MODEL 180

XTEM Prep Kit

Part Number 010-0001

Instruction Manual

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Contents

Warnings and Legal Notices

Important. Read these instructions completely before installing, operating, or servicing the instrument. In addition, read the instruction manual(s) for all other Fischione products mentioned in this document; pay particular attention to all warnings and cautions in the instruction manual(s).

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Introduction

For the study of interfaces by transmission electron microscopy (TEM), it is critical to align the interface of interest parallel to the incident electron beam. One method for preparing such specimens is to fabricate cross-section (XTEM) specimens. Widely used cross-section specimens include semiconductor devices (which often have multiple layers and thus, multiple interfaces), specimens with thin film layers, and composite materials.

The Model 180 XTEM Prep Kit produces controlled XTEM specimens using rectangular wafers obtained from the area of interest of the bulk material. These wafers are readily cut by the Fischione Model 170 Ultrasonic Disk Cutter.

The XTEM Prep Kit makes it easy to stack and bond the wafers together. A small amount of vacuum-compatible adhesive is placed between each wafer. The wafers are then held in place by a vise assembly to produce a consistent adhesive layer thickness. After the adhesive has cured, the wafer stack is cored with the Model 170 Ultrasonic Disk Cutter. The core is inserted into brass tubes for subsequent sectioning into self-supported 3 mm diameter disk specimens. By sectioning the specimen and adhering several layers, each containing an interface of interest, significant amounts of information can be obtained from one specimen.

After the disks are sectioned, the Fischione Model 160 Specimen Grinder and the Model 200 Dimpling Grinder are used for final mechanical specimen preparation. Following mechanical preparation, the XTEM specimen disks are ready for ion beam specimen preparation using the Fischione Model 1050 TEM Mill.

References

The procedure described in this document uses the following Fischione products: Model 160 Specimen Grinder, Model 170 Ultrasonic Disk Cutter, Model 180 XTEM Kit, Model 200 Dimpling Grinder, and Model 1050 TEM Mill. Please refer to the instruction manuals provided with the products for operating instructions:

Document Number	Document Title
483-0002	*Model 160 Specimen Grinder Instruction Manual*
008-0141	*Model 170 Ultrasonic Disk Cutter Instruction Manual*
006-0358	*Model 200 Dimpling Grinder Instruction Manual*
041-0557-01	*Model 1050 TEM Mill Instruction Manual*

Product Description

The Model 180 XTEM Prep Kit (part number 010-0001) includes the parts illustrated in Figure 1.

