A General Guide to Cross-Sectional TEM Specimen Preparation

Stacking and Bonding Substrates (Permanent):

(1) Apply some M-Bond epoxy onto the surfaces of interest of a pair of specimens using a toothpick. No heating is necessary. A thin layer is sufficient. Remove excess amount;
(2) For thin substrates, it may be necessary to stack and bond more than a pair of specimens together to obtain thicker cross-section specimen so that there is sufficient area coverage on the Cu TEM aperture;
(3) Stack the specimens. Apply pressure using a clamp or weights;
(4) Allow curing to occur for about 24 hours.

Bonding (Temporary – To Facilitate Cutting):

(1) Place a cleaned microscope slide onto the hot-plate at a power setting of '3.5' (see Figure 1);

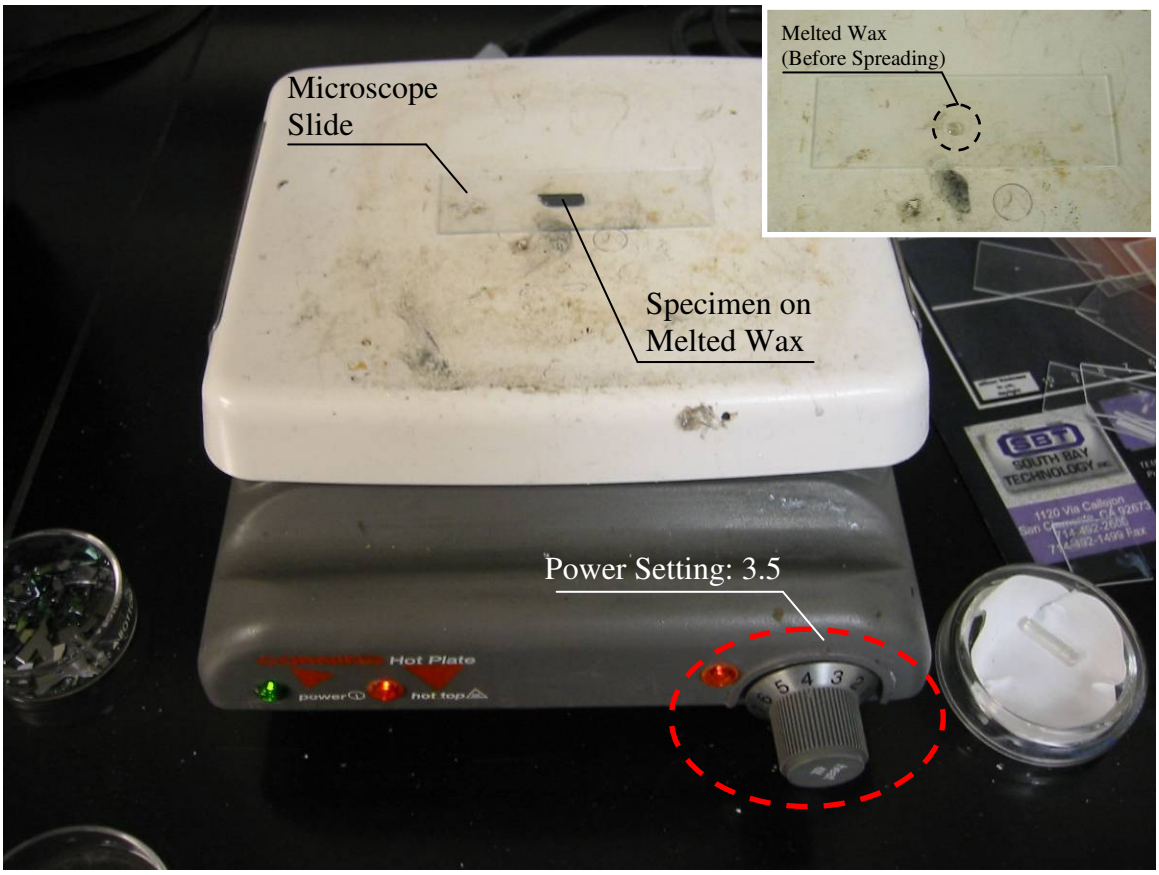

Figure 1 Bonding specimen on microscope slide for cutting. (Inset shows melted wax on heated slide prior to bonding specimen.)

