

## Silicon cross-section specimen preparation for transmission electron microscopy

*Proper preparation of specimens improves the quality of the resulting transmission electron microscope (TEM) images.*

### Materials

- Ruler
- Scribe
- Bulk silicon (approximately 750  $\mu\text{m}$  thick)
- Brass tube
- Acetone
- Water
- Isopropyl alcohol
- Paper towels
- Wooden stick
- Crystalbond™ adhesive
- M-Bond 610 epoxy
- EPO-TEK® 353ND epoxy or equivalent
- Silicon carbide (SiC) abrasive powder
- 1  $\mu\text{m}$  polycrystalline water-based paste
- Tweezers
- Hot plate
- Saw (for example, a diamond-wire saw)
- Fischione Model 160 Specimen Grinder
- Fischione Model 170 Ultrasonic Disk Cutter
- Fischione Model 180 XTEM Prep Kit
- Fischione Model 200 Dimpling Grinder

### Cleaving bulk silicon

To practice this cross-sectioning technique, silicon pieces can be used in lieu of a unique specimen to compile a stack of material.

1. Use a ruler and a scribe to section pieces into approximately 5 x 10 mm rectangles. The silicon stack should be tall enough to accommodate the XTEM Prep Kit's 2.3 mm diameter cutting tool when turned

on edge. The standard thickness of the bulk silicon in this example is about 750  $\mu\text{m}$ ; therefore, six pieces are sufficient.

2. Rinse the cleaved pieces in acetone, followed by isopropyl alcohol, to remove any debris or ink; place the pieces on a towel to dry. The two sides of the silicon will have a different appearance.

### Assembling the stack

1. Orient the silicon so that the two middle pieces are facing each other (i.e., shiny side to shiny side, dull side to dull side); this

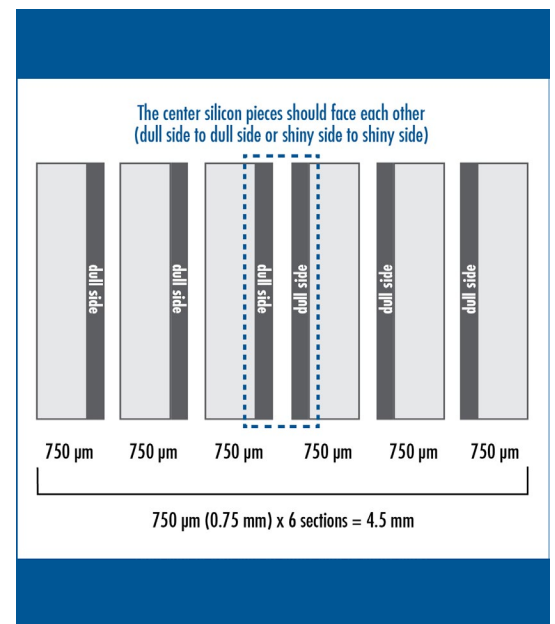


Figure 1. The two middle pieces should face each other (i.e., shiny side to shiny side or dull side to dull side).

creates twice the perimeter for the area of interest. The remainder of the silicon pieces should mirror each other (Figure 1).

If you were creating a stack from a material with a substrate, the two middle pieces would be oriented substrate side to substrate side.

2. Spread a thin coat of M-Bond 610 epoxy between each layer and attach the pieces.
3. Transfer the stack into the Model 180 XTEM Prep Kit vise assembly for curing. Tighten the vise screw to hold the stack together securely.
4. If using M-Bond 610 (or other heat-activated agent), place the vise on a 150 °C hot plate for 45 minutes to cure the epoxy.

### Coring the stack

1. Remove the vise from the hot plate and allow it to cool.
2. Loosen the set screw and gently remove the stack from the vise using a wooden stick to pry it loose.
3. Use Crystalbond adhesive to adhere the silicon stack to the stack mounting plate (Figure 2).
4. Place the stack mounting plate on the Ultrasonic Disk Cutter stage platen (Figure 3).
5. Use the microscope to center the cutting diameter over the middle interface – in this example, between silicon pieces three and four (Figure 4).
6. Apply SiC abrasive powder and water to the surface of the silicon stack and begin making a cut. When a shallow circle is visible, reevaluate the alignment using the microscope.
7. Continue to add powder and water until the stack is cut and can be removed in one piece.
8. Secure the core into a brass tube using EPO-TEK two-part epoxy. Completely fill the brass tube with epoxy. Cure the brass tube/silicon core on a hot plate.

