Brief Instructions

Transmission Electron Microscope LEO 922 OMEGA

1 Microscope Operation

Dim the display on the computer screen when you are finished for the day.

1.1 TEM system turn-ON

Getting the system ready

- Tap START on the computer screen.
  - LEO 922 MAIN menu is displayed.
  - Instrument readiness is indicated by a green LCD.
  - Make sure the BEAM BLANKER is OFF.
  - Scope needs to be left on at 120kV ALWAYS.

1.2 Anticontaminator
Remove lid from anticontamination reservoir.

Fill with liquid nitrogen using the metal funnel and the 1 liter blue container.

Refill after 15 minutes and close the reservoir with the lid. A full reservoir will last 4 hours. Metal of the anticontaminator is around the sample and objective aperture. This device provides a (10⁻⁷) vacuum around the sample.

1.3 High-voltage ON

Push the High Voltage black button on the left panel to go from Ready to ON. The button should say ON when you begin. HV should be at 120kV. P6 should stay 2E/3E -7. The value should stay in the -7 range and not get worse.

Once the Red light on top of key HIGH VOLTAGE is ON, the High voltage has reached its rated value: (on the computer screen), push GUN Filament black button until the ON light stops blinking ~ 2 minutes. Emission current on the computer screen should read generally > 1µA.

Increase to 2 – 3 µA with black knob on the left panel labeled CURRENT (located just below GUN, Filament).

- On the left panel, push black key above Filament. With the Type 1 LaB6 filament (selected on the computer screen), the ON light blinks until the filament is done ramping up (a few minutes)(The LaB6 ramps up 250uA/sec). Once done ramping up, the light will go to ON and stay lit. Exp. Time on the computer screen should have some sort of a reading.

- The scope’s fluorescent screen is now illuminated; if not, move specimen, decrease magnification, set δ E to zero (in the computer screen), adjust brightness and/or check and increase the beam current with the black CURRENT knob on the left panel under GUN, (turn CW until you hear a click and so you can see some brightness of the viewing screen) and so the computer screen says ~5µA (or at least there is some emission current) next to Emission Current on GUN>.

1.4 Operation of specimen holder

SPECIAL NOTE: Lock-in/out is only possible if the red LED display STOP on the goniometer is out. If not, air will penetrate into specimen and filament chambers and the ion getter pump will shut off.
No action if LED display STOP is lit.

1.4.1 Removing specimen rod:
- Slowly pull out rod straight and as far as it will go (Pulls out a lot). Turn it carefully and slowly counter-clockwise (towards you) quite a bit, until it stops and “clicks”.
  - Airlock valve closes (click).
- Release rod (and it will go in a tiny amount).
- Again, turn rod counter-clockwise (towards you) until it stops.
  - Go into the VAC menu on the computer and tap Nitrogen Flush (bottom).
  - The nitrogen will push the rod out for you and pull it out,
  - Using two hands, carefully take rod out.
  - Leave the nitrogen flush ON while changing the sample = this will keep dirt out of the shaft.

1.4.2 Loading specimen in specimen rod:
- Place specimen rod in holder.
- Loosen the tiny flat screw with hex tool and lift it out. Tip out the copper anti-twist washer and grid onto clean tissue paper. Do not lose the flat hex screw as a new one is $350 – operator is responsible for buying a new screw if lost.
- Place new grid with tweezers, in holder, specimen side UP. Add anti-twist washer, with forceps.
- Replace holding o-ring and tighten screw with hex tool. Make sure the top of screw is facing UP and fits the tool properly. Putting screw in specimen holder up-side-down will result in a trip to the machine shop to remove screw and specimen.

1.4.3 Putting specimen rod into column:

SPECIAL NOTE:
The normal position of the specimen rod is in the high vacuum.
Take it only out for specimen exchange.

Leave the filament ON when putting the specimen in and out.

- Slide specimen rod carefully into goniometer.
- In the computer menu, tap VAC and make sure the Nitrogen flush (bottom) is ON. Wait a few seconds.
Push rod in as far as it will go. The rod goes in very hard because of the large o-ring. Make sure it is in all the way.