(2) Place an appropriate (enough to wet the specimen area) amount of wax onto the heated slide. Spread the wax with a toothpick;

(3) Place specimen onto the melted wax. Make sure the melted wax wets the specimen completely before removing the slide for cooling (see Figure 1).

Cutting:

(1) Install the diamond-impregnated blade as shown in Figure 2;

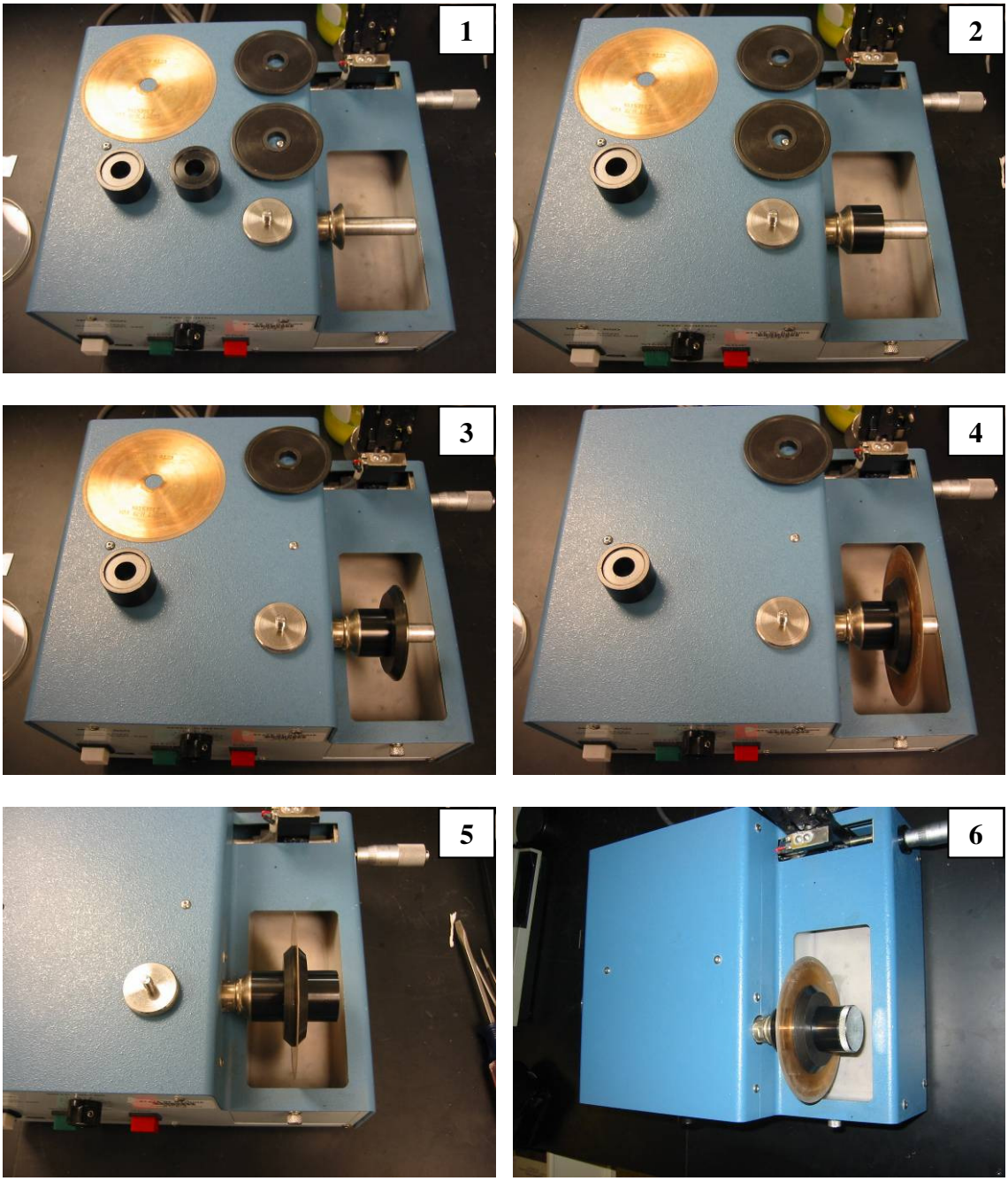

Figure 2 Sequences for installing the diamond-impregnated blade for cutting.