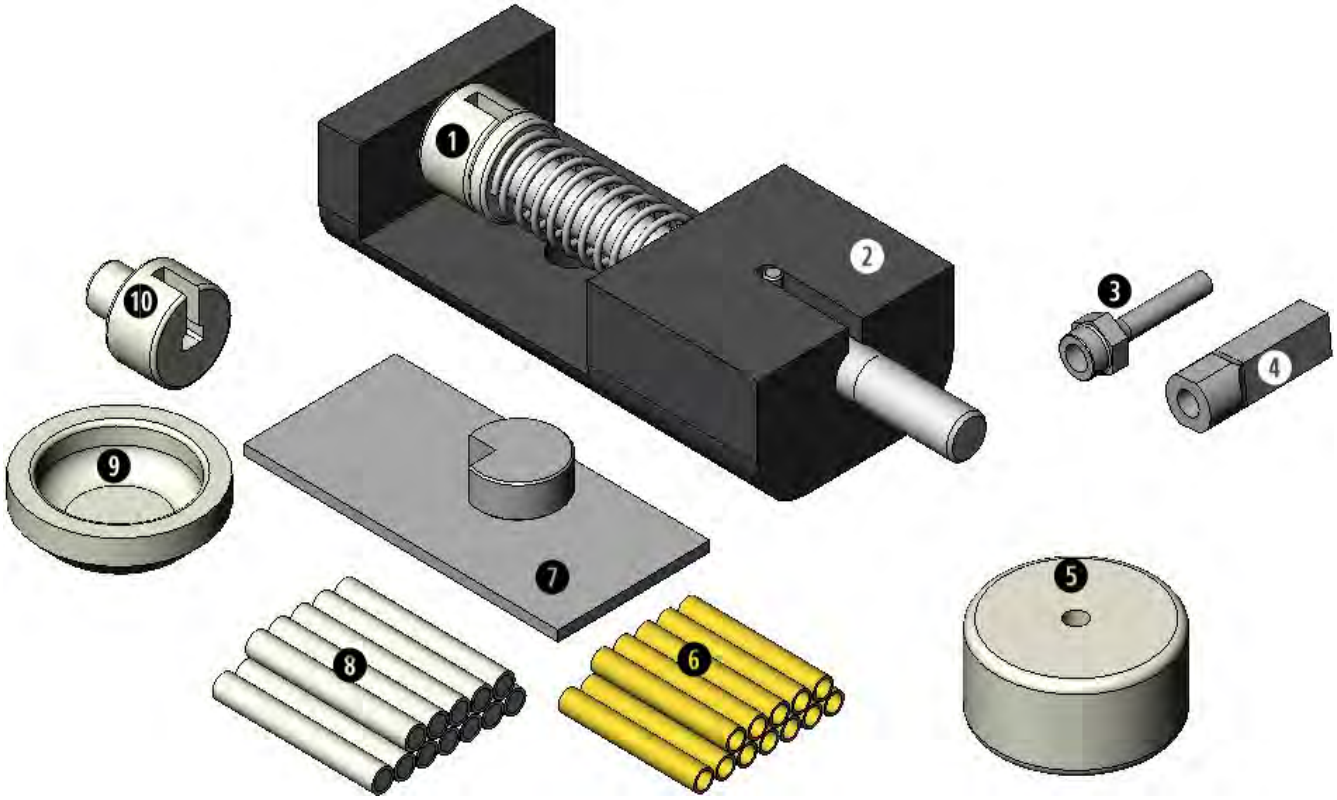

Figure 1. Model 180 XTEM Prep Kit component parts.

Number	Part Number	Description	Quantity
1	010-0005	Slice Holder; 4 mm x 5 mm; Rectangular	1
2	010-0017	Vise Assembly; Spring Type	1
3	008-0003	Titanium Tool; 2.3 mm Diameter Disk	1
4	008-0081	Titanium Tool; 4 mm x 5 mm Rectangular	1
5	010-0014	Mold	2
6	010-0020	Brass Tubes; Pack of 12	1
7	010-0008	Stack Mounting Plate Assembly	1
8	010-0018	Glue Stick; (Crystalbond™ Adhesive) Pack of 12	1
9	010-0007	Mixing Dish	3
10	010-0004	Slice Holder; 2 mm x 3 mm; Rectangular	1

Optional Parts

The following optional parts may be ordered from Fischione for use with the Model 180 XTEM Prep Kit.

Part Number	Description	Quantity
007-0016	Goniometer Platen	1
010-0021	Specimen Mounting Hot Plate Assembly; 110 VAC	1
010-0032	Specimen Mounting Hot Plate Assembly; 220 VAC	1

Consumables and Spare Parts

The following consumable and spare parts may be ordered from Fischione for use with your Model 180 XTEM Prep Kit.

Part Number	Description	Quantity
008-0003	Titanium Tool; 2.3 mm Diameter Disk	1
008-0078	Titanium Tool; 2 mm x 3 mm Rectangular	1

Part Number	Description	Quantity
008-0081	Titanium Tool; 4 mm x 5 mm Rectangular	1
010-0004	Slice Holder; 2 mm x 3 mm; Rectangular	1
010-0005	Slice Holder; 4 mm x 5 mm; Rectangular	1
010-0006	Contact Pin	1
010-0007	Mixing Dish	1
010-0008	Stack Mounting Plate Assembly	1
010-0014	Mold	1
010-0017	Vise Assembly; Spring Type	1
010-0018	Glue Stick; (Crystalbond Adhesive) Pack of 12	1
010-0020	Brass Tubes; Pack of 12	1
010-0022	Vise Assembly; Screw Type	1
306-0002	Adhesive; Crystalbond; Type 509; One Stick	1

Preparing a Cross Section for TEM

Equipment

- Model 160 Specimen Grinder
- Model 170 Ultrasonic Disk Cutter
- Model 180 XTEM Prep Kit
- Model 200 Dimpling Grinder
- Model 1050 TEM Mill
- Mechanical grinding/ polishing machine
- Wire saw
- Digital measuring system
- 7/64 in. Allen wrench (optional)
- Glass cover slips
- Hot plate
- Glass vials