- Turn specimen rod carefully clockwise (to the right and away from you) all the way until it stops. The red light STOP comes ON. Specimen rod pre-pumped in ~ 20 seconds and then the red light will go OUT.

- Wait until another 30 seconds before the next step.

- Pull specimen rod back out a tiny amount to the stop and you hear a click. In the VAC menu, watch that P4 = sample stays in the $10^{-7}$ range and P6 = gun stays in the $10^{-7}$ range.

- Turn rod carefully clockwise (away from you) until it stops and slowly guide the rod into the microscope column.

- Push #0 on the keyboard to remove the motorized AIS aperture and activate the AIS mode in the main menu. (#2 on the computer keyboard inserts AIS aperture).

- Select magnification to 10,000x with black buttons MAGNIFICATION $\Delta \nabla$
  (located on the lower right of the left panel).

### 1.5 Alignment of the spectrometer

- Reset energy loss to zero with the computer screen button HCl/0eV (bottom right) in the menu $\Delta E$ (at the top of the computer screen).

- On the left panel, switch to SPEC with the black button IMAGE/SPEC above SPECTROMETER.
  - The spectrum caustic cone is visible on the screen (as it goes through the filter).

- Select SHIFT (Red) function with the black button SHIFT/FOCUS above SPECTROMETER.

- Shift the tip of the spectrum caustic to the black center point on the TEM screen using both the black knob above the SHIFT/FOCUS button (SHIFT = Red) (left panel) and the IMAGE Y (do not use the X knob) black knob (left panel).

- On the left panel, switch to IMAGE mode with the black button IMAGE/SPEC.

- Switch to TEM (white) with the TEM/SPOT button over ILL MODE (and under BRIGHTNESS on the right panel) and center motorized AIS aperture on the large screen with the 4 black arrow buttons $<^\wedge>$ on the LEO panel (Use the 4 arrows because APERTURE ADJUST is not activated).
1.6 Adjustment of eucentric plane

- Move specimen rod all the way in (turn rod slightly away from you and let it go into the column).
- Go to IMAGE/DIFF button on the left panel and make sure DIFF (red) is lit (This step is to help center the objective aperture).
- Insert an objective aperture (two clicks on the large knob CW) and center it with the 2 knobs on the column.
- Click from DIFF to IMAGE (white) with the IMAGE/DIFF button on the left panel.
- Select magnification 40,000x and move specimen detail to the center of the small screen.
- Push the CALIBRATE button located on the right panel under FOCUS.
- Switch on the FOCUS AID with button AID located on the right panel.
  -- Illuminated ON (Red) indicates FOCUS AID is active; specimen detail is moving (wobbler).
- Focus specimen mechanically using Z-CONTROL buttons (these buttons/arrows ^ are located on the LEO control panel to the right of the joystick), until specimen detail does not move any more.
  -- Specimen is now in the eucentric plane of the goniometer.
- Switch off FOCUS AID with button AID (right panel).

1.7 Centering of spectrometer aperture

- Insert entrance aperture (top) (2 clicks) and center it on the large viewing screen using the aperture knobs (on the column). Magnification should be at least 16,000X.

1.8 Centering of spectrometer slit aperture

SPECIAL NOTE:
The slit aperture (last aperture on the column) has a fixed shape with defined slit widths. The slit width can be selected mechanically by the aperture drive.

It is possible to adjust the slit width and the spectrum to each other. This can be done for each slit width. In this manual the mechanical adjustment and slit selection is...
described.

- Switch to SPEC with button IMAGE/SPEC above SPECTROMETER on the left panel.
  - Spectrum caustic cone should be close to the center of the screen.

- Center tip of the caustic cone to the black point on small viewing screen with black knob above SHIFT (red)/FOCUS (and above SPECTROMETER) on left panel.

- Introduce slit aperture (by turning 2 clicks CW) with the aperture's large knob on the column and adjust the desired slit using the right knob on the column, so that the spectrum caustic cone is within the slit.
  - The tip of the cone should not be too close to the left side of the slit (slit should not cut off the cone's tip).

- Switch to IMAGE mode with button IMAGE/SPEC on the left panel.

