

ZEISS 10C - OPERATING INSTRUCTIONS

(Version 3.01 - February 2008)

COLD START UP PROCEDURE

1. Turn on **O/I** button. An alarm will come on for about 5 sec. before the pump starts.
2. Scope takes about 20 min. to warm up and come to an operating vacuum.


PRE-START CHECKLIST

1. Green **HV** ⚡ light will be on when there is an operating vacuum.
2. Turn on Display console - Middle upper right console.
3. Sign Log Book.
4. Fill the anti-contamination device dewar (cold finger) with liquid nitrogen before you turn on the high voltage.
5. Be sure that the **DESICC.** light is pushed in and lit up *BEFORE* inserting a sample into the scope.
6. Setup room lighting


BEAM GENERATION

1. Select **HIGH VOLTAGE** (20, 40, 60, 80, 100 KV) on left top console.
2. Select spot size with the **CONDENSER 1** control on the top right console (start at **3**)
3. **MAGNIFICATION** to **2,000x**.
4. **FILAMENT HEATING** fully CCW.
5. Set **CONDENSER 2** course setting to **4**
6. Check to see if the condenser aperture has been inserted into the column and insert it if necessary. The first aperture is recommended.
7. Depress the green **HV** ⚡ button on the left top console ☞ red **HV** ⚡ button lights up.
8. Depress the red **FIL** button (you should now have a dim glow on the screen)
9. Bring the beam to crossover with the inner (small) **CONDENSER 2** knob.
10. Turn up the **FILAMENT HEATING** knob until the Wehnelt image is visible.
11. Center the beam with **BEAM ALIGNMENT III** and **IV** on lower right console.
12. Spread beam to fill the screen.
13. Center condenser aperture.
14. Bring the beam to crossover with the inner (small) **CONDENSER 2** knob.
15. Beam tilt is controlled by **BEAM ALIGNMENT Y-I & Y-IV** and **X-II & X-III**. They work by turning/adjusting them together to get the beam tilted properly (x versus y tilt). Use the lefthand controls (**Y-I** or **X-II**) to adjust the Wehnelt image while simultaneously re-centering with the righthand controls (**Y-IV** or **X-III**, respectively). Do not use them separately because they will just move the beam instead of changing the tilt.
16. Check for condenser astigmatism. If astigmatic, use **C₂ STIGM** rollers on right lower console to correct and make beam round.
17. Saturate filament - turn up **FILAMENT HEATING** until all traces of wehnelt image disappear. **Spread beam!**
18. Insert objective aperture and center.
19. Adjust optical focusing aide.

SPECIMEN INSERTION

1. Be sure that the stage controls are at 500, 500 before inserting a sample.
2. Pull specimen insertion lever out, up and around until the lever clicks into the slot.
3. Rotate entire sample holder (silver wheel) CCW until it stops.
4. Using the insertion tool, remove sample holder (do not touch holder with your hands) and place in silver specimen manipulation holder and raise the sample cartridge into place with fork tool.
5. Unscrew cap on sample holder with cap remover and place grid in holder with the sample side up (down in scope).
6. Replace sample cap and insert sample holder back into the column, turn silver wheel CW so that it locks into place.
7. Depress red button on lever and turn CW to 3 o'clock position and wait for vacuum to read 1 -2 on the display console.
8. Turn lever around and down to 6 o'clock position and push into first stop.
 NEVER leave air lock lever in 3 o'clock position for longer than 1 min.
9. Push red button on lever in and push into next stop. Repeat until sample is in column.
10. Adjust brightness with **CONDENSER 2** and change magnification with the corresponding control knob.

SPECIMEN REMOVAL

1. Center stage at 500, 500.
2. Pull rod out and rotate entire sample holder (silver wheel) up and around until the rod clicks into the slot.
3. Using the insertion tool, remove sample holder and place in silver specimen manipulation holder and raise the sample cartridge into place with fork tool.
4. Unscrew cap on sample holder with cap remover and remove grid from holder .
5. Replace sample cap and insert sample holder back into the column, turn silver wheel so that it locks into place.
6. Turn rod to 3 o'clock position and wait for vacuum to read 1 -2 on the display console.
7. Turn rod around and down to 6 o'clock position and push into first stop.
 NEVER leave air lock lever in 3 o'clock position for longer than 1 min.