(2) Pour the lubricating/cooling fluid (green liquid in a beaker) into the reservoir. Make sure the level is sufficiently high to cover the edge of the blade (see Figure 3);

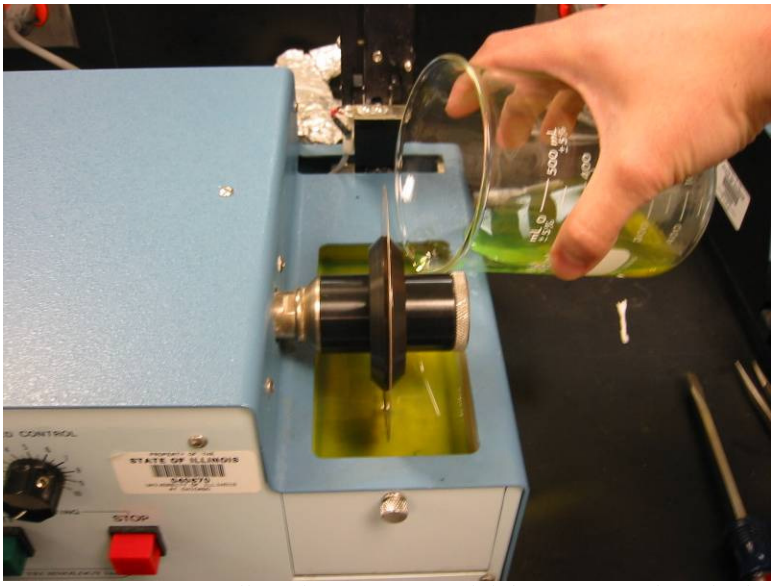

Figure 3 *Filling the reservoir with lubricating/cooling fluid.*

(3) Clamp the glass slide onto the diamond saw cutter arm. Do not over-tighten the screws as you may risk breaking the slide (see Figure 4);