1.9 Correction of Imaging Astigmatism

- Select a magnification of 10,000x with buttons MAGNIFICATION Δ,∇.

- Switch to STIG with button SHIFT/STIG above IMAGE on the left panel.

- Compensate astigmatism with the black X and Y knobs above IMAGE on the left panel, while observing the Live FFT computer screen on the right (for the GATAN digital image).
  - The astigmatism is compensated, if there is no directional stretching of the concentric circles in under- and over-focus.

- Switch back to SHIFT with button SHIFT/STIG above IMAGE.

- Adjust brightness and magnification on the viewing screen to the desired values.

1.10 Low Magnifications (LM)

- Select LM mode by tapping M/LM in the main computer menu.

- Take all apertures out:

- Retract slit aperture (bottom on column).
  - The field of view might be limited by the objective lens aperture.

- Retract objective lens aperture manually and motorized condenser aperture by pushing # 2 = (in/400 µm) or # 0 (no aperture) on the computer keyboard.

- Select magnification of 1,600x with black buttons MAGNIFICATION ∨Δ under IMAGE on the left panel.
Center image on the large screen using the black Image X and Y knobs (above IMAGE on left panel). Make sure you are on SHIFT on the SHIFT/STIG button and IMAGE on the IMAGE/DIFF button (left panel).

Adjust brightness with buttons ILL APERTURE \( \nabla \Delta \) under BRIGHTNESS on the right panel.

Store desired specimen positions in the GON (top) menu:

- In computer menu, tap GON to activate
- Make sure both Auto Store Inc and Auto Rec Inc are activated (top middle right)
- Put area of interest in middle of microscope screen and
- Tap Store Pos. (top middle)
- To start over with position numbers, Edit Address (bottom), type 1, tap keyboard Enter, Store Position (can also try Recall Position)

Go to 8,000X while all the apertures are out.

Insert objective lens aperture and center it to the index point before switching to magnification range M (Specimen protection).

### 1.11 Medium and High Magnifications

- Insert objective lens aperture (if not done yet after finishing LM).
- Insert motorized condenser aperture (AIS) by pushing # 2 on the keyboard.
- Select M mode with a tap on LM/M in the main computer menu.
- Select a magnification of 4,000x with buttons MAGNIFICATION \( \nabla \Delta \) on left panel below IMAGE.
- Adjust brightness with the buttons ILL APERTURE \( \nabla \Delta \) under BRIGHTHNESS on right panel
- Insert slit aperture and select the desired slit width

### 1.12 Focusing the Specimen

- Adjust binocular to your eye sight (each eye piece turns separately) and focus it on the center black point of the small viewing screen. (Binoculars move from side to side).
- Move specimen detail to the center of the viewing screen.
Switch ON FOCUS AID by pushing button ON and push CALIBRATE button (Both buttons are located under FOCUS on the right panel).

Specimen detail is moving in the center.

Minimize the specimen movement with the 2 Z-Control buttons on the LEO panel and then focus the specimen with the black OBJECTIVE knob on the right panel, until the detail stops moving.

Push button AID to switch OFF (located under FOCUS on the right panel).

### 1.13 Turning Down MICROSCOPE when Session is Finished

Switch off FILAMENT by turning CURRENT knob counter clockwise until emission readout is < 1μm. Push the black filament button on left panel under GUN. Once you have pushed the button, wait for the blinking ON to shut OFF (This process takes 2-3 minutes).

Return to the main computer menu by tapping RETURN (in lower left corner).

Remove your specimen and reinsert the empty specimen rod all the way into the vacuum.

F10 on the computer keyboard shuts off all panel lights and turn OFF the computer screens.

Remove and Shut OFF Digital camera.

### 1.14 To Reboot Computer

Shut OFF filament (blinks until it is down) and shut OFF kV (will say Ready).

Takes a bit less than 5 minutes.

Tap EXIT (top right on computer screen).

Using a wooden Q-tip stick, push in just over the green tape (located to the right of the ON key).

Wait several minutes.

Tap START on the computer screen (bottom left).

Tap High Voltage on left panel to restart kV.