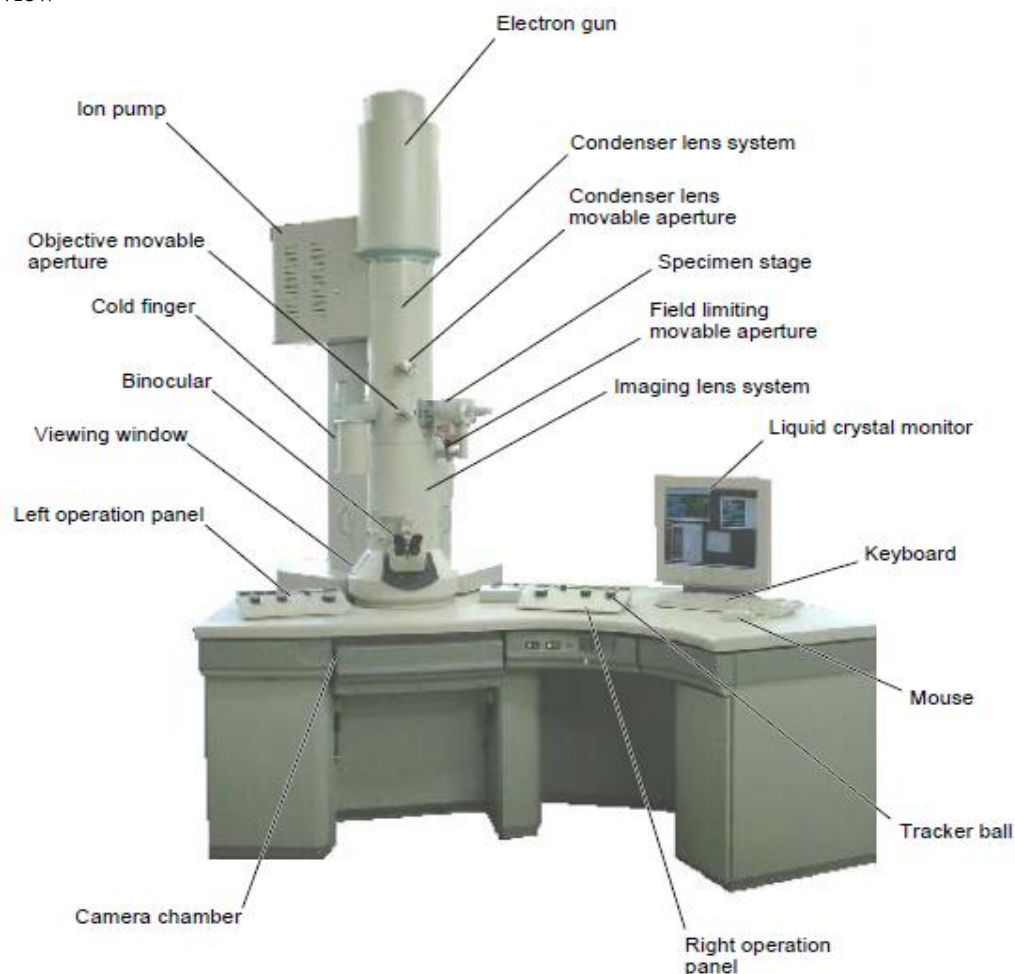


Basic Operation Guide of HITACHI H-9500 Environmental Transmission Electron Microscope

