HITACHI HF2000 TEM

Operation Manual

- Alignment and Operation -

Dear users,

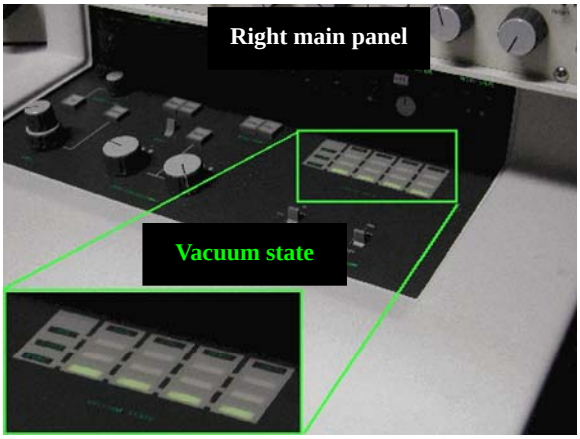
To help you have a smooth and successful TEM observation, I wrote up this basic operation manual. Systematic theoretical training plus hands-on practical training may be the best way to understand the details of this operation instruction for HF2000 TEM.

There are some notes in this instruction. Please read and follow them carefully, and please make sure you follow what I told you ask before you touch the TEM if you do not know the consequence of your action.

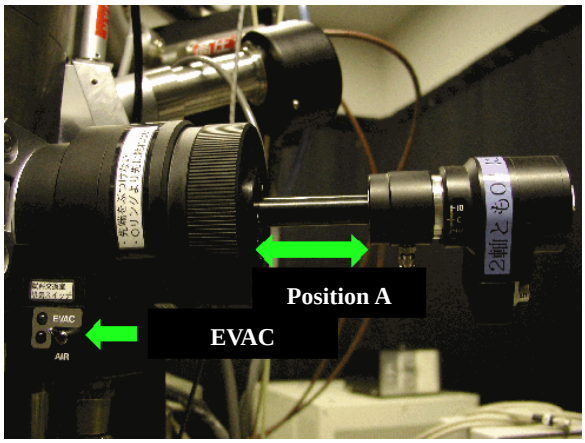
Takamichi MIYAZAKI / June 2009

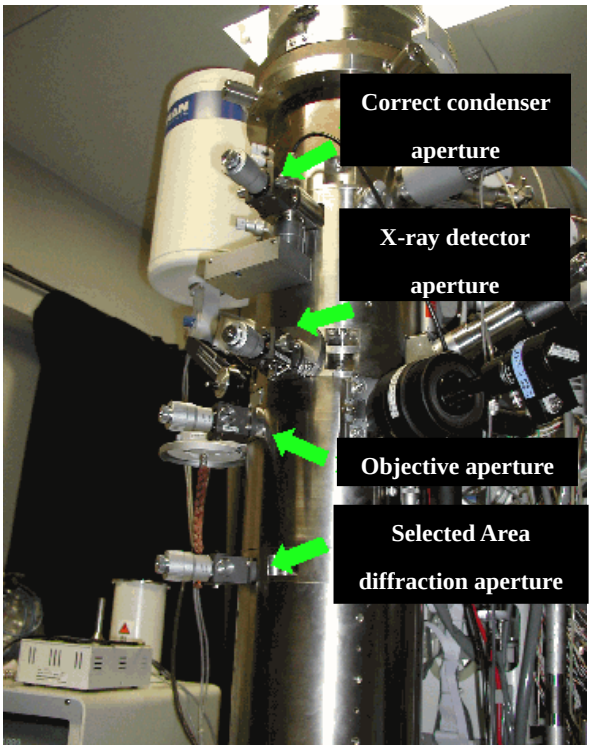
Contents

1. TEM startup



- Check the comments in the TEM log book. Enter your name, laboratory and contact information on the log book before any action on the TEM.
- Make sure that TEM vacuum is good. At the vacuum state (right panel), SPEC, COL, and CAM indicators should be VAC position (all green), and GUN indicator is at EVAC position (green flashing).
- Make sure that TEM is set at standby positions:
    a) OBJ. TEMP. MONITOR: 21^{0}C and below
    b) Objective, selected area and condenser apertures are out (position 0).
    c) GV (Gun Valve) is Closed
    d) ACC. VOLTAGE: off; HV: on at 200kV
    e) Sample position: X = 0, Y = 0; Sample tilts: 0
    f) Bright/Dark field selector set at Bright field.
    g) Microscope is at [ANA1] mode.
    h) Magnification: 1000K – 1500K
    i) Normally, the sample holder stays TEM. It is situated at position A
    j) Lens Current (C1, C2, OBJ) : not 0
    k) Unexposed film number: 50 FULL
    Film number: 1 0
- After confirmation of the standby positions, enter chiller temperature and pressure, COMPR pressure, HV tank pressure, and vacuum state on the log book.



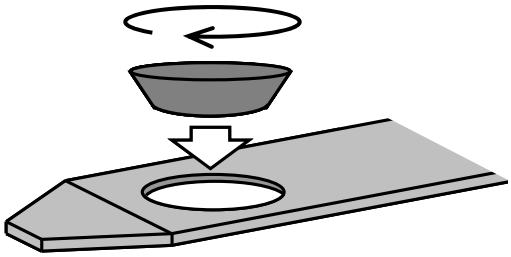


2. Set the sample (loading)

Normally, the sample holder stays in TEM. It is situated at position A (see in the following diagram).

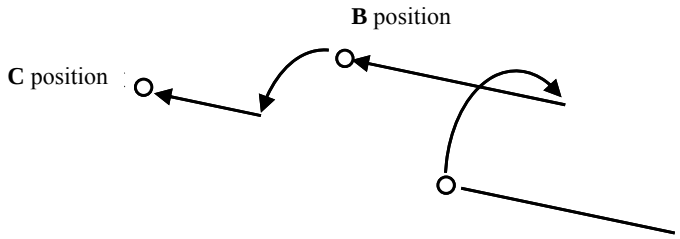
Note: Objective aperture must be out during sample holder loading / unloading.

