

# OPERATING INSTRUCTIONS FOR HITACHI H-7000/H-7110

(Version 2.02 - December 1994)

## PRESTART CHECKLIST

- 1) Vacuum system on
  - Turbo pump at **NORMAL** operation
  - Green Lights for **GUN, COL, and CAMERA**
- 2) Electronics on (Lower right console panel key switch to **COL ON**)
- 3) Sign in to **LOG BOOK**
  - Turn up **CRT IMAGE ADJ**
  - Press **INDEX** button to record start time
- 4) kV on and one kV selected (Press **READY/OFF** button and desired kV button)
- 5) Fill liquid nitrogen cold trap
- 6) Press **RESET**

## INITIAL SETUP

Many people use the H-7000 for a variety of techniques. If you have problems attaining a "normal" image for TEM please check this list.

- 1) **ZOOM** button depressed
- 2) **MAGNIFICATION** set to 6,000x
- 3) **TV** button Off
- 4) **FILM FEED STOP** button depressed and illuminated
- 5) **SPOT SIZE** at 5 or 6
- 6) Condenser aperture #2
- 7) Objective aperture #1 or #2, or Obj. Apt. insertion lever to the right (i.e. no aperture)
- 8) Diffraction aperture out
- 9) **WOBBLER ANGLE** Off
- 10) **BRIGHT** button depressed
- 11) **HV** and **C3/OBJ** buttons Off
- 12) Toggle **BRIGHTNESS CENTERING RESET** switch

## FILAMENT SATURATION

- 1) Remove specimen
- 2) Select desired accelerating voltage (for intermediate voltages see **INTERMEDIATE ACCELERATING VOLTAGES** below)
- 3) Press **RESET** button, center the **BRIGHTNESS** knob (based on the indicator lights)
- 4) Toggle **BRIGHTNESS CENTERING RESET** switch

- 5) Set **MAGNIFICATION** to 6,000x
- 6) Slowly turn up **FILAMENT** emission knob until illumination appears on phosphorescent screen
- 7) Adjust **BRIGHTNESS** knob until crossover / condensed spot is obtained
- 8) Use **FILAMENT** knob to obtain Wehnelt (filament) image
- 9) Center image with **BRIGHTNESS CENTERING** knobs on right panel
- 10) Increase **FILAMENT** emission until all traces of Wehnelt disappear (This is filament saturation point)
- 11) Adjust **BIAS** control until **HV/BEAM** meter is two divisions above kV setting
- 12) Spread beam

### DAILY ALIGNMENT

- Specimen out, **MAG.** at 6,000x, **ACCELERATING VOLTAGE** on
- Condenser aperture #2, all other apertures out

### BRIGHTNESS CENTERING

- 1) Use **FILAMENT** emission to obtain Wehnelt image
- 2) Select **SPOT SIZE #2** (lower left panel), and converge beam with **BRIGHTNESS** knob  
(Note: If beam sweeps across screen when converging the beam see **CONDENSER APERTURE ALIGNMENT**)
- 3) Center beam with **BRIGHTNESS CENTERING** knobs on right panel
- 4) Select **SPOT SIZE #7** and converge beam
- 5) Center beam with **GUN HORIZ.** control knobs (lower left panel)
- 6) Repeat steps 2-5 until there is no shift between **SPOT SIZE #2** and #7
- 7) Select **SPOT SIZE #4** or #5 (for general use), and center

### TILT CENTERING

- 1) Select **SPOT SIZE #4** or #5 and obtain Wehnelt image
- 2) Using **GUN TILT X/Y** knobs center Wehnelt image  
( It should look like the CBS "eye" ( ⊙ ) )
- 3) Repeat **BRIGHTNESS CENTERING**

### CONDENSER LENS STIGMATOR

- 1) Select **SPOT SIZE #4** or #5 and obtain Wehnelt image
- 2) Converge and Center the beam
- 3) Toggle **CONDENSER STIGM IMAGE SHIFT RESET** switch
- 4) Using **CONDENSER STIGM X/Y** knobs obtain sharpest Wehnelt image (Note: Correctly stigmated beam will not "twist" or "pull" when changing **BRIGHTNESS** knob)

## CONDENSER APERTURE ALIGNMENT

- 1) Converge and center the beam
- 2) Spread beam to just fill screen
- 3) Center edges of beam on edges of screen with condenser aperture X & Y adjustment knobs