Consumables

- Crystalbond 509 adhesive
- M-Bond 610 adhesive
- EPO-TEK® 353ND adhesive
- Silicon carbide (SiC) grinding paper, 600 (14.5 µm) grit
- Diamond lapping films (6, 3, and 1 µm)
- SiC powder, 600 grit
- Diamond polishing paste (6, 3, and 1 µm)
- Acetone
- Deionized water
- Lint-free paper
- Cotton swabs
- Toothpicks

Preparing the Specimen Sections

1. Cleave or cut the specimen to a size that will fit on the Model 170 Ultrasonic Disk Cutter's Aluminum specimen slide. Cut the specimen so that the area of interest falls near the center.

   When cutting the specimens, keep in mind that you will be assembling the XTEM specimen from six wafers that are 750 µm thick after grinding and polishing.

2. Set the hot plate temperature to 150° C. If the specimen:

   — **has a pattern,** mount the specimen with Crystalbond 509 adhesive to the Aluminum slide, device side up.

   Cover the specimen with a glass cover slip; use the Crystalbond adhesive to adhere the cover slip to the specimen.

   — **does not have a pattern,** mount the specimen face down on the Aluminum slide.

3. Insert one of the following rectangular cutting tools into the Model 170 Ultrasonic Disk Cutter:

   — 4 mm x 5 mm

   — 2 mm x 3 mm

4. Cut the area of interest from the mounted wafer.

5. Use 600-grit SiC powder for cutting.

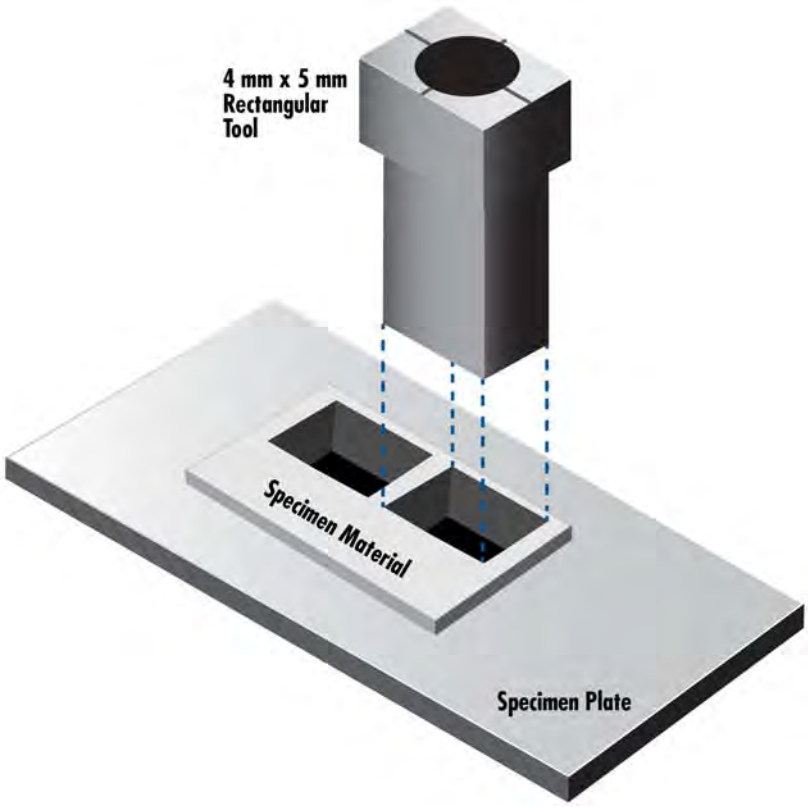

Figure 2. Cutting rectangular sections for XTEM specimen preparation.

6. If Crystalbond adhesive was used, heat the specimen sections on a hot plate to remove the glass cover slip.
7. Use acetone to remove all traces of Crystalbond adhesive. Rinse the sections several times with acetone.
8. Mount the sections substrate down on the Model 160 Specimen Grinder and back-side thin:
   a. Thin to approximately 200 µm with 600-grit SiC paper.
   b. Thin to final thickness in steps using diamond lapping films in the following order: 6 µm, 3 µm, 1 µm. Remove at least 25 µm per step.