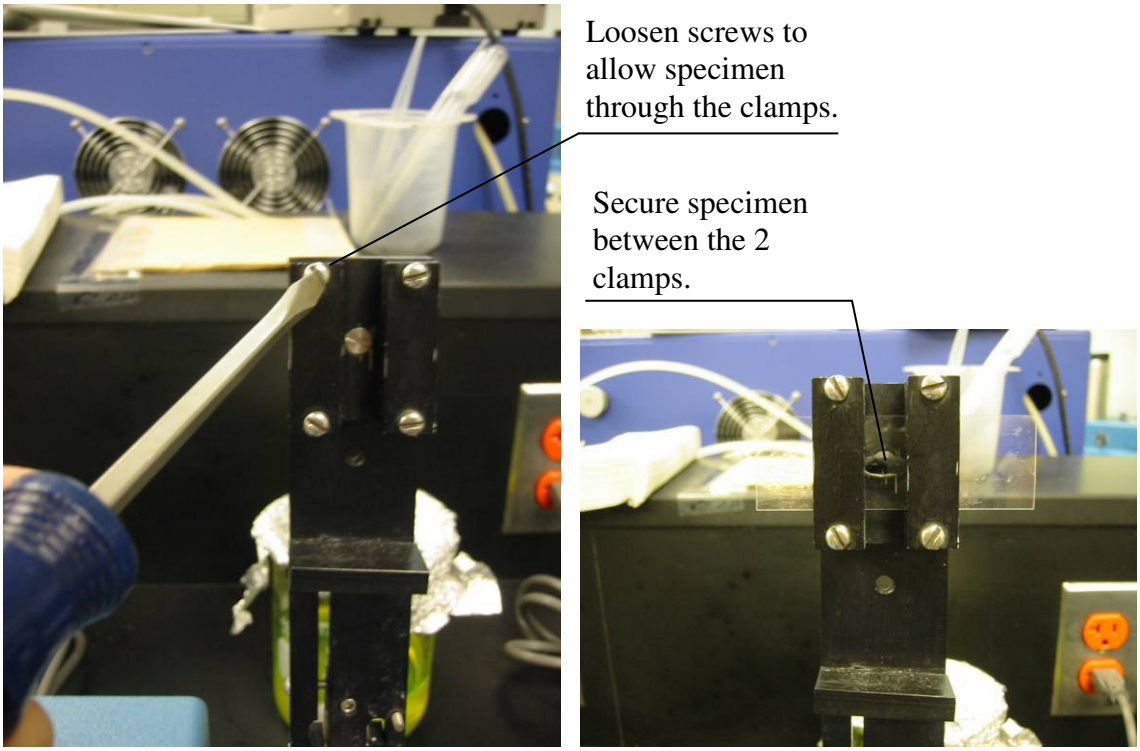

Figure 4 *Securing specimen on the cutter arm.*

(4) Adjust the arm to the appropriate location and lower the specimen onto the diamond-impregnated blade (see Figure 5). **DO NOT** adjust the arm while the blade is in contact with the specimen or during cutting. Switch off the saw and raise the arm before lateral adjustments;

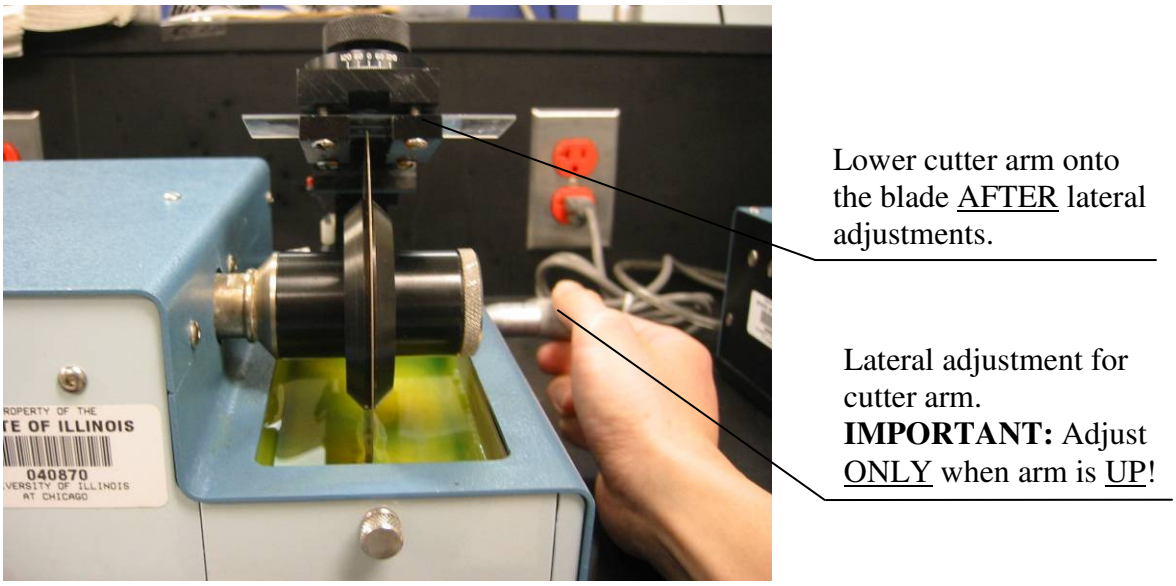

Figure 5 *Lateral adjustments of the cutter arm position before cutting.*

(5) As a precaution, set the saw to the slowest speed. Press 'Power' then 'Start' to start cutting (see Figure 6);

(6) Increase to your desired target speed slowly (usually around setting '3' to '5');

Figure 6 *Diamond saw controls.*

(7) When cutting is completed, press ‘Stop’ then ‘Power’ to terminate the cutting;
(8) Remove slide from the cutter arm;
(9) Remove diamond-impregnated blade and its assembly. Rinse and dry the blade before storing;
(10) Drain out the lubricating/cooling fluid from the reservoir and pour it back into the beaker for reuse (see Figure 7);

Figure 7 Removing the lubricating/cooling fluid reservoir.