## SAMPLE ROD REMOVAL AND INSERTION

- TEM sample rod should be stored fully inserted in the microscope column

- 1) **MAGNIFICATION** at 6 k, beam spread, **FILAMENT** off (fully CCW)

### OUT:

- 2) Pull rod out until it stops - approx. 2cm
- 3) Turn rod counterclockwise 10° until it stops — this is the rest position
- 4) Pull rod out until it stops - approx. 3cm
- 5) Turn rod counterclockwise 180° until it stops **Do not pull on rod!**
- 6) Toggle specimen chamber switch to **AIR**
- 7) When **RED** light appears on **SPEC** panel fully with draw rod

### IN:

- 8) Insert sample rod with alignment pin located at the notch and hold in place
- 9) Toggle specimen chamber evacuation switch to **EVAC.**, when **ORANGE** light appears on **SPEC** panel you can release rod
- 10) When **GREEN** light appears on **SPEC** within 20 seconds turn rod CW 180° and gently ease rod into column (If you wait too long the specimen light will return to the **ORANGE** light - you must toggle specimen switch to **AIR** and repeat steps 9 - 11!!!!!!)
- 11) Turn rod counterclockwise 10° until it stops and ease rod the final way into the microscope column  
🔊 **Do not allow rod to slam into the far side of the column!** 🗣️

## PHOTOGRAPHY

- 1) Locate, center, focus and stigmatize area of interest on imaging screen
- 2) Focussing screen in down position
- 3) **FILM EXPOSURE SIZE** set to **FULL** (selection knob next to camera chamber)
- 4) **DENSITY ADJ** knob set to Red index line (Green index line for **SAD**)

## ABS - AUTO BRIGHTNESS SET mode

( Analogous to Shutter Speed Priority photography)

- 1) Set **EXPOSURE TIME** knob to desired speed (generally 2-4 seconds)
- 2) Press **ABS** button. When optimum brightness level is reached the green light above the **DENSITY ADJ** is illuminated. If optimum brightness cannot be set "**CANNOT ADJUST**" will appear on the **CRT** screen, and a new exposure time must be selected.
- 3) Depress **FILM FEED** button (illuminated)
- 4) Gently lift right hand screen lever. Illumination will be shut off, film will load, film will be exposed, and illumination will be shut off again.
- 5) After exposure has been made, lower right hand screen lever
- 6) Depress **FILM FEED STOP** On.

#### **ATS - AUTO EXPOSURE TIME SET mode**

(Analogous to Aperture Priority photography)

- 1) Set desired imaging brightness on the phosphorescent screen
- 2) Set **EXPOSURE TIME** knob to **AUTO**. When optimum exposure time is determined the green light above the **DENSITY ADJ** is illuminated, and exposure time will be displayed in lower right corner of **CRT**. If optimum exposure time can not be set for current imaging brightness **DO NOT EXPOSE !** will be displayed on the **CRT** and a new brightness level must be selected.
- 3) Depress **FILM FEED** button (illuminated)
- 4) Gently lift right hand screen lever. Illumination will be shut off, film will load, film will be exposed, and illumination will be shut off again.
- 5) After exposure has been made, lower right hand screen lever
- 6) Depress **FILM FEED STOP** On.

#### **MANUAL EXPOSURE SETTING**

- 1) Set **EXPOSURE TIME**
- 2) Adjust **BRIGHTNESS** knob until Green light appears over **DENSITY ADJ** knob.
- 3) Depress **FILM FEED** button (illuminated)
- 4) Gently lift right hand screen lever. Illumination will be shut off, film will load, film will be exposed, and illumination will be shut off again.
- 5) After exposure has been made, lower right hand screen lever
- 6) Depress **FILM FEED STOP** On.