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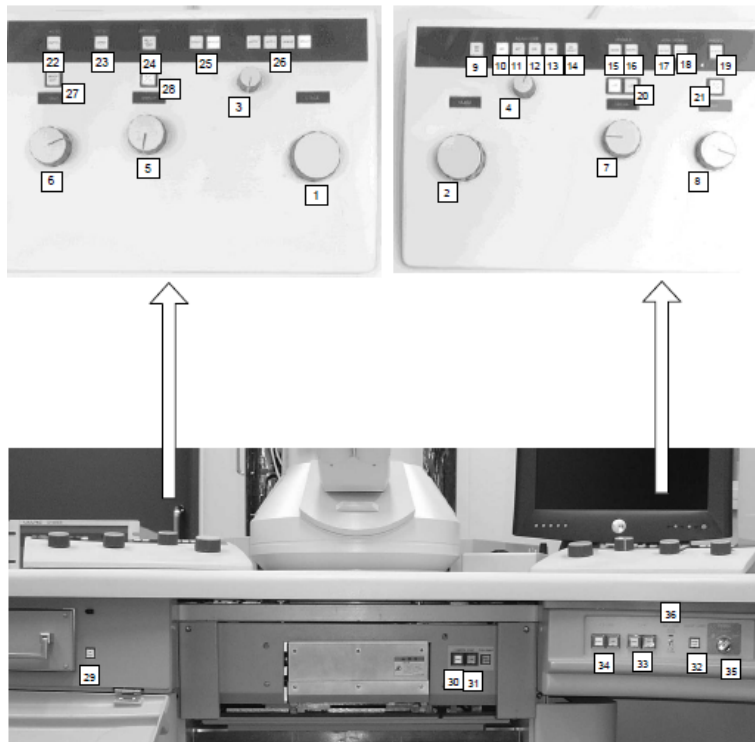
I. Overview



I-1. Specifications:

Item	Specification/Configuration	Notes
Resolution	0.102 nm lattice; 0.180 point	
Max specimen tilt angles	+/- 15°	Specimen holder dependent
Acceleration Voltage	300 keV; 200 keV; 100 keV	300 kV standard
Magnification	Zoom1: x1,000 – x1,500,000 Zoom2 (SA): x4,000 – x500,000 Low Mag: x200 – x500	
Camera Length (SAED)	0.25 – 3.0 m (5 steps)	Rotation corrected
Spot size	Micro: 0.05 – 0.2 μ m (4 steps) Nano: 1 – 10 nm (4 steps)	
Condenser Lens Aperture	10, 30, 50, 100 μ m	
Objective Length Aperture	10, 30, 70, 150 μ m	
Selective Area Aperture (ED)	20, 50, 100, 250 μ m	
Probe size	Micron 1-4 = 50 – 200 nm Nano 1-4 = 1 nm, 2 nm, 4 nm, 10 nm	

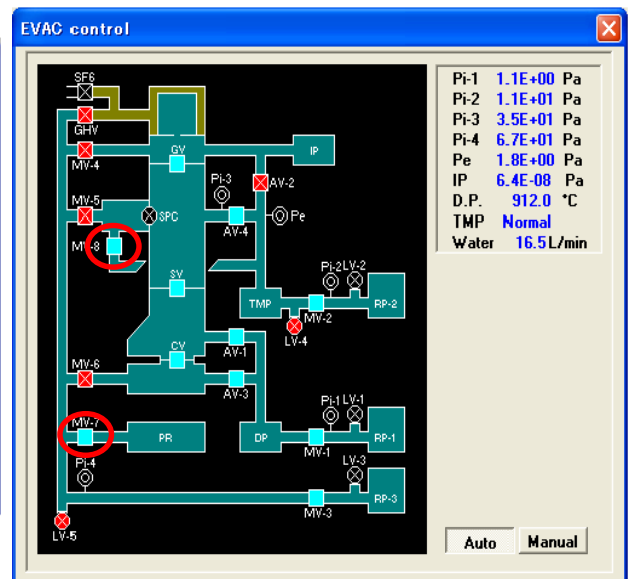
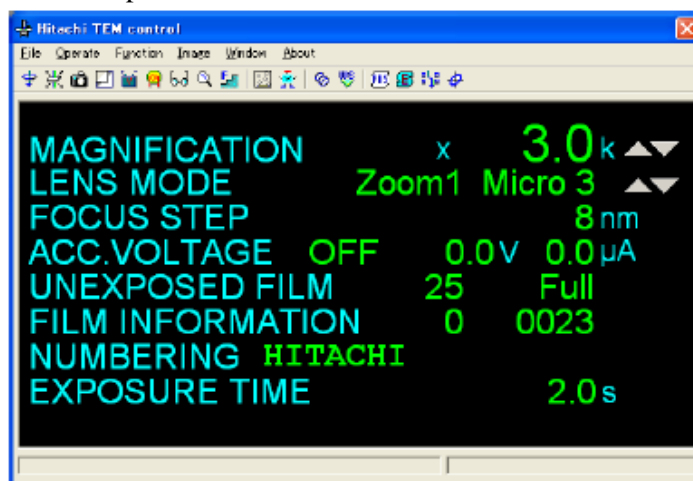
I-2. Operation Panel Layout