### Cutting and grinding the tube

1. Securely chuck the brass tube containing the silicon core into a saw (e.g., a diamond-wire saw) and cut thin slices (Figure 5).

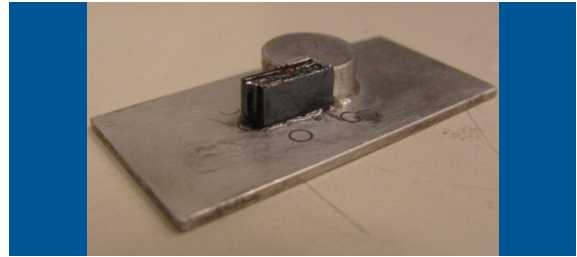


Figure 2. Silicon stack waxed to the stack mounting plate.



Figure 3. Aluminum plate and silicon stack mounted on a platen.



Figure 4. Center the cutting diameter over the third and fourth pieces in the silicon stack.

Cut the slices as thin as possible without compromising the structural integrity of the brass tube; be careful to not crack the core.

2. Use Crystalbond adhesive to adhere a silicon specimen onto a Model 160 Specimen Grinder platen; insert the platen into the grinder.

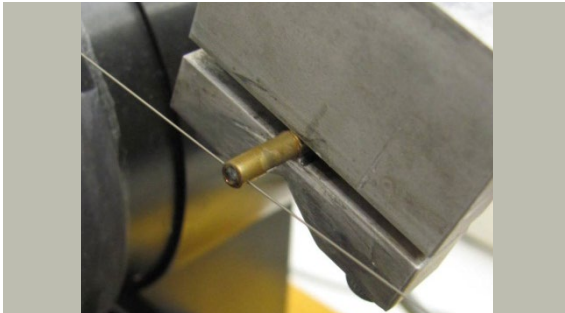


Figure 5. Cut the brass tube/silicon core into thin slices.

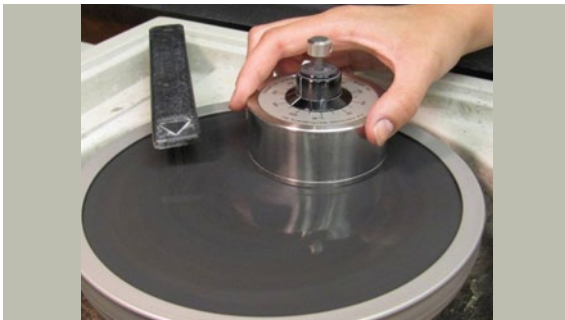


Figure 6. Use the Fischione Model 160 Specimen Grinder to grind the cross section to 100 µm thickness.

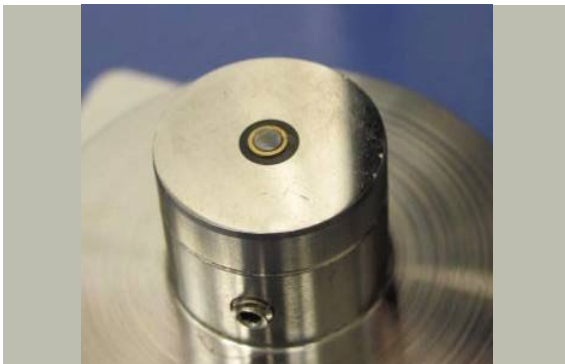


Figure 7. Place the specimen on the glass window platen.

3. Grind one side of the cross section to a 1 µm finish (Figure 6).
4. Flip the specimen over and grind this side to a 1 µm finish, making sure that the overall specimen thickness when finished is about 100 µm (some additional coarse grinding will be necessary).

### Dimple grinding

1. Transfer the specimen onto the Fischione Model 200 Dimpling Grinder glass window platen (Figure 7).
2. Line up the silicon portion of the specimen with the Dimpling Grinder's microscope attachment cross hairs.
3. Grind with 1 µm diamond paste. Add water during grinding; make sure the surface of the platen retains a slurry. Grind the specimen until achieving a thickness of about 10 µm.
4. Remove the specimen from the platen.
5. Rinse the specimen with acetone, followed by isopropyl alcohol, to remove any adhesive residue.