- Turn the switch on goniometer to Air. Wait till SPEC is at Air position (red light on vacuum state) and pull the holder out.
- The sample is securely seated in the holder
- Loading sample holder into TEM

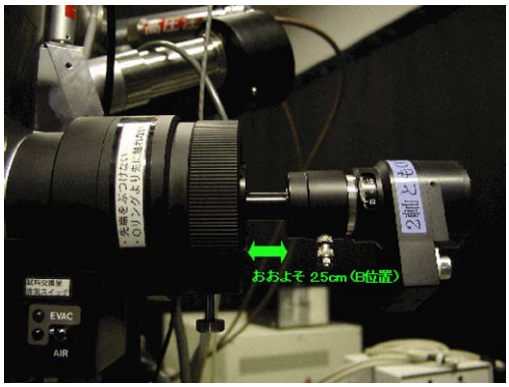


Follow the route indicated in the diagram below to load your sample holder into the TEM.

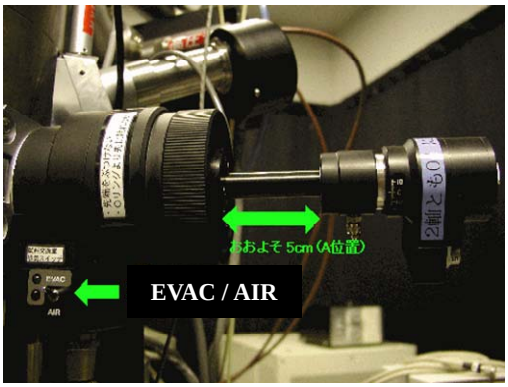
1) Insert the sample holder horizontally along and press it, then turn the switch to EVAC.
2) When electron sound rings, turn the holder clockwise till it stops.
3) Let the sample holder go into the TEM horizontally along and smoothly. The sample holder must be under control.
4) Turn the sample holder counter-clockwise till it stops
5) Let the holder go into the TEM fully.



（EVAC⇔AIR）



B position



A position

3. Applying HV

- Apply HV when vacuum is good
- Make sure that the applying HV conditions are set at following state:

V0 VOLTAGE	200kV
R RATIO	5.5
I1 CURRENT	30μA

- Press the [FE] bottom once (left panel) and press [FLASH] when it's flashing. IF current rise up, if the IF current is lower than 0.30 mA, repeatedly press [FLASH] bottom till IF current is > 0.30mA.
- After IF current exceeds 0.30mA, wait till V0 VOLTAGE rises up to 200kV. Then V1 and V2 Voltage rises gradually up to the setting value (watch the ACC VOLT on the CRT monitor).
- Enter the V1 Voltage and IF current on the log book (Yellow book).

****

When the emission current drops below 10μA, press [I1C] and recalibrate extraction voltage to raise the emission current to 30μA. The beam wander off during the process but return by the end of the I1C state. Repeat alignment procedure because the changed extraction voltage affects the caustic.

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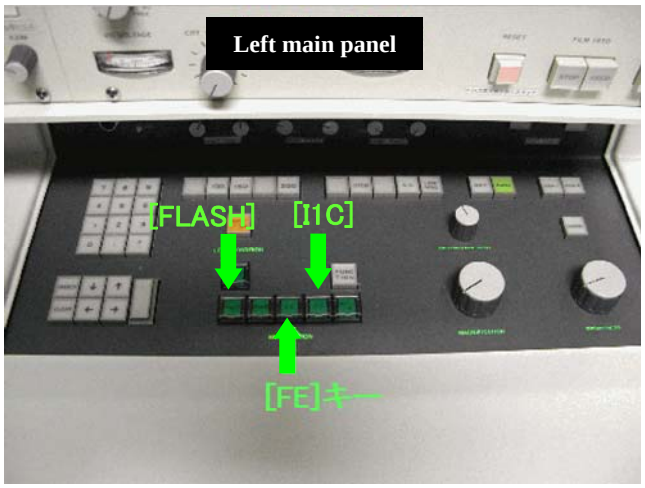


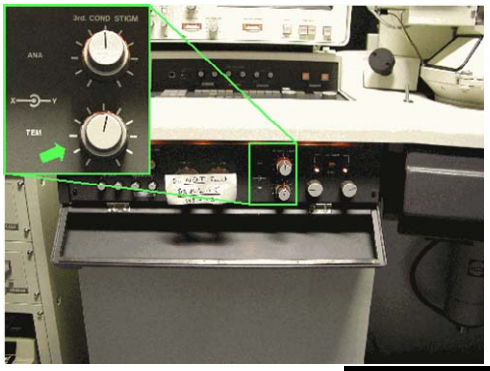
4. Prepare for beam alignment

- Press [ANA1] to change selected mode: Analysis → TEM mode.
- Reset switch (Red square button)

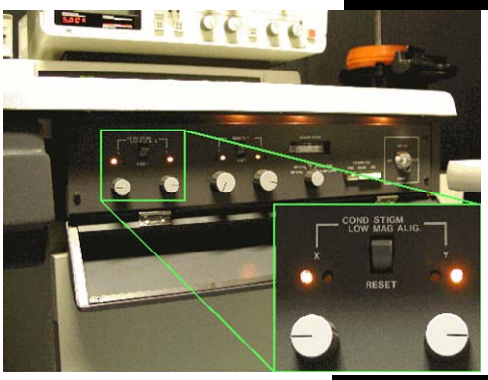
5. Correct condenser astigmatism（Magnification : 100K ~ 200K）

Be sure to use the correct condenser astigmatism for TEM mode:

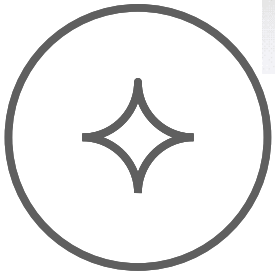
- Open the GV and make sure you hear it click open.
- Turn the Brightness control (left panel) to crossover, and center the beam with the Brightness Centering knobs (right panel).
- Using the condenser stigmator (right sub panel) make the caustic as round or axial symmetry. And using the 3rd order stigmator (left sub panel) makes the caustic as symmetry. The 3rd order correction will be slight and the controls should be close to the center positions.
- The correctly aligned caustic will appear as a circle with Star in TEM mode. The image is shown below.