(11) Place the slide back onto the hot-plate;
(12) Remove specimen from the slide when the wax has re-melted.

Grinding and Polishing:

(1) Place the specimen stub of the Gatan Disk Grinder onto a heated hotplate;
(2) Place some wax (enough to wet the specimen) on the heated stub;
(3) Place a specimen on the melted wax. Cross-sectional side up;
(4) Use a pair of tweezers to apply some pressure over the specimen to ensure that it is flat against the stub surface;
(5) Remove the stub for cooling;
(6) Turn the dial on the disk grinder counter-clockwise to retract the piston (see Figure 8 – top);
(7) Insert stub into the hole of the polishing platen such that it is against the piston (see Figure 8 – bottom);

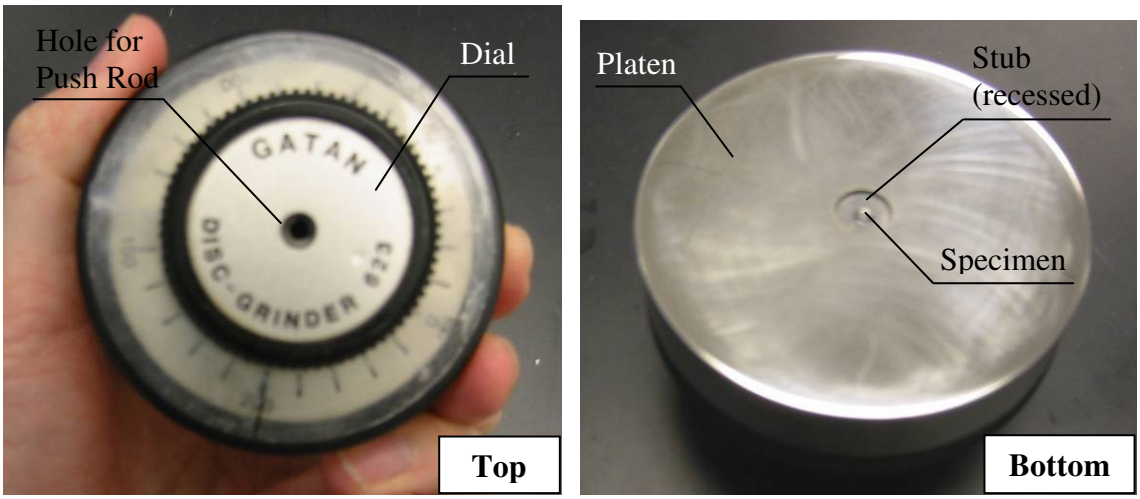

Figure 8 Gatan Disk Grinder (top-view showing the dial; bottom view showing the platen and stub with specimen).

(8) Turn the dial of the disk grinder clockwise to push the stub outwards. Stop when the specimen is slightly protruding from the polishing surface of the platen.

(9) Mount a piece of SiC abrasive paper on the polishing wheel (see Figure 9). For most specimens, start with 240- or 600-grid papers for rapid thinning;

(10) Turn on the water supply on the polishing wheel (see Figure 9). Flow rates that are slightly higher than a 'fast drip' is sufficient. Too high a flow will result in splashing;