   The thinned sections should now be 750 µm thick.
9. Add acetone to a clean, glass vial.
10. Remove the sections from the specimen grinder.

11. Soak the sections in the acetone to remove any Crystalbond adhesive. Rinse several times.
12. Pour off the excess acetone and place the glass vial containing the sections on a hot plate to evaporate the remaining acetone.
13. Place a piece of lint-free paper on a clean work surface.
14. Carefully empty the vial contents onto the lint-free paper. The sections are fragile; handle the sections as little as possible.

Assembling the XTEM Stack

The assembled stack of 750 µm sections will be 4.5 mm x 5 mm x 5 mm.

1. Follow the manufacturer's directions to mix the M-Bond 610 adhesive.
2. Arrange the sections to prepare to build the XTEM stack; handle the fragile sections as little as possible.

   Be certain to place the ceramic or Silicon substrate surfaces of the center 2 sections face-to-face (see Figure 3).

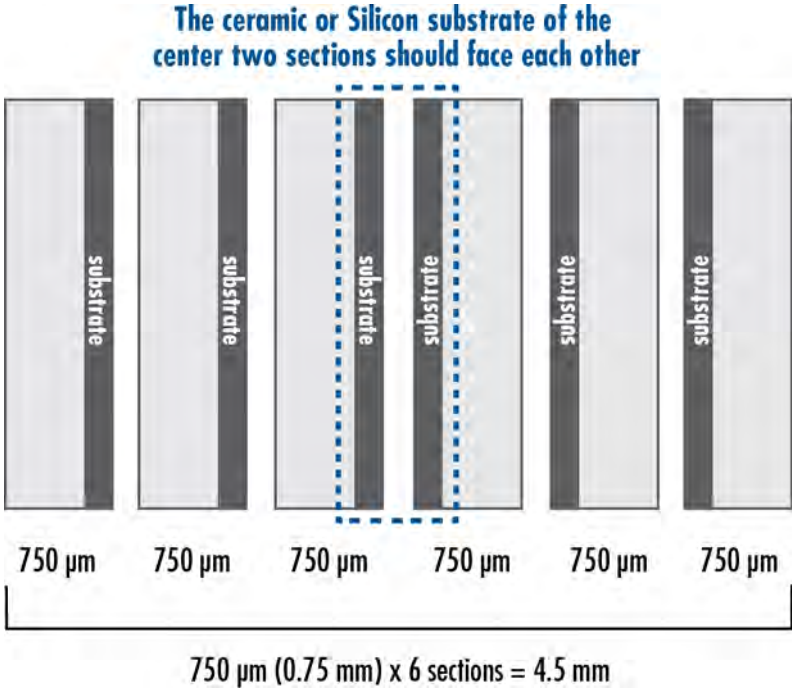

Figure 3. Specimen section layout to create the XTEM stack.

3. Apply M-Bond 610 adhesive to the inside surface of the first section and place it in the 4 mm x 5 mm slice holder.

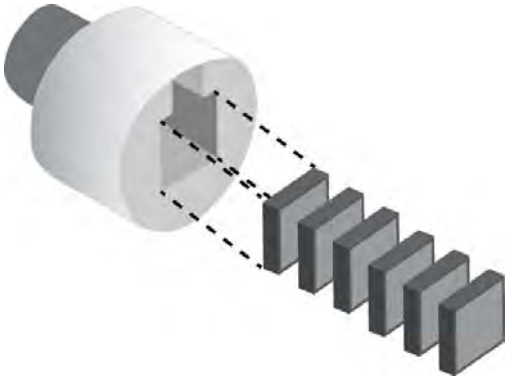

Figure 4. Use M-Bond 610 adhesive to adhere the sections together; place each section in the 4 x 5 mm slice holder.

4. Repeat with the remaining sections until the complete stack is built. Use enough M-Bond 610 adhesive to completely cover the pieces.
5. Wait 5 minutes for the adhesive to set.
6. Place the XTEM stack and the slice holder into the vise assembly.