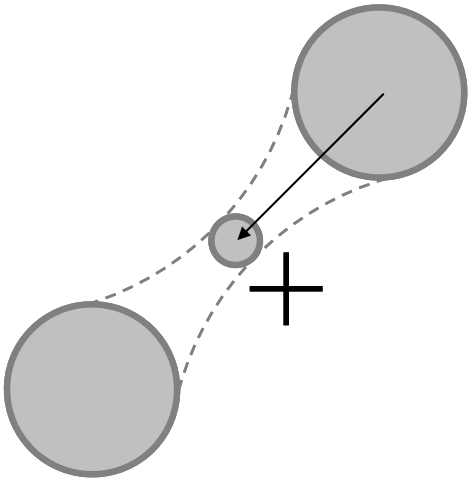
3rd Cond stigmator



2nd Cond stigmator



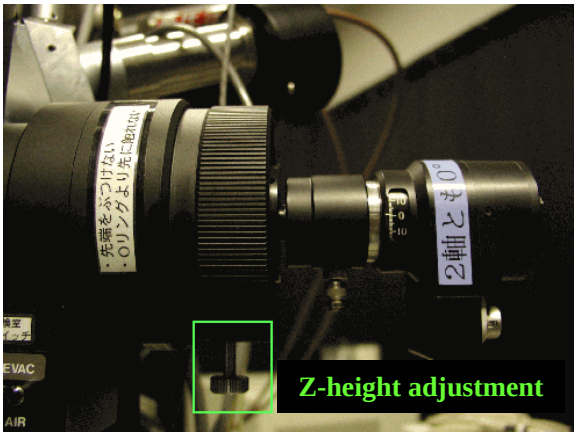
- Insert condenser aperture (First aperture) and minimize the swinging beam using the condenser X/Y adjustment screws when passing through crossover.

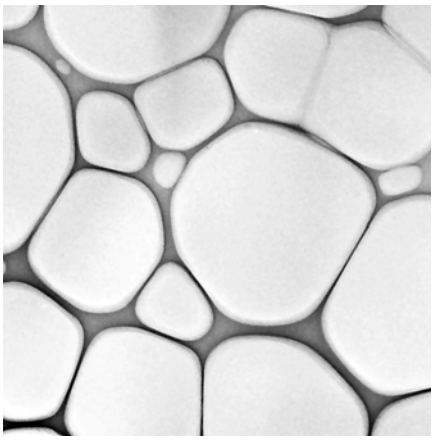


6. Z-height adjustment

- Move edge of sample and set the objective lens current to 5.72 using focus control. Focus the sample using the Z-height adjustment screw on the specimen holder entry port.

  Note: An objective lens current of 5.72 is the optimal setting for minimum spherical aberration. You may also focus sample using wobblers method or minimum contrast method for the image focus.





7. Voltage center

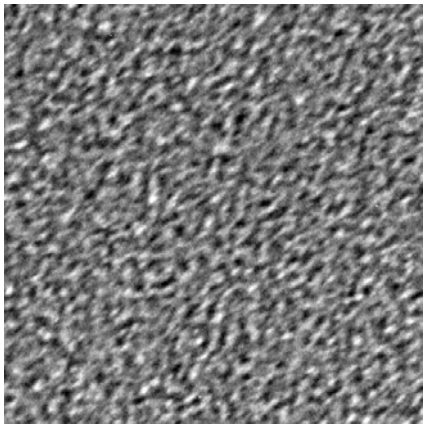
- Move edge of sample.
- Turn on [HVM] (HV MODULATION, right panel). The image feature may oscillate.
- Adjust BEAM TILT (right sub panel) to minimize the vibration of the feature.
- Turn off [HVM] (right panel).
- Repeat the alignment procedure a few times because the beam tilt and objective stigmatism correction have disturbed the condenser stigmatism corrections. In general, completing the alignment procedure twice will be sufficient.

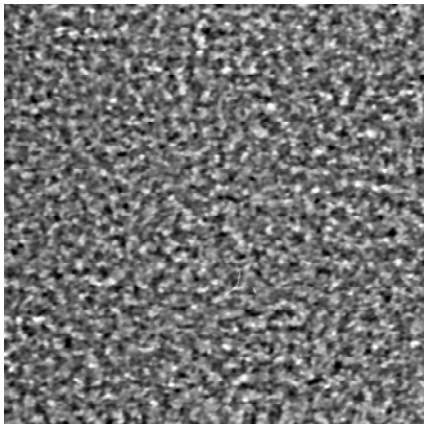
8. Astigmatism correction of objective lens

- Reset the OBJ STIGM-X and Y control knobs (stigmator) on the left sub panel.

＊ This returns setting to the stigmator to preset level.

- Set a magnification 2 or 3 times higher than used for photographing.
- Turn the focus control knob clockwise so as to obtain slightly over-focused image, and a fringe appears outside the collodion hole.
- Adjust the OBJ STIGM-X and Y control knobs so that the width of the fringe outside the collodion hole becomes uniform.
- Manipulate the focus control knob so as to approach just focus from over-focus in order to narrow the fringe width.
- Find a as granularity a amorphous as possible and adjust the OBJ STIGM-X and Y control knobs so that granularity a amorphous becomes finer granularity as shown in the figure below.





9. Image focus and Objective lens astigmatism correction
(Including Selected area electron diffraction (SAED))

This is to tell you how to correct objective lens astigmatism using amorphous film which is useful in recording a high resolution image.

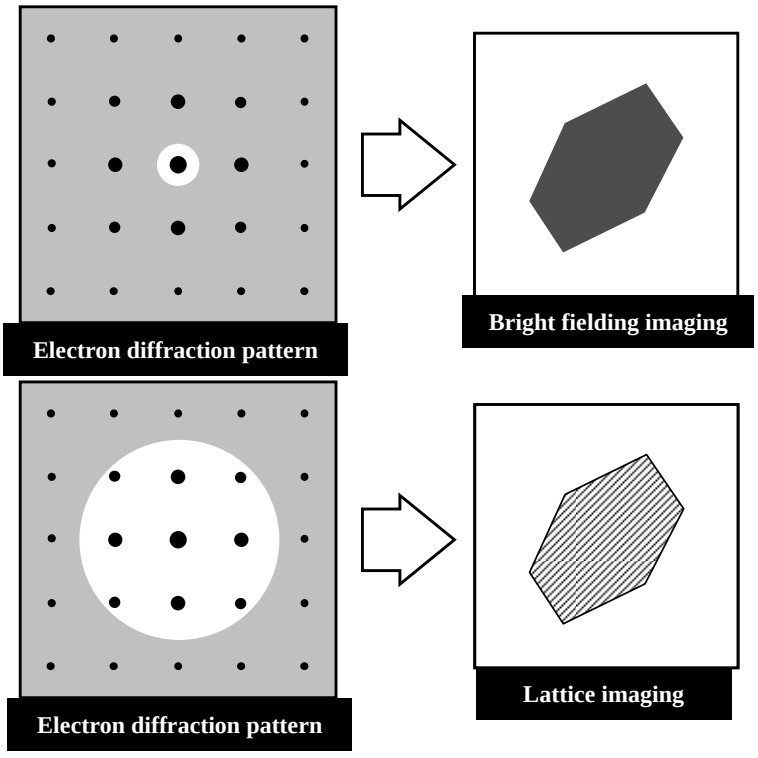
- Center a small hole / particle / the sharp edge of sample.
- Insert and center a suitable Selected Area (SA) aperture.
- Press [DIFF] (left panel) and set the camera length to 0.80m using Magnification knob. If the central spot (transmitted wave) is still disk, focus the pattern by adjusting Diffraction Spot (left panel).