#### **FILM CHANGE**

- 1) **FILAMENT EMISSION** off

- 2) Open outer camera chamber cover
- 3) Room lights off, **PANEL LIGHTS** off, red safe lights on, put on lint free gloves
- 4) Depress **CAMERA - AIR** button, toggle film desiccator to **AIR**
- 5) Wait until red **CAMERA VACUUM STATE** lamp is illuminated
- 6) Open Camera air lock door
- 7) Using handle lift and remove "Film Receiver Box", and place aside
- 8) Reach in grab "Cassette Guide Rail", while lifting up on guide rail pull out Cassette carrier until it stops, and lower front half of guide rail
- 9) Using handle lift and remove "Cassette Magazine Box" and place aside
- 10) Install loaded "Cassette Magazine Box" from film desiccator with handle facing outward
- 11) Lift up on front half of guide rail and while lifting up on guide rail assembly slide assembly into microscope until it stops
- 12) Install empty "Receiver box" from film desiccator with handle facing outward
- 13) Remove one glove and clean camera chamber door O-ring and seat with a finger
- 14) Close camera chamber door and hold, depress **CAMERA EVAC** button
- 15) Replace glove
- 16) Unload exposed plates
- 17) Reload plates with fresh film
- 18) Load new plates in Cassette Magazine box with blanking plate on top
- 19) Place both receiver box and cassette magazine box into film desiccator
- 20) Remove gloves, clean O-ring seal and seat on desiccator door
- 21) Close and hold desiccator door, toggle **EVAC/AIR** switch to **EVAC**
- 22) Turn **PANEL LIGHTS** back on
- 23) Reset **UNEXPOSED FILM #** on CRT screen

#### **FINDING LOST APERTURES**

- If reducing **MAGNIFICATION**, or pressing **RESET** does not work
  - Center one aperture at a time
- 1) Remove problematic aperture, Filament saturated, beam spread, and sample inserted
  - 2) Insert the aperture
  - 3) Depress **DIFF** mode button (**CAMERA LENGTH** should be at 0.8m, use **MAGNIFICATION** to change)
  - 4) Use aperture X & Y knobs to center aperture shadow on bright spot  
 ⚠ Do not center the bright spot with **BRIGHTNESS CENTERING** knobs
  - 5) Depress **ZOOM** mode button

#### **CHANGING ACCELERATING VOLTAGE**

- 1) **FILAMENT** emission off - fully CCW
- 2) Press **READY/OFF** button once (Twice sets to 0 kV)
- 3) Press desired kV button
- 4) Resaturate filament, and realign for new kV

#### **INTERMEDIATE ACCELERATING VOLTAGES**

- 1) **FILAMENT** emission off - fully CCW
- 2) Set **ACCELERATING VOLTAGE** to kV button just higher than that desired
- 3) Use ↑ ↓ to highlight the **ACCELERATING VOLTAGE** on **CRT** screen
- 4) Press the **ENTER** key, use ↑ ↓ to set desired accelerating voltage (kV's below 25kV are only selectable in **STEM** mode)
- 5) Press **ENTER**, resaturate and align for new kV

#### **TV IMAGING**

- 1) Best imaging is obtained with an accelerating voltage of 75kV or higher
- 2) **ZOOM, SA** or **LOW MAG** mode button selected
- 3) TEM menu **BRIGHTNESS** should be set to **LINK** - Use ↑ ↓ to select **BRIGHTNESS**, and press the **ENTER** key until **LINK** appears
- 4) Depress **TV** mode button
- 5) Adjust **CRT ADJ** to maximum, fully CW
- 6) Florescent screen is no longer functional due to TV Camera interrupting the beam above the viewing chamber. However, photography is as normal
- 7) Depressing **TV** mode button again deselects TV Imaging

# DARKFIELD IMAGING

## DARK FIELD IMAGING MODE

- Typically image brightness is the limiting factor for Darkfield Imaging is is therefore suggested that you use the highest accelerating voltage your samples will handle, and work with a **SPOT SIZE** of **6** or **7**
  - Any magnification can be used, but again image brightness will determine your limits
- 1) Perform all **PRESTART CHECKLIST**, **INITIAL SETUP**, and **DAILY ALIGNMENT** procedures for **TEM - ZOOM** mode, align at desired operating kV - Usually the highest your samples will handle
  - 2) Insert sample, locate, center, and focus an area of interest (in **ZOOM** mode)
  - 3)