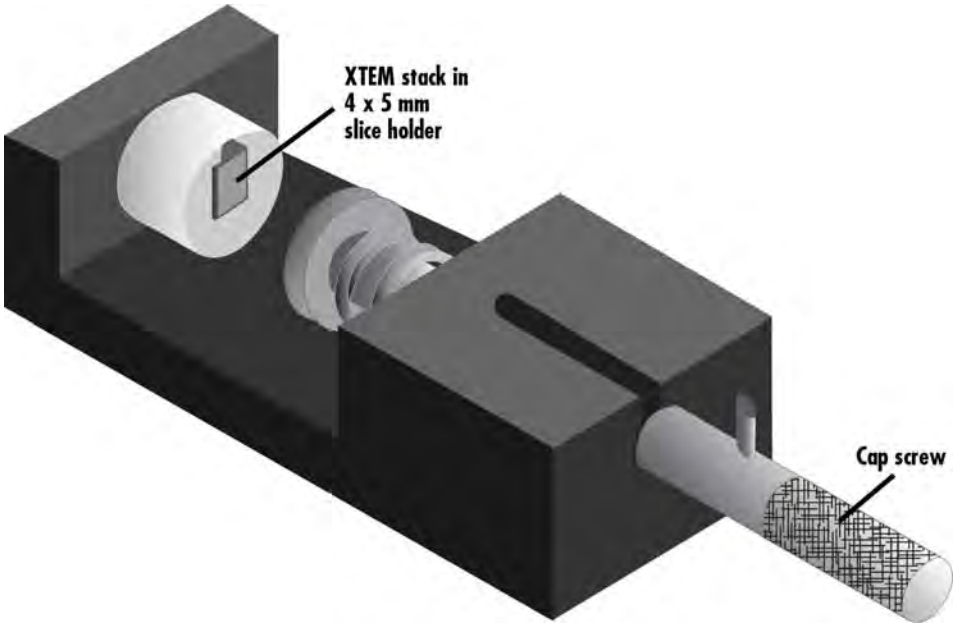

Figure 5. Place the 4 x 5 mm slice holder with the XTEM stack in the vise assembly.

7. Turn the vise assembly cap screw; the vise assembly spring will release and maximum force will be applied to the XTEM stack in the slice holder.
8. Turn the vise assembly cap screw until finger tight or use a 7/64 in. Allen wrench to tighten the cap screw until snug.
9. Place the entire vise on the hot plate to cure the adhesive for approximately 40 minutes.
10. Remove the rectangular tool from the Model 170 Ultrasonic Disk Cutter and replace it with the 2.3 mm diameter disk tool.
11. Make a partial cut into a piece of scrap Silicon with the 2.3 mm diameter disk tool.
12. Move the alignment microscope into place.
13. Looking through the microscope, align the microscope with the partially cut circle in the scrap Silicon; use the 4 round thumb screws at the base of the microscope eyepiece so that the circle is in the center of the field of view.

    The microscope is now aligned with the 2.3 mm diameter disk tool.
14. Remove the scrap Silicon from the Model 170 Ultrasonic Disk Cutter.
15. Remove the XTEM stack from the vise and the slice holder and place the stack on edge in the stack mounting plate.
16. Use Crystalbond adhesive to secure the XTEM stack in place.

Coring the XTEM Stack

1. Place the stack mounting plate under the disk cutter and, using the alignment microscope, move the specimen until the center of the cross section is in the center of the field of view.

   Do not cut too close to the wedge-shaped walls of the specimen mounting plate because proximity of the cutting tool to these walls will automatically terminate cutting.
2. Cut the 2.3 mm diameter core from the stack using 600-grit SiC powder. If cutting slows while coring the specimen, add abrasive near the disk tool and push and pull lightly on the plunger of the cutting water assembly. This will allow cutting to continue without moving the disk tool and possibly changing the cutting course. If the process of cutting slows, place additional abrasive near the cut with a cotton swab.