  Note: Open the beam to form a parallel illumination on the sample by turning Brightness clockwise. Diffraction spots will become weaker and smaller. If the central spot is disk instead of a spot, adjusting Diffraction Spot is needed.

- Insert objective aperture into the central spot that should be centered by using the objective X/Y adjustment screws.
- Press [ZOOM] to seen a bright field image.
- Pull out SA aperture
- Correct the astigmatism by adjusting fine focus and OBJ STIG X and Y controls if necessary.
  (Using minimum contrast method and amorphous contrast)

10. Bright fielding imaging

- Find an interesting sample area.
- Focus the beam and sample to the crossover.
- Press [DIFF]. (see Sec.9, SAED)
  Note: If the central spot is not at the viewing center, center it using the INTER ALIGN X and Y knobs (left sub panel). If the spot is not round, make it round using INTER STIG X and Y knobs (left sub panel).
- Insert the objective aperture into the beam by turning the aperture handle clockwise.
- Press [ZOOM] to see a bright field image.
- Adjust fine focus and OBJ STIG if necessary.
- Adjust Brightness so that Auto exposure time is between 2-5 seconds.
- Cover the viewing window
- Press [Photo] to expose the film and record a bright field image.



11. Photographing for film

Usage of auto exposure time set mode

Time mode provides a function for automatically setting the exposure time so as to obtain optimum photographic density on a film in response to a determined brightness.

- Set the exposure time setting knob at AUTO.
- Press the PHOTO key to start film exposure.

Manual mode

- Set a desired exposure time by turning the EXPOSURE TIME control knob. The set exposure time is displayed at the bottom right on the CRT.
- Optimum exposure time lines for

  Bright field image: 1 to 4 seconds

  Dark field image: 12 to 30 seconds

  Selected area diffraction pattern: 0.5 to 1 seconds

  Nano beam diffraction pattern: 8 to 16 seconds
- Press the PHOTO key to start recording

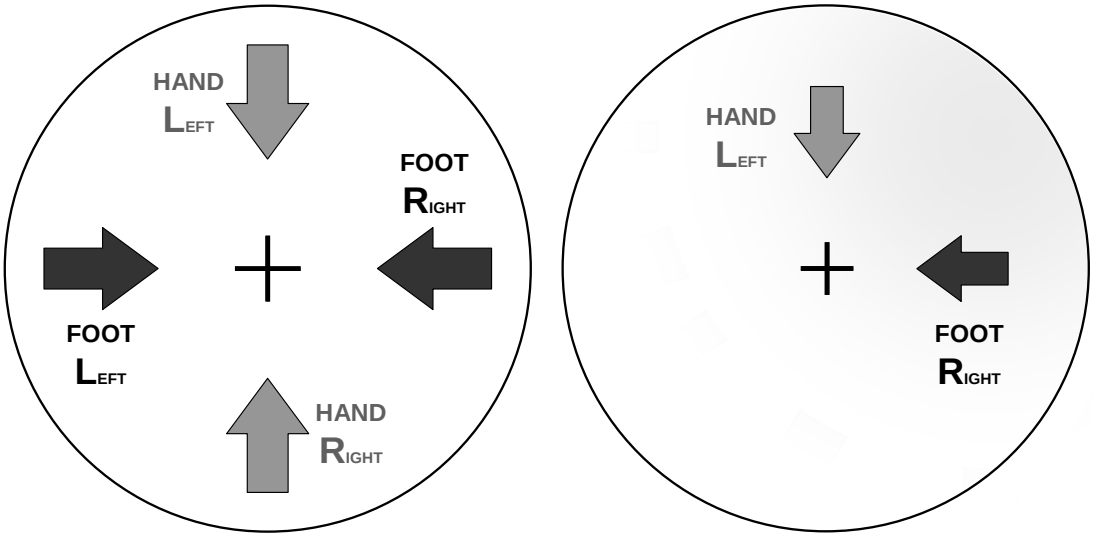
12. CCD camera

- Turn on the power to TV monitor and control.
- Open the beam by turning Brightness clockwise. Brightness will become weaker.

  Note: Do not use CCD camera for diffraction pattern, low-magnification image and direct beam.
- Cover the viewing window.
- Press [STOP] and [PHOTO].

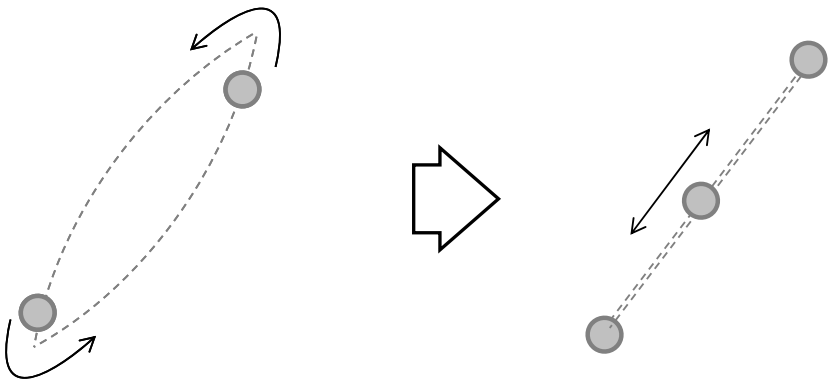
13. Sample tilt

- Press [DIFF]. (see Sec.9, SAED)
- Adjust the sample tilt with electron beam direction along the zone axis.



14. Astigmatism correction of beam tilt

- Turn the WOBBLER ANGELE control on the right main panel fully clockwise.
- Press the WOBBLER ON/OFF switch.
- When astigmatism is not corrected, rotating motion in a specific direction is observed. Adjust the [BTX] and [BTVX] controls on the left sub panel so that non-directional image rotation is observed as shown below.
- Turn the WOBBLER ANGELE control counterclockwise by 3 steps. When astigmatism is not corrected, adjust the [BTY] and [BTVY] controls on the left sub panel so that non-directional image rotation is observed as shown below.