Knob/Switch Indication	Purpose or Function
(1) STAGE (left operation panel)	Specimen motion in Y direction
(2) STAGE (right operation panel)	Specimen motion in X direction
(3) Multi-knob (left operation panel)	Beam axis alignment and its correction
(4) Multi-knob (right operation panel)	Beam axis alignment and its correction
(5) BRIGHT	Brightness variable
(6) MAG	Magnification changeover
(7) FOCUS	Focusing
(8) DIFF	Focusing in DIFF mode
(9) BD PS	Setting of BD data (BD PRESET)
(10) BH	Setting of beam shift (BH)
(11) BT	Setting of beam shift (BT)
(12) CS	Setting of condenser lens stigmator
(13) OS	Setting of objective stigmator
(14) BD RESET	Loading of set BD stigmator value
(15) WOB.	Focusing and astigmatism correction
(16) MODU.	Adjustment of voltage center (current center)
(17) Option 1	
(18) IMAGE SHIFT	Setting of image shift
(19) PHOTO	Image recording switch
(20) x10, x20	Setting of focus variation step
(21) F/C	FINE/COARSE selection for DIFF knob
(22) HV	Automatic application of accelerating voltage
(23) LENS	Resetting of lens data
(24) Option 2	
(25) WHOLE, SPOT	Screen up/down
(26) ZOOM, ZOOM, DIFF, LOW MAG	Lens mode selection
(27) Option 3	
(28) F/C	FINE/COARSE selection for BRIGHT knob
(29) PR EVAC	Pre-evacuation chamber EVAC/AIR selection
(30) CAMERA EVAC	Camera chamber EVAC/AIR selection
(31) FULL/HALF	Film FULL/HALF size selection
(32) ROOM LAMP	Lighting of room lamp
(33) GUN EVAC	Gun EVAC/AIR selection
(34) COLUMN EVAC	Column EVAC/AIR selection
(35) POWER	Key switch for power supply
(36) GV LOCK	Opening/closing of GV

II. Basic Operations

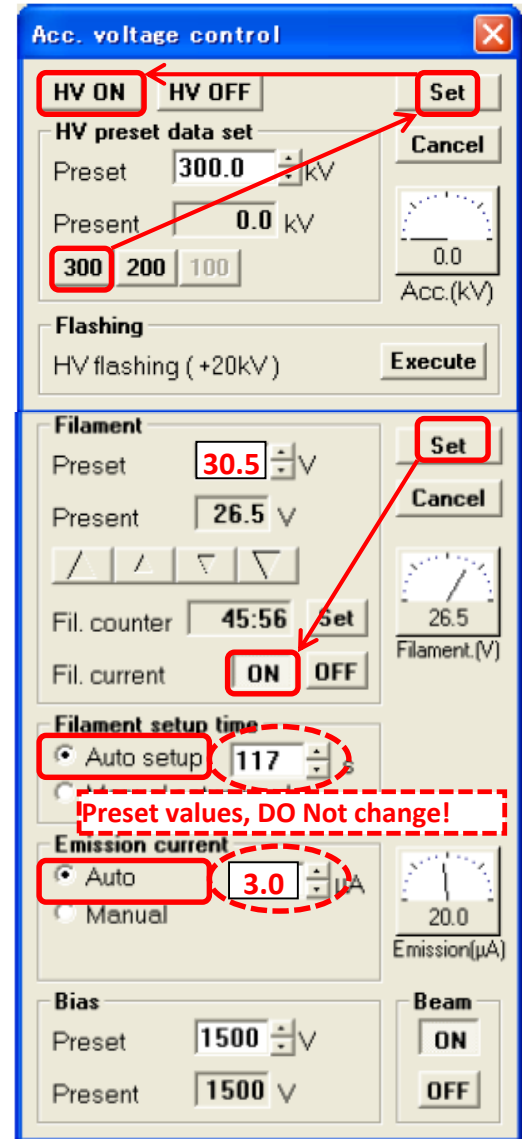
- 1) Check the POWER status of the system: make sure the POWER key switch is at **COL. ON** position and the **GV LOCK** (gate valve lock) switch is at **CLOSE** position.
- 2) Check vacuum readings for **Ion Pump**, **Column**, and **Specimen Chamber** are in the 10^{-5} range.
- 3) Check the main **Hitachi TEM Control** window as shown for any error messages. Proceed to next step if no error message is shown; otherwise, report to NFCF staff for troubleshooting and/or repair.