# SELECTED AREA DIFFRACTION — SAD

## SELECTED AREA DIFFRACTION IMAGING MODE

- 1) Perform all **PRESTART CHECKLIST**, **INITIAL SETUP**, and **DAILY ALIGNMENT** procedures for **TEM - ZOOM** mode, align at desired operating kV - Usually 125 Kv
- 2) Insert sample and perform **Z-AXIS CORRECTION**
- 3) Depress **S.A.** mode button (**Selected Area Mode** is used for locating specimens for diffraction)
- 4) Center electron beam
- 5) Locate and center the area of interest. Select desired **MAGNIFICATION**, and focus the image with the objective lens focus controls as normal.
- 6) Insert and center a **DIFFRACTION APERTURE** to exclude all but desired specimen region
- 7) Focus the edge of the Diffraction Aperture image with the **DIFFRACTION SPOT** knob (you may need to off-center the aperture and use the optical focusing microscope and re-center the aperture), refocus specimen image with objective lens focus knobs if needed
- 8) Remove **OBJECTIVE APERTURE**
- 9) Depress **DIFF** mode button
- 10) Focus the diffraction pattern with the **DIFFRACTION SPOT** knob (greatly overfocusing the diffraction pattern will reveal the specimen currently in the beam)
- 11) "Magnification" or camera length of the diffraction pattern is controlled with the **MAGNIFICATION** knob.

## PHOTOGRAPHY OF DIFFRACTION PATTERNS

- **ATS** exposure or manual exposure only
- 1) Set **DENSITY ADJ** to the **GREEN** calibration setting
  - 2) Set **EXPOSURE TIME** knob to **AUTO**
  - 3) Obtain desired diffraction pattern
  - 4) Lower right hand corner of CRT Screen will display exposure time. Times above 2 seconds are desired, extremely long exposure times maybe necessary. Adjusting brightness control will alter both the brightness of the diffraction pattern AND the appearance of the pattern; compromise to obtain best image.
  - 5) Depress **FILM FEED** button (illuminated)
  - 6) Gently lift right hand screen lever. Illumination will be shut off, film will load, film will be exposed, and illumination will be shut off again.
  - 7) After exposure has been made, lower right hand screen lever
  - 8) Depress **FILM FEED STOP** On.

## **SPECIMEN TILT AND Z-AXIS CORRECTION**

- Tilting the specimen can greatly improve the diffraction pattern by aligning the beam with the crystalline plane

🔊 **TILT MUST BE RETURNED TO 0° BEFORE REMOVING SPECIMEN ROD !!!** 🗣️

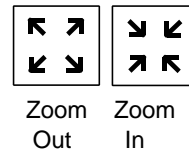
- 1) **SPEED CONTROL** module switch ON (Upper far left panel), and set **TILT** knob fully clockwise
- 2) Insert sample, locate, center and focus an easily recognizable object at 6.0k
- 3) Use the **TILT** foot pedals to tilt specimen while watching direction of motion of centered object on flat screen
- 4) Return object to center with **Z-axis** correction knob (Left side of sample airlock)
- 5) Return specimen tilt to **0°**, Re-center object with X-Y Stage controls, and re-focus object
- 6) Repeat steps 3 - 5, tilting further each time, until object stays centered
- 7) The **TILT** knob on the **SPEED CONTROL** panel can be adjusted to control the rate of tilt change during diffraction pattern observation

# SEM MODE OPERATION

## SEM IMAGING MODE

### INITIAL SETUP

- 1) Perform all **PRESTART CHECKLIST**, **INITIAL SETUP**, and **DAILY ALIGNMENT** procedures for **TEM-ZOOM** mode, align at desired operating kV - Usually at 125 kV.
- 2) **STEM** mode button depressed on left panel
- 3) **MAGNIFICATION** - Controls on upper right pannel
  - **COURSE/FINE STEP** switch to **FINE**
  - **HIGH/LOW RANGE** switch to **HIGH**
  - **MAGNIFICATION** at 2K
  - **PRESET MAG** button **OFF**
- 4) **SCANNING MODE** on **NORMAL**
- 5) **SCANNING SPEED** on
- 6) **DUAL MAG** Controls
  - **SPLIT/FULL** switch to **FULL**
  - **H.M. ONLY/DUAL MAG** switch to **DUAL MAG**
  - **DM** button **OFF**, unlit
- 7) **X/Y POSITION** and **DUAL MAG POSITION** to 12:00
- 8) **IMAGE SHIFT** Knobs to 12:00
- 9) **ABC** button **OFF**, **SIGNAL PROCESSOR** to **NORMAL**
- 10) **COND LENS** at **50**  
[ Lower **COND LENS** setting = high brightness, lower resolution  
Higher **COND LENS** setting = lower brightness, higher resolution]
- 11) Toggle **STIGMATOR RESET**
- 12) **WAVEFORM** button **OFF**, unlit
- 13) **MODE EDX/NORMAL** switch (Top right Console panel) to **NORMAL**
- 14) **PHOTO SPEED** for **STEM/SEM** set to **2**  
[ 1 = 35 sec; **2 = 100 sec**; 3 = 200 sec; 4 = 400 sec]