15. High resolution imaging

- Find an interesting sample area.
- Focus the beam and sample to the crossover.
- Press [DIFF] (see Sec.9, SAED)
- Set the sample tilt along zone-axis by seeing SAED.
- Insert the largest objective aperture into the beam by turning the aperture handle clockwise.
- Press [ZOOM] to see a high resolution image.
- Turn Magnification up to 300K – 700K.
- Adjust fine focus and OBJ STIG if necessary. (see Sec.8)
- Adjust Brightness so that Auto exposure time is between 2-5 seconds.
- Cover the viewing window.
- Press [Photo] to expose the film and record a bright field image.

16. Microbeam diffraction

- Insert condenser aperture (Second aperture) and minimize the swinging beam using the condenser X/Y adjustment screws when passing through crossover.
- Sample tilt (see Sec.9, SAED)
- Change the condenser aperture to forth aperture. And minimize the swinging beam using the condenser X/Y adjustment screws when passing through crossover.
- Adjust the objective focus, and confirm to select an interesting area.
- Press [DIFF]

17. Exchange the sample (unloading)

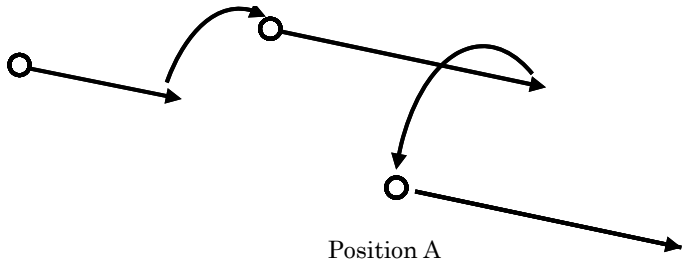
- Make sure that GV should be closed. Please remember that the unloading without closing GV cause a lot of problems, FE-tip's fatal damage in a worst-case scenario.
- Unloading sample holder from TEM

  Follow step 5 to take it out.

  2) Close GV and pull the sample holder till it stops.
  3) Turn the sample holder clockwise till it stops.
  4) Pull the sample holder till it stops.

5) Turn the sample holder counterclockwise till it stops.

6) Turn the switch on goniometer on Air. Wait till SPEC is at Air position (red light on vacuum state) and pull the sample holder out.



Position A

（EVAC → AIR）

18. TEM shutdown and film exchange (Shutdown procedure)

- Close GV
- Press [FE] twice to shutdown high voltage
- Set Objective, selected area and condenser apertures out (position 0).
- Press [ANA1], and turn Magnification up to 1000K-1500K to keep lenses warm and stable, and prevent any water vapor condensation.
- Set sample shift: X = Y =0, sample tilts = 0
- Take the sample out of the sample holder (see II, III)
  Note: Carefully unload the sample holder and set the holder A position after removing sample.

19. Film exchange

- Wear gloves.
- Turn off all lights (room light, CRT monitor, panel light, CCD monitor) except the safelight.
- Turn the switch of the desiccators down to Air position.
- After a few minutes, open the desiccators door, take out full film magazine and prepare empty receiver.
- Open the cover of the camera chamber and turn the CAMERA EVAC switch to AIR.
  Note: Air is leaked to the camera chamber. Wait for a few minutes until air leak complete. Upon completion of air leak, the AIR (red) lamp of VACUUM STATE is lit (right panel) and the buzzer sounds.
- Open the front door of the camera chamber.
- Take out the receiver. And take out the film magazine from inside the receiver. Pull out the guide rail

while raising the stopper, and the film magazine will come out.

- Load a new full film magazine into the camera chamber.
- Load an empty receiver.
- Close the front door of the camera chamber.
- Turn the CAMERA EVAC switch to EVAC. Usually after about 10 minutes. EVAC (green) lamp of VACCUM STATE lights up.
- Loading of new film
    1) Take out exposed film and the light shielding plate
    2) Place film in the magazine.
    3) After loading the films, don't forget to insert the light shielding plate
- Open the desiccator's door and introduce the full film magazine.
- Turn the knob to EVAC.
- Reset film number to 50 on the CRT using the keypad. Listen for the shielding plate to properly feed through into the film receiver.
- Change the FILM NUMBER to 0 using the keypad.

20. Final check procedure

- Turn off all lights (room light, CRT monitor, panel light).
- Complete the log book.
- Fill final check list.

HITACHI HF2000 TEM

Operation Manual

- EDX analysis in zoom mode -

1. Start up

- Adjust the beam alignment in [ZOOM] mode.
- Insert the X-ray detector.
- Insert condenser aperture (2nd aperture) and minimize the swinging beam using the condenser X/Y adjustment screws when passing through crossover.
- Remove the any objective aperture and turn off the column pressure gauge filament by pressing the "FILAMENT" switch.

  Notice: not use any objective apertures when the detector is in! The objective aperture will generate hard x-rays and burn out the detector.

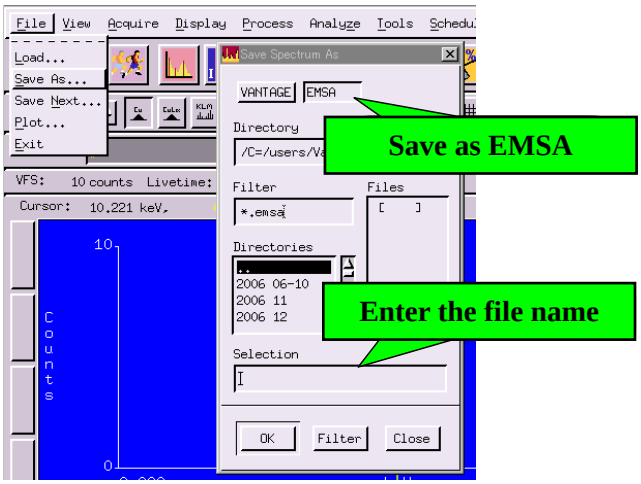
2. The EDS procedure

- Make sure to turn off the column pressure gauge and insert condenser aperture (2nd aperture).
- Turn on the power switch of the EDS workstation.

-Screen-

- Click "Spectral Display" icon.
- Select "Acquire" → "Parameter" → "Set up". Set the max. energy to 20.48 keV.
- Find region of interest and focus it properly with brightness knob.