- 4) Remove specimen holder from the goniometer:
 - i. Pull the specimen holder till it stops;

- ii. Turn the holder **CLOCKWISE** to its stop (15 degree);
- iii. Pull holder till its second stop;
- iv. Turn holder **CONTERCLOCKWISE** to its third stop;
- v. Toggle the **EVAC/AIR** switch from **EVAC** to **AIR**;
- vi. Wait for the specimen chamber open to atmosphere (valves: MV-8 and MV-7);
- vii. Slowly pull holder straight out.

- 5) Load specimen holder to the goniometer:
- i. Set the specimen holder securely on the specimen loading station;
 - ii. Use the specimen clamp handling tool to retract the clamp lock and then remove the specimen clamp from the holder;
 - iii. Load specimen on the holder;
 - iv. Replace the specimen clamp and lock it with the handling tool;
 - v. Insert the specimen holder into the goniometer with the guide pin aligned correctly;
 - vi. While pushing the specimen holder to its first stop position, toggle the EVAC switch from **AIR** to **EVAC**;
 - vii. Wait until the alert sound of **EVAC** ready;
 - viii. Turn specimen holder **CLOCKWISE** to its stop;
 - ix. The specimen holder will be pulled in due to the negative pressure – Do not take off your hand in this step!!
 - x. Turn specimen holder **CONTERCLOCKWISE** to its stop and slowly insert it to the last stop.
 - xi. From the menu bar, click **Operate** → **Stage** to open the **Stage Control** window.
 - xii. From the **Stage Control** menu, click **Holder** → **Holder** and select the specimen holder being used.
 - xiii. Wait until the Specimen Chamber vacuum is better than 10^{-4} Pa.