SPECIMEN VIEWING

1. Check the objective astigmatism. If it needs to be corrected, find a round hole in the sample and use **OBJ STIGM** rollers on left lower console to correct. (Correct at the highest magnification that you will be using.)
2. Focus using the **COARSE** and medium-fine **FOCUS STEP** control knobs. The **FOCUS STEP** knob can be adjusted to make the focus steps larger or smaller by changing the inner small knob to the appropriate step size.
3. Medium and fine focus are then achieved with the outer ring of the **FOCUS STEP** control.

PHOTOGRAPHY

1. Make sure that the **CAMERA SELECTOR** is pointing toward **PL** (plate).
2. Focus and stigmatite field of view to be photographed.
3. The red **FOC AID** (WOBBLER) button in the **EXPOSURE CONTROL** area can be used to help you find the correct focus if so desired.
4. While watching the **EXPOSURE TIMER** meter and using the **CONDENSER 2** knob, adjust the brightness so that the needle reads 2 sec. on the timer meter.
5. Depress the green **START** button in the **EXPOSURE CONTROL** area. The microscope will automatically take the picture at a 2 second exposure.
6. You are now ready to take another picture.
7. When you have finished taking pictures, follow directions for sample removal and changing film.

CHANGING FILM

1. Spread the beam with **CONDENSER 2** and turn **FILAMENT HEATING** fully counter-clockwise.
2. Depress red **FIL** button and then depress red **⚡ HV** button. Filament and HV are now off.
3. Be sure that red **DESICC** button is pushed IN and LIT (not pumping) before continuing.
4. Turn off room lights and panel lights.
5. Open nitrogen tank valve.
6. Depress red **CAM** button to begin venting of the camera. When camera lid begins to flutter, turn off the nitrogen tank valve.
7. Remove camera lid and disengage receiver box below. Slide receiver box down and remove it from the retaining bar.
8. Remove exposed film plates from receiver box and set them aside.
9. Return receiver box to sliding retaining bar, wipe off o-ring with naked finger, push receiver box up until it clicks to a stop. Fasten the retaining bar into place.
10. Open desiccator drawer and pull up on the knob. This will release the vacuum in the drawer.
11. Starting with the box on the left side (closest to user), take one plate from the top and run 'Static Master' lightly over negative to remove any dust or dirt on film.
12. Place into top of camera with raised ridge of negative holder on the right side.
13. Replace all film in this manner until camera is full (29 negatives total.)
14. When camera is full again, wipe off o-ring with naked finger, replace camera lid.
15. Depress the **PV-STOP** button (button in and dark), and depress and release the **CAM** button to begin pumping of camera (Button dark).
16. Remove exposed film from holders and replace with fresh film. Place new film into the far right box (film side down) and move older film towards the left to be used next, wipe off desiccator o-ring with naked finger and close desiccator lid. Push drawer in.
17. Check vacuum display until vacuum reads **5 -2** on display (or better) then depress and release the **PV-STOP** button (Dark)
18. Depress and release **DESICC** button. A loud click should be heard. If readout on display moves quickly from **5 -1** through to **1 -2**, then the pump has NOT been engaged.


19. Depress **DESICC** again and watch the display. A slow drop of the vacuum indicates that the pump has been engaged. Pump out desiccator until vacuum reads **5 -2** on display (or better) and then turn off by pressing **DESICC** (so that it stays depressed and lit.) **Do not pump overnight !!!!!**
20. Record negative information in negative logbook on top of left console.
21. Sign logbook.