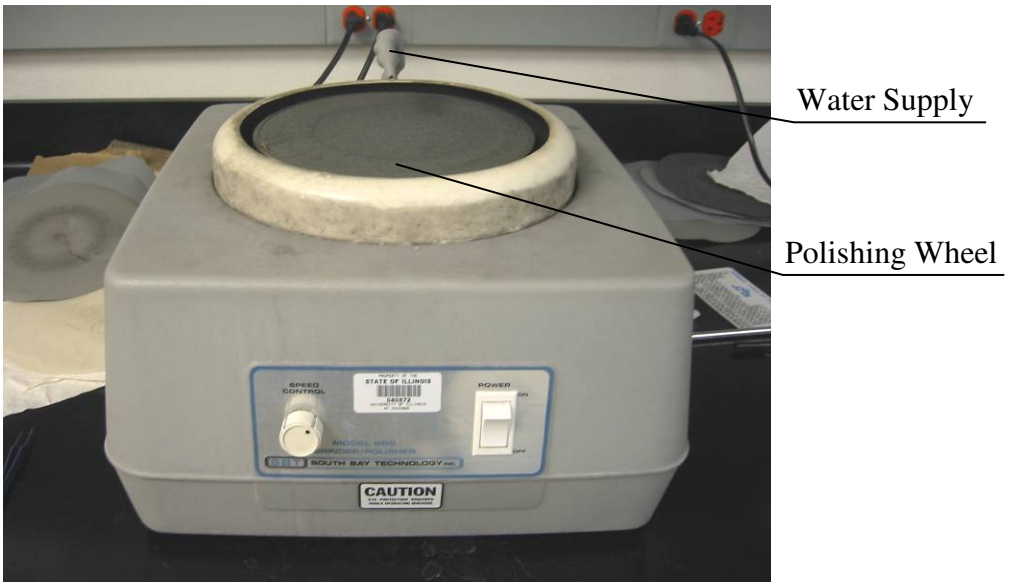

Figure 9 Polishing Wheel.

(11) Switch on the polishing wheel power. Reduce wheel speed to minimum;

(12) Hold onto the polishing platen with the specimen in contact with the abrasive paper;

(13) Increase the wheel speed appropriately. Refrain from applying too much downward pressure on the platen while grinding/polishing as the specimen may break off from the stub;
(14) Before the specimen becomes substantially thin, remount the specimen to expose the other side for grinding/polishing;
(15) The stub can be removed from the platen using the supplied push rod. Slide the rod through the hole from the top-side. The stub should pop out;
(16) Switch to progressively finer polishing papers as specimen becomes thin;
(17) Stop the grinding/polishing steps before it becomes too hard to handle with tweezers (typically about 50 microns).

Note: Thickness measurements can be performed using a micrometer. However, be warned that there is a substantial risk of specimen breakage associated with micrometer measurement.

Bonding of Specimen to TEM Aperture Grid:

(1) Apply some M-Bond epoxy onto the TEM slot grid (copper) using a toothpick. No heating is necessary. A very thin layer is sufficient. Excess amount may cause the glue to spread over the area-of-interest;
(2) Carefully lay the glue-side of the TEM grid onto the specimen. Align the slot with the specimen's glue-line;
(3) Apply slight pressure onto the TEM grid using a pair of tweezers;
(4) Allow curing to occur for about 24 hours.

Ion Milling (High-Angle):

(1) Place a circular-aperture bottom-plate (groove face up) onto a gear-tooth outer ring (see Figure 10);