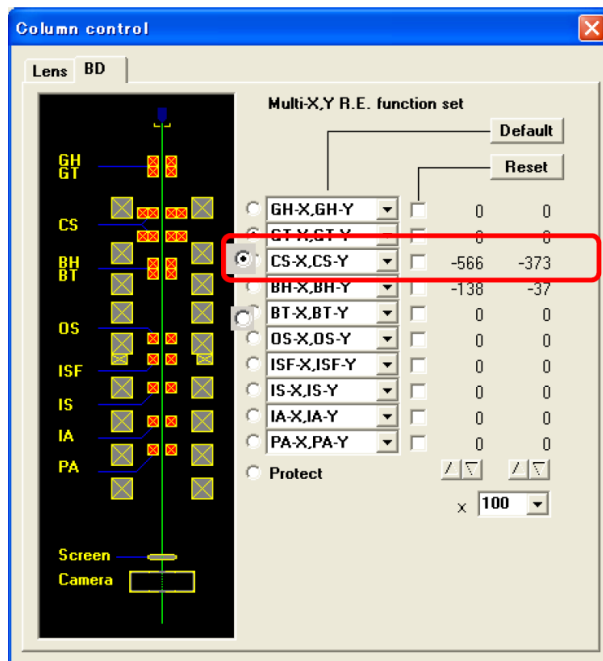


- 6) Apply Accelerating Voltage (if not being set)
- i. From the menu bar, click **Operate** – **HV Control** – **ACC. Voltage** to open the acceleration voltage control window.
 - ii. Click the reference voltage button (e.g. 300) you prefer to use, and then click **Set** button to apply the HV setting, after that click **HV ON** button to turn on HV.
 - iii. Once the HV reaches the preset voltage, from the menu bar, click **Operate** -> **HV control** – **Filament/Bias** to open the Filament/Bias control window;
 - iv. On the **Filament/Bias** control panel choose **Auto setup** for **Filament setup time** and **Auto** for **Emission current**, click **Set** and then **ON** button to turn on the Filament emission. **DO NOT CHANGE THE PRESET VALUES!!!**
 - v. Turn the **GV LOCK** switch to **AUTO (OPEN)** – to open the gate valve and generate an beam to illuminate specimen and further down the viewing screen;

III. Daily Column Alignment Procedure (BD window, column from top to bottom)

Before doing alignment, press “**Lens Preset**” on the left hand panel to reset the objective lens current to the optimum level. Magnification is reset to 2kx and bright beam should appear on fluorescent screen. Increase magnification to 10-20kx. Adjust brightness, and center **Condenser lens aperture**.

- Condenser lens alignment (Micro 1 = smallest spot size, Micro 4 = largest.** For general use of the H-9500 (imaging, diffraction,...), use Micro 2 or 3)
 - Center the condenser lens aperture.
 - From the menu bar, click **Operate** → **Observation** → **Column Mode**:
 - Click **Micro 1** (Spot Size 1, smallest)
 - Focus the beam
 - Center beam using **BH** Multifunction Knobs.
 - Click **Micro 2**, repeat above step ii and iii.
 - Click **Micro 3**, repeat above step ii and iii.
 - Click **Micro 4** (Spot Size 4, largest), check **GH** (BD in Column control window) → Focus the beam → Center the beam using Multifunction Knobs,
 - Repeat the above steps till changing spot size will not shift the beam spot away from the screen center.
 - Condenser lens stigmatism correction:
 - Choose the spot size to be used, for example Micro 2,
 - Push the **CS** button on the right operation panel
 - Use Multifunction Knobs to make the beam spot circular in shape.
 - Push **BH** button to return to normal operation.
- Eucentric height (z-position) of the specimen**
 - Push **Lens Preset** button on the left hand panel to reset the objective lens current to the optimum (default current). Increase magnification to 20,000×.
 - Please make sure the z-control is not locked when making z-change.
 - Focus the image by adjusting **z-control** on the specimen holder control unit
 - can use image wobbler to help the judge of the focus if necessary: from the Hitachi TEM control menu, click **Function** → **Wobbler condition** to open the Wobbler condition window;
 - select Wobbler and click **Set** to accept the wobbler condition;
 - to start wobbler, click **ON** from this window or push the **Wob. MODULE** button on the right control panel.
- Voltage center alignment**
 - Center a feature of the specimen image at 5,000-10,000× to viewing screen center.
 - Center the condenser lens aperture.
 - Center the focused beam using **BH** Multifunction Knobs, and then spread the beam.
 - From the menu bar, Click **Function** → **Modulation Condition** to open the Modulation condition window.
 - Check **HV Modulation**, and then click **ON** button to start HV modulation; alternatively push the **BT** button on the right control panel.
 - Use the *Multifunction Knobs* to make no shaking for the specimen image.



- vii. Turn OFF HV modulation; Switch back to **BH** control, and use Multifunction Knob to center the focused beam again.
- viii. Repeat the above iv-vi steps for 2-3 times.