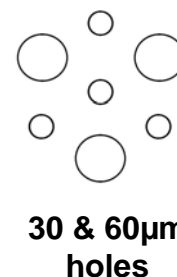
VIDEO DISPLAY SYSTEM

1. CCD Camera requires either 80KeV or 100KeV
2. Turn on the electrical power strip (left side of scope)
3. Turn on monitor.
4. Turn on CCD camera (immediate right side of column)
5. Use small black lever above CCD camera to swing camera into or out of the beam.

OBJECTIVE APERTURES: LOCATING AND CENTERING

— Position #1 Multi-holed aperture (30 & 60 μ m), #2 80 μ m, #3 25 μ m

1. Specimen in beam (aperture out at #0 if needed to locate sample)
2. Increase **MAGNIFICATION** 50K (no need to focus at high mag.),
3. Converge and center the beam.
4. Switch to **DIFFRACTION** mode
5. Use **DIFFRACTION** mode focus to focus the aperture on the screen
6. Center aperture on brightest spot  When nearing bright spot spread the beam with course control
7. Switch back to **IMAGE** mode
8. Correct brightness
9. Correct Magnification



SELECTED AREA DIFFRACTION — [SAD]

- SAD Apertures are 400, 200 and 50 μ m diameters and correspond to 8, 4 or 1 μ m diameters on sample (20, 10 & 2.4 μ m at Low Mag.)

1. Locate specimen in normal **IMAGE** mode
2. Insert an Selected Area Aperture to exclude all but desired specimen area
3. **C2** course control set to above 6
4. Switch to **DIFFRACTION** mode
5. Remove Objective aperture (i.e. Obj. Apt at #0)
6. Insert beam stop to block central “caustic spot”
7. Use **DIFFRACTION** mode focus to focus the spot pattern on screen
8. Select camera length with **MAGNIFICATION** knob
9. Adjust specimen X/Y position and tilt angle to obtain optimal desired pattern
10. Photograph as normal - note exposures may be long
11. Switch back to **IMAGE** mode

LOW-ANGLE DIFFRACTION

- Up to Lattice constants of approximately 1.6 μ m
- 1. Locate Sample area
- 2. Switch **C1** to step 4 or 5
- 3. Switch off **C2** lens (depress C2 Lens control lower left panel)
- 4. Switch off **OBJECTIVE** lens
- 5. Use 30 μ m objective aperture as selective aperture.
- 6. Select camera length with **MAGNIFICATION** knob

Dark Field Imaging

Simple:

1. Obtain diffraction image (no SAD aperture)
2. Insert objective aperture
3. Align aperture to exclude zero order diffraction spot.
4. Switch back to **IMAGE** mode

Beam Tilt Mode:

1. Obtain diffraction image (with largest SAD aperture#1)
2. Insert and center objective aperture on central bright spot
3. Turn on **DF** mode button (Lower right panel Beam Tilt) — Set X and Y tilt controls to 0
4. Tilt beam to relocate central bright spot by turning X and/or Y beam tilt knobs until central bright spot (zero order diffraction spot) disappears behind perimeter of objective aperture. If desired you can center a specific diffraction spot.
5. Switch back to **IMAGE** mode
6. Remove SAD Aperture (if below mag. is 20,000X).
7. In DF-Mode converge and center beam using the **BEAM TILT CORR.** controls.
(Note: center BF-mode beam as normal with **Y-IV** or **X-III**)
8. You should be able to switch between brightfield and darkfield modes by depressing and releasing DF button.

(Note: focusing is generally easier in BF mode)

USING GONIOMETER

1. Specimen cartridge must be OUT to start with.
2. Selector switch to Goniometer
3. Green **REM P** button depressed
4. Turn on I/O switch —> Wait until **REM P** is illuminated
5. Both display toggles in up positions (rotation and tilt selected)
6. Insert goniometer cartridge slowly as normal
7. Go to 100X, and spread beam to fill screen
8. Press and release **REM P** button
9. Press **Zero P** button and wait until Tilt is **00.0** and Rotation is **000**
10. Release Zero P button.
11. Use foot pedals to adjust tilt and rotation as needed.