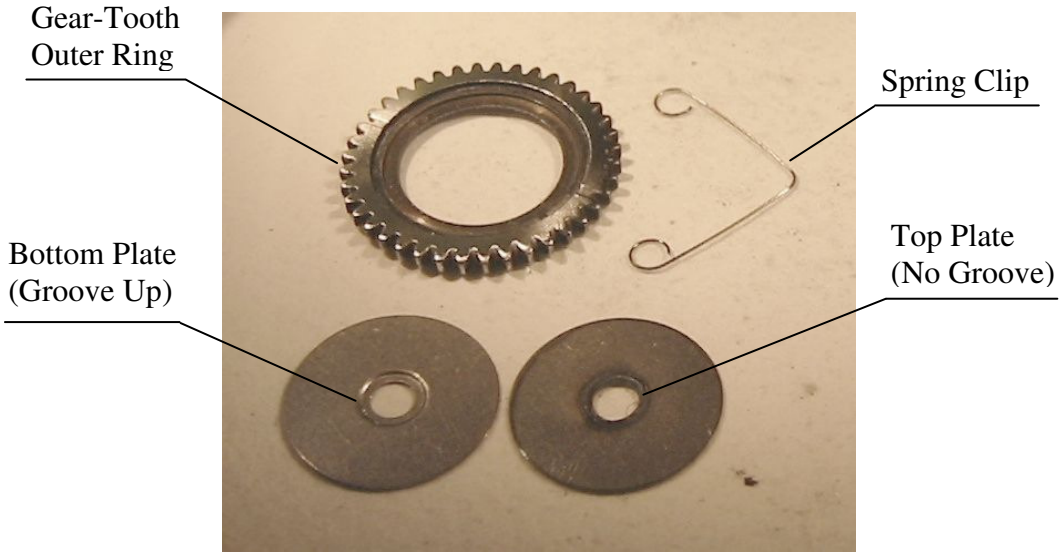

Figure 10 Individual components of a Fishione high-angle specimen holder.

(2) Place TEM grid onto the grove of the bottom-plate;

(3) Place a circular-aperture top-plate (no grooves) onto the TEM grid, aligning it with the bottom-plate;

(4) Set spring clip over the top plate as shown in Figure 11. Make sure the spring ends and elbow rest in the recessed edge of the gear-tooth outer ring, while avoiding the grooves on the ring.

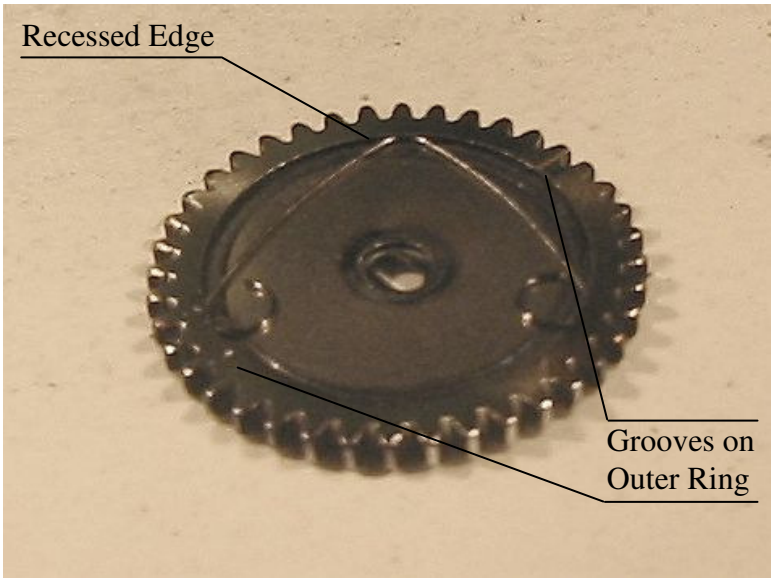

Figure 11 Assembled Fishione 3000 specimen holder.

(5) Switch on the power to the Ion Miller (switch located at the back of the instrument). If the milling chamber is in vacuum, go to 'vacuum' on the monitor screen (see Figure 12) using the keypad and press 'enter' to toggle status to 'off'. System will vent;