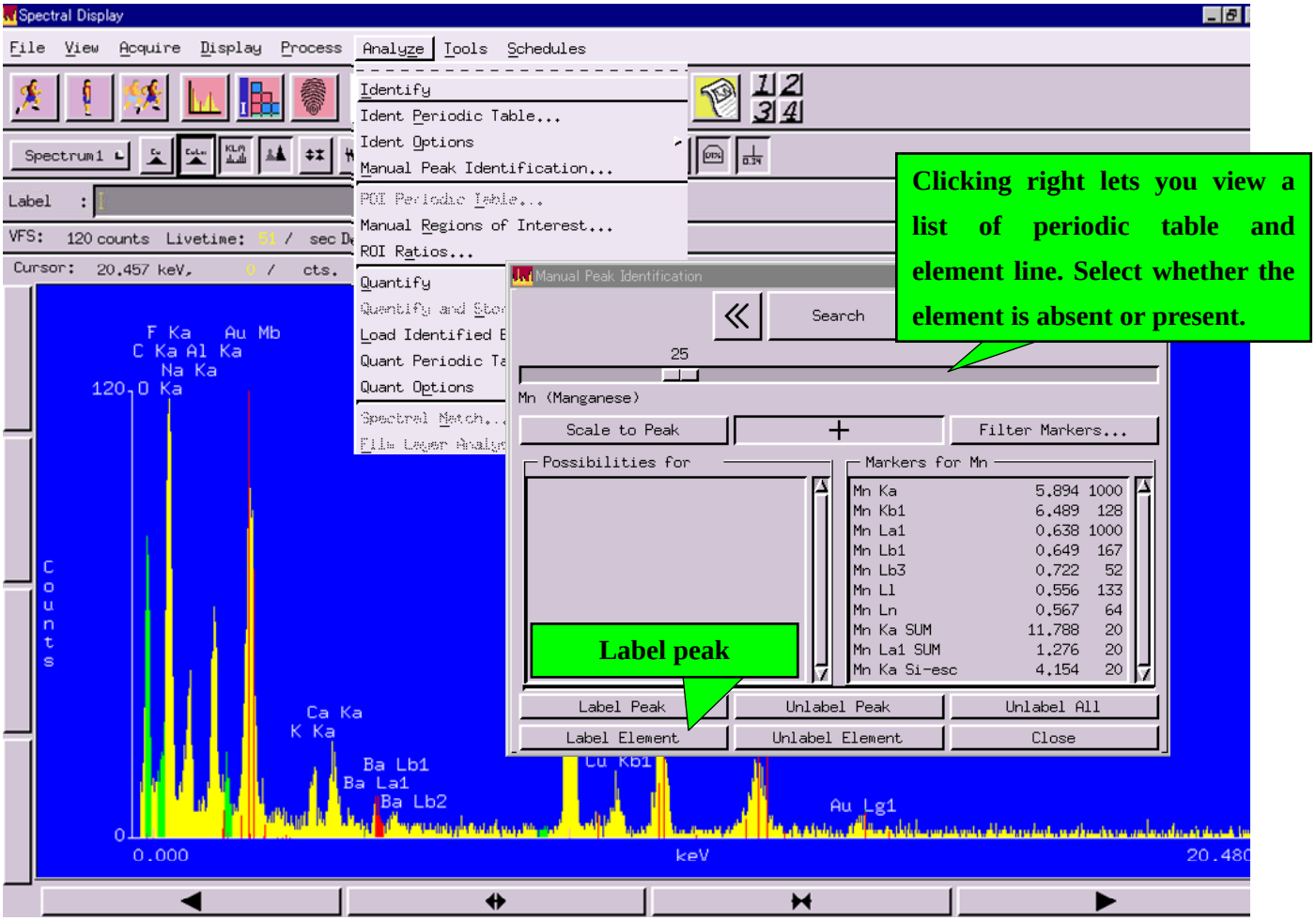
  Notice: When the dead time is too high (over 30%), select smaller condenser aperture, or acquire spectrum from thinner region.



- Depress  on the quick button.
- Depress  , when the intensity reaches certain intensity.
- Enter the file name, save the spectrum as "EMSA".

3. Peak identification (Label peak)

- Open the “Analyze” menu and select “Manual Peak identification“
- Click the expand button (+) to display the set of markers for the selected element.



4. Quantification

- Open the “Analyze” menu and select “Quant Periodic Table”.
- Choose the element and lines for quantification, and set “Correction method” to “MBTS”.

  K-ratio: Fit is performed and intensity ratios are reported.

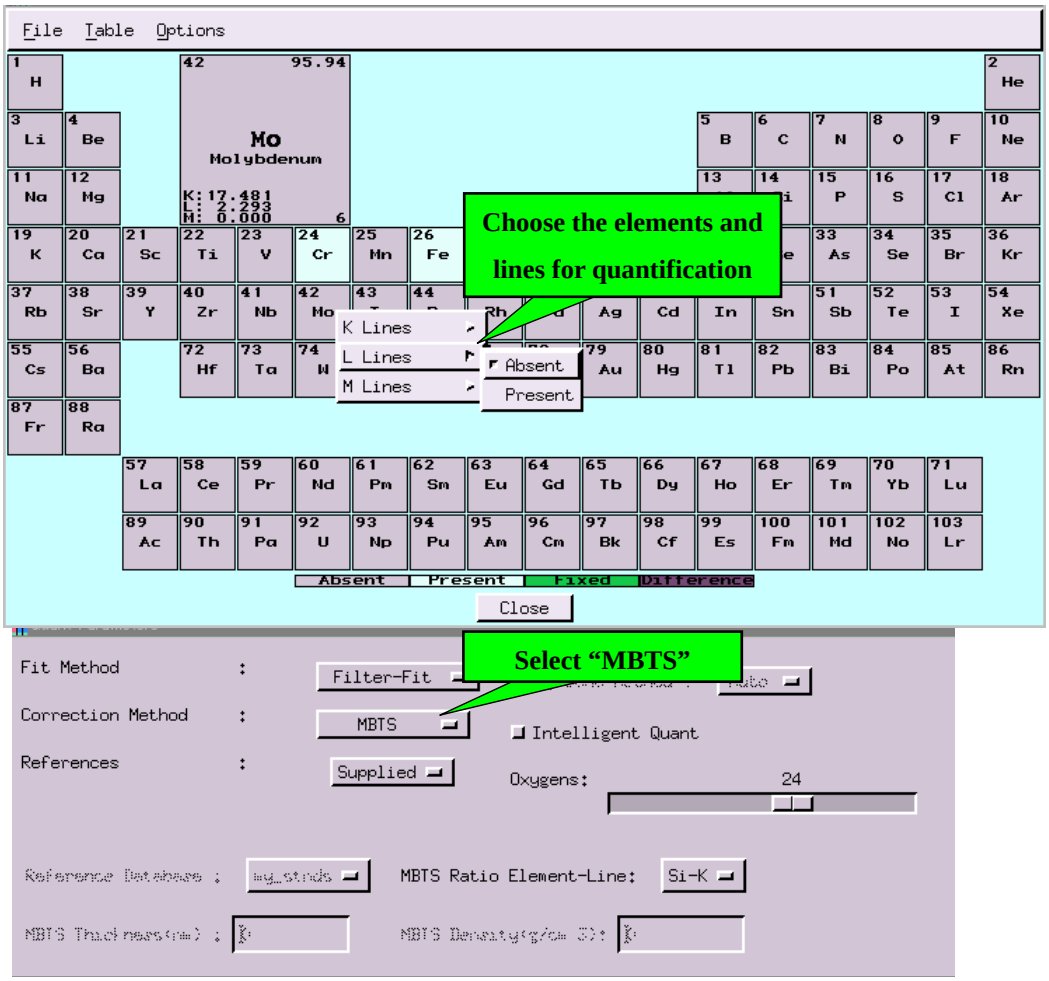
  ZAF: Adjusts the theoretical spectrum for average atomic number, absorption, and fluorescence factors, which influence spectrum differences between elements in pure and composition. Used in metallurgical SEM applications.