4. Intermediate lens stigmation correction

- i. Find an empty area without specimen image, Push **Diff** button on the left hand panel to enter the diffraction mode.
- ii. Expand the beam illumination using the **Brightness** Knob (over-focus the knob) on the left operation panel.
- iii. Defocus (counter clockwise) the diffraction pattern using the **Diff Focus** Knob on the right hand panel to get *Caustic image* in the center of the direct spot.
- iv. From the menu bar, click **Function**→**Column control** and select **BD Control**
- v. Click **“IS”**
- vi. Get a perfect caustic image (Benz image) by adjusting the Multifunction Knobs.
- vii. Focus the diffraction pattern using the **Diff Focus** Knob.
 - If the central transmission spot (000) is not at the screen center, center it by clicking **“PA”** in the **BD window**, and using the Multifunction Knobs.
 - Align (000) spot for all camera lengths using **“PA”** in **BD control** on the Column control window.
- viii. Push the **ZOOM1** button on the left hand panel to go back to image mode.

5. Objective lens stigmation correction (*Optional at low mag.*)

Method 1: Using Fresnel fringes (Coarse astigmatism correction)

Get a hole edge image→Over focus or under focus the image to get Fresnel fringes at the edge of the hole→Push the **“OS”** button on the right hand panel→Adjust the Multifunction Knobs to make the Fresnel fringes to be uniform along the hole edge.

Method 2: Using the wobbler (Stigmator mode)

- i. Click **Function**→**Wobbler Conditions** to open the window
- ii. Choose the **Stigmator** button, and click **“Set”** button, then click **“ON”** button to turn on wobbler.
- iii. Focus the image. If no objective lens astigmatism, the image stands still. If there is astigmatism, the image will rotate along with the beam.
- iv. Push the **“OS”** button on the right front panel→Adjust the Multifunction Knobs and the Focus Knob to make the image still.

Method 3: Objective lens stigmation correction, Fine astigmatism correction

- v. Find an amorphous film→Go to a TEM magnification to be used for HREM application (100,000× or higher)→Get live image on CCD camera.
- vi. Get live FFT of the image.
- vii. Push the **“OS”** button on the right hand panel→Adjust the Multifunction Knobs to make the halo in the FFT pattern perfectly circle. (note, image focus knob needs also to be adjusted in this process)
- viii. If large area amorphous film is not available for FFT, use the amorphous films on the edge of the specimen, and adjust the **“OS”** Multifunction Knobs to make the amorphous feature clear and free of contrast at focus.

 Up to here, the daily column alignment is completed.

- From TEM control window, click **Function** → **PDS condition** to open the **PDS** window
 - Click an empty line in the popped up window and type in a file name, and push **Enter** key
 - Click **Backup** to save the parameters.
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IV. Image recording on Gatan CCD camera

- i. Start the Digital Micrograph Suite by clicking the DM icon in the taskbar on the left monitor.
- ii. Push the **WHOLE SCREEN** button on the H-9500 left control panel to raise the viewing screen
- iii. From the **Camera View** panel in Digital Micrograph, click **Start View** button to start live view of image (click Yes if popup window asks you “Insert Orius SC1000A 1 camera?”).
 - Camera setup: **Search Mode**, always 0.06s (= 14 frames/s at full size 4x bin, 1002x668 pixels of Orius CCD)
- iv. From the **Camera Acquire** window, click **Start Acquire** to record image.
 - **Record Mode**: can do Auto Exposure by selecting *Auto* from the *Exposure Mode* drop-down menu. If choose “Optimize, faster but reduce exposure time. Can also choose User, then need to input exposure time (usually 1-2s)
- Image format: always save in Gatan dm3 format first. After experiment, can convert all images to other format (JPEG, TIF etc)
 - Click **File**, → **Batch Convert**
 - Browse to find the Folder name
 - Go to Save Display as, choose desired file format
 - Click OK to convert the images to the file format you select.
- v. After you finish image recording, push **WHOLE SCREEN** again to return the fluorescent screen.