### 10 - 25 kV OPERATION ELECTRON BEAM ALIGNMENT

- 1) **FILAMENT** emission off (fully CCW), sample out, and **OBJECTIVE APERTURE** removed
- 2) Select **25 kV ACCELERATING VOLTAGE**
- 3) In **Zoom Mode**, turn on filament, locate and center the beam following **TEM daily alignment instructions**
- 4) **FILAMENT** emission off (fully CCW)
- 5) Insert SEM Sample
- 6) Depress **STEM** mode button on left console panel

- 7) Depress **SIGNAL SELECTOR SEM** mode button on H-7110 panel (Top right)
- 8) Set **SEM MAGNIFICATION** to 20 x
- 9) Use ↑ ↓ to highlight the **ACCELERATING VOLTAGE** on **CRT** screen
- 10) Press the **ENTER** key, use ↑ ↓ to set desired accelerating voltage
- 11) Depress **LA SCANNING MODE** button - a white line should appear on the SEM imaging CRT, if necessary adjust **SEM CONTRAST & BRIGHTNESS** controls
- 12) While watching the line, increase the **FILAMENT** emission knob until line reaches a maximum (adjustment of the **SEM CONTRAST & BRIGHTNESS** controls maybe necessary if peaks extend above the top of the screen, adjusting the **SEM FOCUS** controls will sharpen the peaks)
- 13) Use the **GUN TILT X/Y** (lower left panel) to center the filament and critically maximize the line peak height. Resaturate as described in 12.
- 14) The filament should now be saturated and aligned for SEM imaging.
- 15) Depress **NORMAL SCANNING MODE** button.

#### **CENTERING HIGH / LOW MAGNIFICATION RANGES**

- 1) Sample inserted, **SEM** mode selected, filament turned on and image formed on imaging CRT
- 2) Toggle **BEAM TILT RESET** (Lower righthand panel)
- 3) Set **RASTER ROTATION** control to **140°**
- 3) Using X/Y Stage controls locate and center easily identifiable object or area at 2k **HIGH MAG** range (**RASTER ROTATION OFF**)
- 4) Switch to **LOW MAG RANGE**, turn **RASTER ROTATION ON**. Locate and center the same object or area with **BRIGHTNESS CENTERING** Controls (**NOT** stage controls) - You may have to decrease magnification in order to find the previous high mag area
- 5) Sample area should now remain centered between **HIGH** and **LOW MAG**. except for the image rotation which can be compensated for with **140° RASTER ROTATION**

#### **SAMPLE REMOVAL \ EXITING SEM MODE**

- Scanning coils must deenergized before specimen exchange or removal
  - Accelerating voltage must be above **24 kV** in order to exit SEM mode
- 1) **FILAMENT** emission off - fully CCW
  - 2) If **kV** between **10 - 25**, press **READY/OFF** button once, press **25 kV** button
  - 3) Depress **ZOOM** mode button - scanning coils are now deenergized
  - 4) Follow normal specimen rod removal instructions above

## SYSTEM SHUTDOWN

- 1) **FILAMENT** emission off (fully CCW), - kV on
- 2) Sample out, rod in column
- 3) **ZOOM** mode selected, reset depressed, **INITIAL SETUP** conditions correct
- 4) Press **INDEX** button to record filament **ON-TIME** in log book
- 5) **CRT IMAGE ADJ** fully CCW
- 6) Change TEM film if used

## Overnight Shut-down

- 7) kV at 0 - depress **READY/ON** twice
- 8) Key switch to **EVAC ON**

## FULL SYSTEM SHUTDOWN

☞ **Full System shutdown should only be performed for**

- **Power outages**
- **Filament Changes**
- **Emergency situations**
- **Maintenance**

- 9) Key switch to **OFF**
- 10) After approximately 35 minutes the rotary pumps will shut off, and the turbomolecular pump will decelerate. At this point the scope is fully shutdown. If needed the cooling water and wall switch can be shut off.

## STARTING FROM FULL SYSTEM SHUTDOWN

- 1) Cooling water **ON**. Wall switch **ON**. Key switch to **EVAC ON**.
- 2) Wait approximately 35 minutes until:
  - Turbo pump at **NORMAL** operation
  - Green Lights for **GUN, COL, and CAMERA**