  Proza: Same as above. Used on SEM applications, especially for light elements in heavy matrix.

  Bence-Albee: Use in SEM applications, especially for mineral samples

  MBTS: Correction assumes that there is no absorption of x-rays within the sample. Used in TEM applications.

- Press “Quantify” button. 



5. Shut down

- Depress the “Retract” button to retract the X-ray detectors.
- Close the spectra display window.
- Shutdown the Windows software.

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Operation Manual

- Alignment in Analysis mode

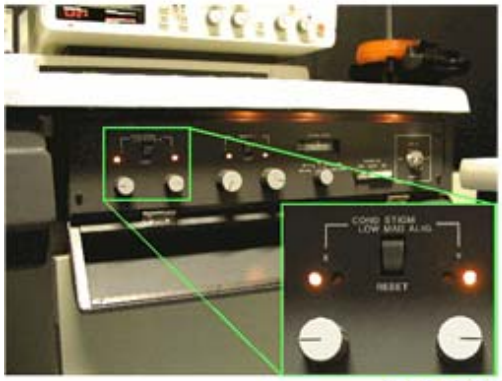
1. Start up

- Execute the beam alignments in zoom mode. (See the manual above)
- Place the beam through the hole in the sample and increase magnification to 150kX. Remove all apertures, center the beam, and go to crossover.
- Depress [ANA-1] and reset.

2. Correct condenser astigmatism （Magnification : 100K ~ 200K）

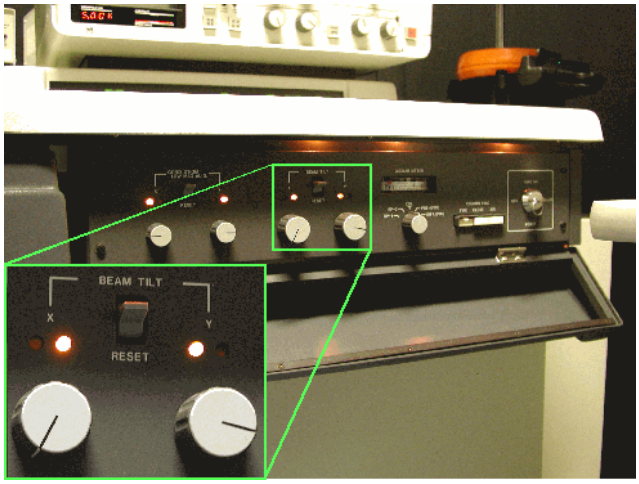
- Using the condenser stigmators (right sub panel) make the caustic as round as possible and using the 3rd order stigmators (left sub panel) make the caustic as symmetrical as possible.
- The 3rd order correction will be slight and the controls should be close to the center positions. Be sure to use the correct 3rd order stigmators for the selected mode: TEM and ANA mode.
- The correctly aligned caustic will appear as a circle with the Star of David in the center in TEM mode and it has the appearance of the Mercedes-Benz star in Analysis mode.

3rd Cond Stig



2nd Cond Stig

3. Voltage center of condenser lens



Note the direction of the caustic movement when going to crossover.

- Use the beam tilt controls (right sub panel) to shift the caustic in the same direction of the movement and brightness centering controls (right panel) to return the caustic to the center of the phosphor screen.
- Correct both 1st and 3rd order condenser astigmatism.

Repeat step 3 until caustic does not move with directionality when passing through crossover. This correction requires significant beam tilting if the previous operator used the TEM alignment procedure.

- Insert condenser aperture (3^{rd} or 4^{th}) and centering by minimizing the tails of intensity surrounding the central bright spot using by the condenser X/Y adjustment screws.

HITACHI HF2000 TEM

Operation Manual

- STEM imaging and element mapping -

1. Start up

- Move area of interest to the crosshairs on the phosphor screen and focus.
- Adjust the z-height until the sample and correct objective astigmatism using the stigmators on the left sub panel.
- Insert the X-ray detector.
- Insert condenser aperture (2nd aperture) and minimize the swinging beam using the condenser X/Y adjustment screws when passing through crossover.
- Remove the any objective aperture and turn off the column pressure gauge filament by pressing the "FILAMENT" switch.

2. Beam alignments in STEM mode（Magnification: 100K ~ 200K）

- Depress the [STEM] (left main panel).
- Insert the "D-STEM DET" on left side of column.
- Depress the [IMAGE SHIFT], centering the black circle on CRT monitor using image shift.
- Adjust the FOCUS, CONTRAST, and STIGMA.