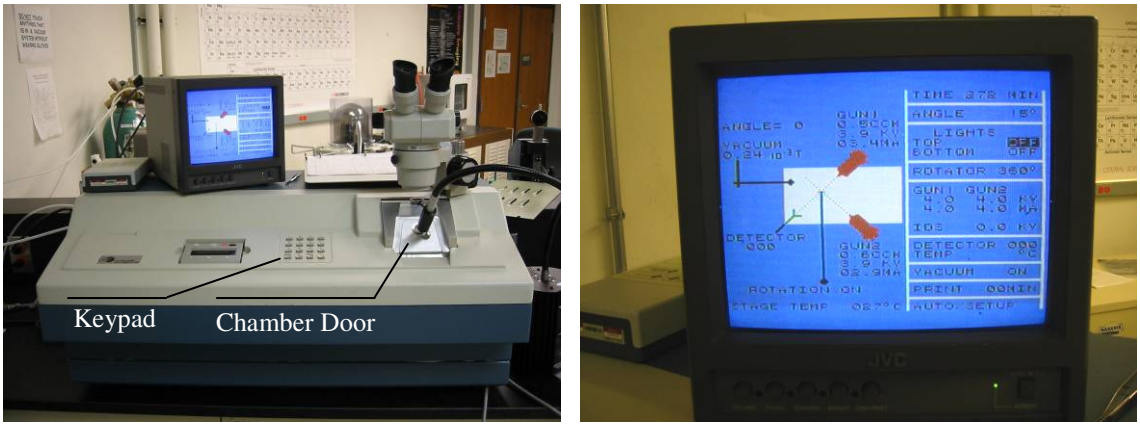

Figure 12 Fishione 3000 Ion Miller and display unit.

(6) Open the chamber door and load specimen onto the stage, making sure the gears are aligned with those on the stage (see Figure 13);

(7) Make sure that the specimen's glue-line is aligned at the 3 – 9 o'clock position;

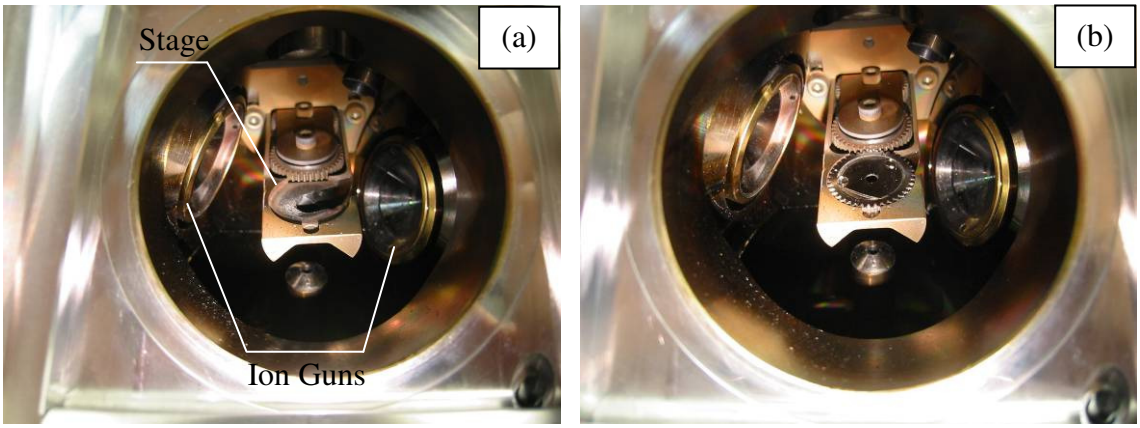

Figure 13 Ion Miller chamber showing specimen stage (a) before and (b) after loading.

(8) Set 'rotation' to '60°'. The specimen will 'rock' ±30° with respect to the 3 – 9 o'clock position;
(9) Close chamber door and set 'vacuum' to 'on'. System will pump down and chamber will be purged automatically;
(10) Set the milling angle. Typically 15° for initial milling;
(11) Wait for 'gas purge' status to go off on the monitor before setting up the ion parameters;
(12) Set gun to desired voltages and current. Typically 4 kV and 4 µA. Guns will purge automatically;
(13) Set milling timer;
(14) Monitor milling progress periodically. The aim is to terminate the milling just before/after ion beam penetration;
(15) The milling angle should be lowered to about 8° - 10° just before or upon ion beam penetration. Lower the gun voltages and currents to 3kV and 3 µA, respectively;
(16) Mill for an additional 1 to 2 hours;
(17) After terminating the milling process, toggle 'vacuum' to 'off' using the keypad and wait for chamber to vent;
(18) Remove specimen holder and then re-evacuate the chamber;
(19) Switch off the ion miller when chamber is in vacuum.

Note: All operations are controlled using the keypad. Using the arrows to navigate and press 'Enter' to confirm values or toggle between 'on' and 'off'.