V. Selected-area electron diffraction (SAED):



- Press **Zoom 1** button,
- Go to a desired magnification, find the interested area, focus and shift it to the central area of the fluorescent screen
- Insert a selected area aperture (usually #2, #3)
- Widely spread the beam by turning the **Brightness** knob clockwise
- Press the **Diff** button
 - Can change the camera length using the magnification knob on the right hand panel. For recording on CCD, use 250mm
- Focus the diffraction pattern using the **Diff Focus Knob** on the right hand panel.
 - If the (000) spot is not at the screen center, center it by clicking “PA” in the **BD** window, and using the **Multifunction Knobs**
 - From the Hitachi TEM control window, click the **Beam stopper** icon to insert the beam stopper to cover the (000) spot. Or if the beam stopper is not desired, adjust the Brightness knob, and if necessary, lower the beam emission, until the (000) spot is very weak, barely recognizable by eyes
- Record the selected-area diffraction pattern by CCD with short exposure time, e.g. 0.2 second.
- Immediately lower the **WHOLE SCREEN** after the SAED pattern recording.
Note: One can do SAED either in Zoom mode or in SA (Zoom 2) mode by pushing corresponding button on the left operation panel.

In SA mode, one can select the area more exactly for doing SAED by focusing the edge of the selected-area aperture using **Diffraction focus knobs**, which means SAED comes from the exact area observed inside the SA aperture. However, the magnification range for the Zoom 2 (SA) mode is limited to 4000× to 500k×.

In the Zoom mode, the selected area is a little bit different from the real area but the difference is very small. Therefore usually we use ZOOM mode even when doing SAED if the area selection accuracy is not a concern.

VI. Elemental Analysis using QUANTAX ESPRIT EDS

1. Start **Esprit** program by double clicking the Esprit icon on the PC desktop
2. Log in to the program using default user name and password (in case needed, User: edx; Password: edx)
3. Use the icon in the bottom left corner of the Microscope configurator to open the **MICROSCOPE CONFIGURATION** dialog and check the settings;

- a. If configuration parameters do not match with that of the Hitachi H-9500 TEM, report to NFCF staff.
4. Click the first icon  in the bottom left corner of the EDS configurator, check the **EDS CONFIGURATION** dialog:
 - a. Select **Pulse throughput** (*lower throughput → higher resolution*) and **Maximum energy**
 - b. Check Detector temperature (should be a green number of ~ -25°C)
5. Click the second icon in the bottom left corner of the **EDS** configurator to open the **DETECTOR POSITION** window and click **OK** to move the detector to acquisition position;
6. Click the icon in the bottom left corner of the **Sample** configurator to open the **SAMPLE PROERTIES** dialog and type in sample name and description of your choices.
7. Select the Spectra workspace from the lower left of the QUANTAX window.
8. Click the **Preview** button to start a live spectrum view; if count rate (ICR) is good, then click **Preview** again to stop the live spectrum view.
9. Click the icon  on the **Acquire** button to set:
 - Acquisition parameters: if quantification is the goal, >120 second of **Live time** is recommended;
 - Automatic quantification: None
 - Cyclic acquisition:
 - Spectrum numbering:
10. Click **Acquire** button to start acquisition of an EDS spectrum.
11. Start quantification by clicking the Quantify button:
 - a. If quantification is set to automatic, the quantification results appear in the spectrum list;
 - b. If quantification is set to interactive, the **QUANTIFICATION** dialog pops up:
 - i. Use the Elements tab to identify elements;
 - ii. Click **OK** to close the **QUANTIFICATION** dialog – the results will be displayed in the spectrum list.
12. Once your EDS work is done retract the EDS detector by clicking the second icon in the bottom left corner of the **EDS** configurator and **OK**.

VII. After the session's work is done

- 1) Stop the camera view;
- 2) Close the GV by toggle the **GV LOCK** switch from **AUTO (OPEN)** to **CLOSE**;
- 3) Remove the specimen holder and unload specimen;
- 4) Replace the single-tilt specimen holder in the goniometer to protect the vacuum of specimen chamber;
- 5) Keep **HV** and **filament bias** on at **300kV** (if you operated at 200 kV or 100 kV, please return it to 300 kV for optimized operation of the microscope).