3. The element mapping

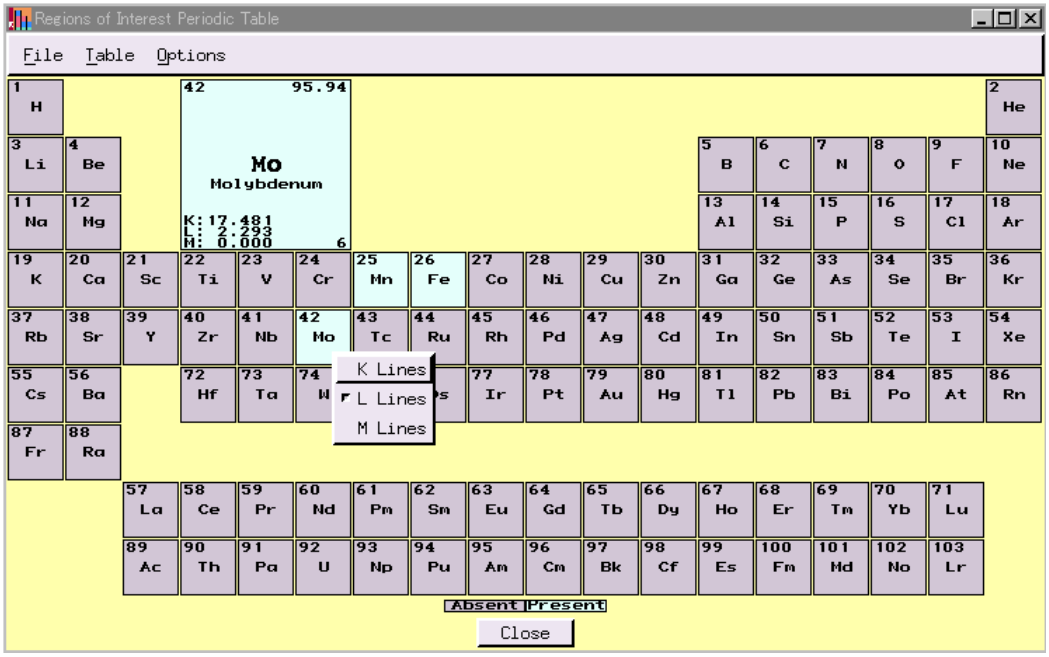
- Make sure to turn off the column pressure gauge and insert condenser aperture (2nd aperture).
- Turn on the power switch of the EDS workstation.

-Screen-

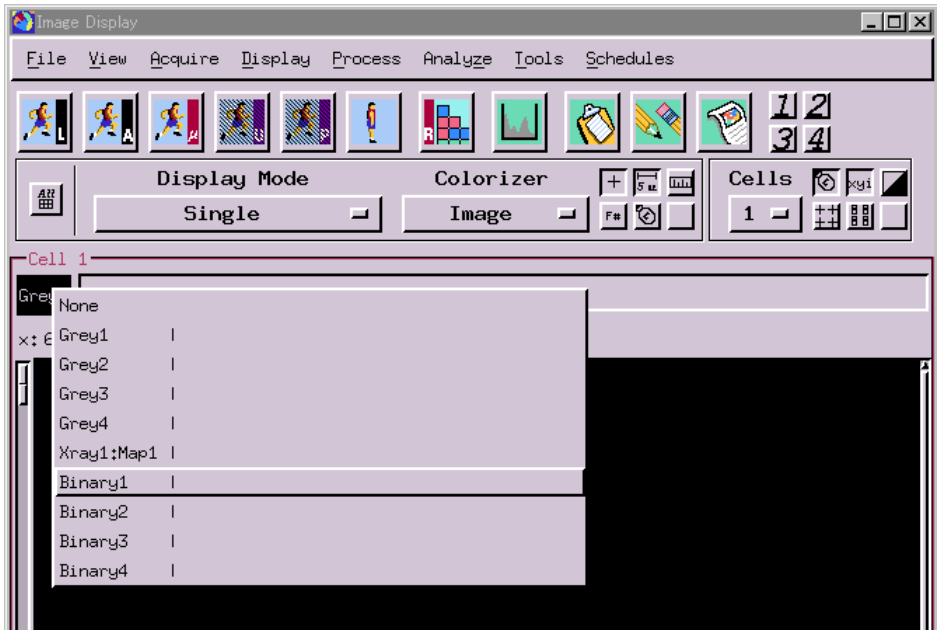
- Click "Spectral Display" icon.

- Select “Acquire” → “Parameter” → “Set up”. Set the max. energy to 20.48 keV.
- Find region of interest.

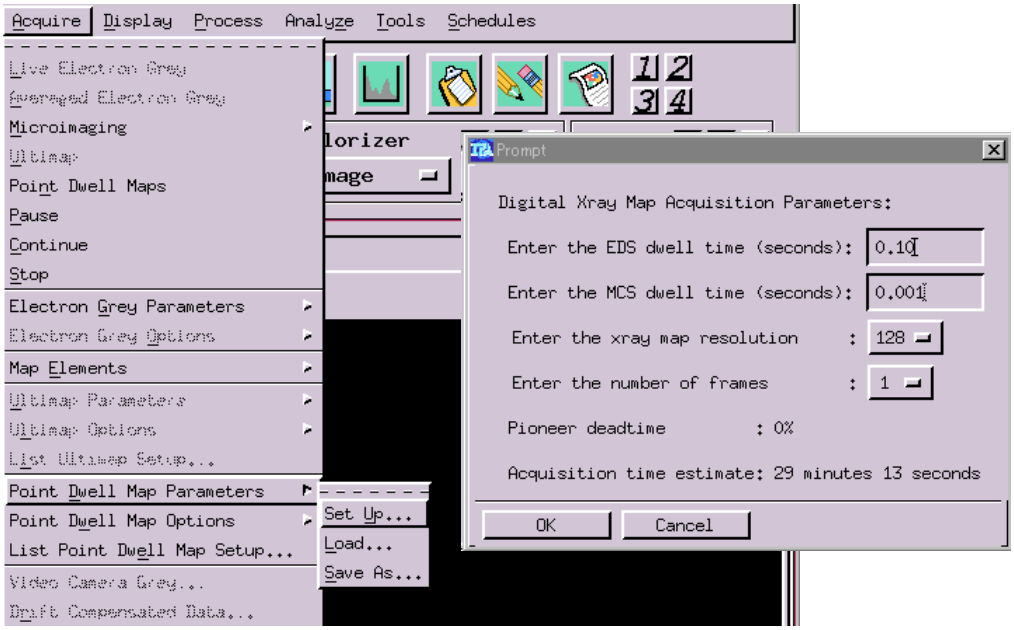
  Notice: When the dead time is too high (over 30%), select smaller condenser aperture, or acquire spectrum from thinner region.
- Open the “Analyze” menu and select “Manual Region of Interest …“
- Choose the element and lines for element mapping



- Open the “Image Display” and select [Xray1: Map1] on left cell.



- Select “Acquire” → ““Point Dwell Acquire” and then set parameter up below.



- Set [Scanning Speed] to 1. Depress  .
- Save image as tiff-format with respect to each element.