12. To remove: Green **REM P** button depressed and wait until button illuminated.

Loading Goniometer cartridge:

- Ha! Good luck!

SHUTDOWN:

NORMAL SHUT DOWN

1. **FILAMENT HEATING** knob fully CCW
2. Turn OFF Filament: Depress and release the red **FIL** button (button unlit)
3. High Voltage off: Depress the Red **HV** ⚡ button on the left top console
 - ☞ green **HV** ⚡ button lights up.
4. **MAGNIFICATION** to **2,000x**.
5. Set **CONDENSER 2** coarse setting fully CW (#30)
6. Remove Sample.
7. If photomicrographs have been taken proceed to **FILM CHANGE** (Above)
 - ☞ Scope must be back at high vacuum (green **HV** ⚡ button lights up) before you leave the scope room.
8. Turn off Video system if it is on.
9. ☞ **SIGN OUT OF LOG BOOK** ☞
10. Lights off

FULL SHUT DOWN

- ☞ Full shut down is to be done ONLY in case of:
 - a. Interruption of building utility services (Electrical or Water)
 - b. Vacuum system failure
 - c. Instruction of EMF Staff
1. (If time permits) Proceed with **NORMAL SHUT DOWN**
 2. Depress and release the red lit **I/O** button (left most button on the main console left panel) - lights will all go out, pumps will still run.
 3. Wait 30-40 mins (scope room will be quite - all pumps off)
 4. Switch breaker box to **OFF**
 5. Leave cooling water recirculator in **ON** position.
 6. Post message on scope describing reason for **FULL SHUTDOWN**

TURNING SCOPE ON AFTER FULL SHUT DOWN

- ☞ To be done ONLY after correction of the reason for the full shut down
1. Breaker box to **ON**
 2. ✓ Check that cooling water recirculator manual switch is **ON**
 3. Turn on **O/I** button. An alarm will come on for about 2 sec. before the pump starts.
 4. Scope takes about 20 min. to warm up and come to an operating vacuum.
- ☞ After 20-30 mins green **HV** ⚡ button (Left upper panel) should be illuminated indicating the scope is ready for operation.

Zeiss Magnification “Click Stop” table.

Click Stop	Magnification (Normal)	Magnification (*Low Mag 0.4x)	Camera Length		
			(Normal)	(*Low Mag)	Low-Angle
1	100	40	—	—	18 m
2	1,000	400	56.5 mm	150 mm	2.5 m
3	1,250	500	72 mm	191 mm	3.5 m
4	1,600	640	101 mm	268 mm	5 m
5	2,000	800	130 mm	345 mm	6.5 m
6	2,500	1,000	163 mm	432 mm	8 m
7	3,150	1,260	212 mm	656 mm	10 m
8	4,000	1,600	212 mm	656 mm	10 m
9	5,000	2,000	212 mm	656 mm	10 m
10	6,300	2,520	212 mm	656 mm	10 m
11	8,000	3,200	246 mm	700 mm	13 m
12	10,000	4,000	320 mm	850 mm	15 m
13	12,500	5,000	435 mm	1150 mm	20 m
14	16,000	6,400	435 mm	1150 mm	20 m
15	20,000	8,000	435 mm	1150 mm	20 m
16	25,000	10,000	435 mm	1150 mm	20 m
17	31,500	12,600	435 mm	1150 mm	20 m
18	40,000	16,000	435 mm	1150 mm	20 m
19	50,000	20,000	435 mm	1150 mm	20 m
20	63,000	25,200	435 mm	1150 mm	20 m
21	80,000	32,000	435 mm	1150 mm	20 m
22	100,000	40,000	435 mm	1150 mm	20 m
23	125,000	50,000	435 mm	1150 mm	20 m
24	160,000	64,000	435 mm	1150 mm	20 m
25	200,000	80,000	435 mm	1150 mm	20 m

“Click Stop” Printed on Film

“Magnification” as it appears on Display

* For “Low Mag” - Low Mag holder must be used and Switch 6.3 turned CW [0.4x Lit]