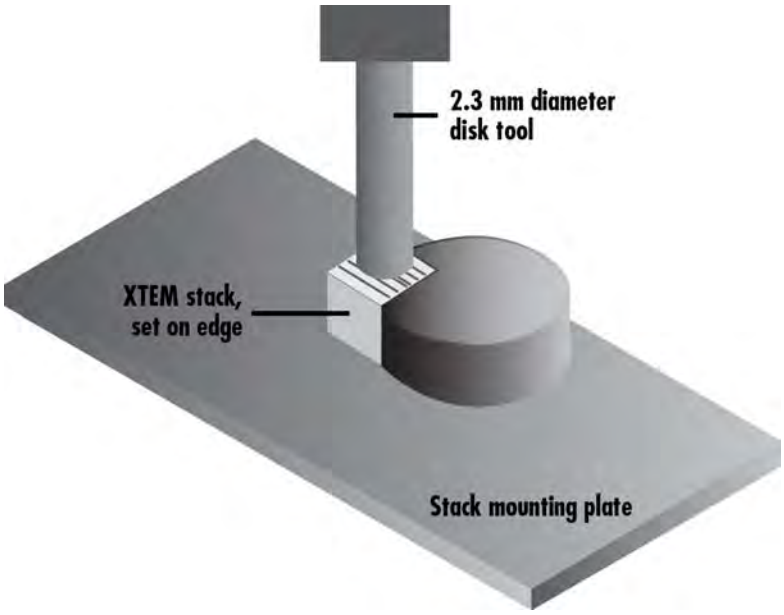

Figure 6. Use the 2.3 mm diameter disk tool to core the XTEM stack.

3. Soak the 2.3 mm core in acetone for 5 to 10 minutes. Soak up to 30 minutes for optimal cleaning of Crystalbond adhesive from the top and bottom of the core.
4. Fix the core in a 3 mm brass tube:
   a. In the supplied mixing dish, mix EPO-TEK 353ND adhesive. Follow the manufacturer's directions for mixing.
   b. Insert a brass tube in the mold to hold it upright.
   c. Use a toothpick to coat the inside of the brass tube with EPO-TEK 353ND adhesive.
   d. Insert the stack core into the brass tube.
   e. Completely fill the brass tube with adhesive.
   f. Place the mold upright on a hot plate and allow the adhesive to cure for at least 40 minutes at approximately 150° C.

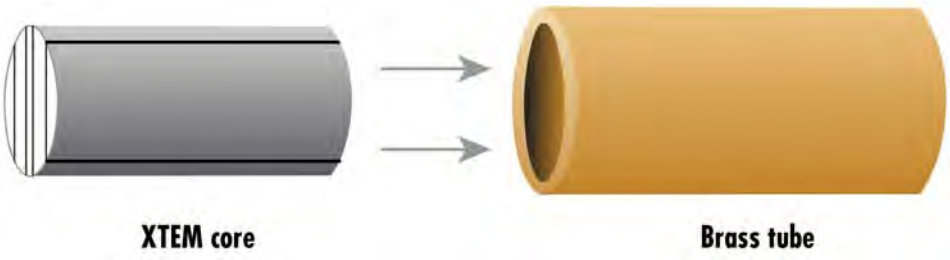

Figure 7. Insert the XTEM core into the 3 mm brass tube.

5. Using a wire saw, cut the brass tube into 400 to 500 µm thick disks. You may cut thinner sections, if desired.

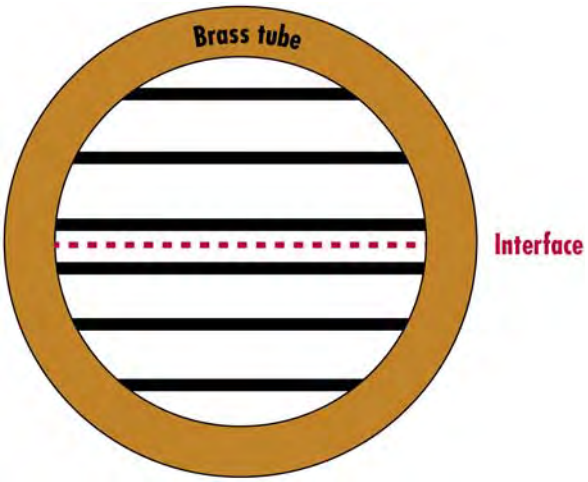

Figure 8. Disk cut from 3 mm brass tube with XTEM stack core.

Polishing and Dimple Grinding XTEM Disks

1. Mount one thick disk to a glass-topped platen and use the Model 160 Specimen Grinder to polish one side of the disk to a 1 µm diamond finish:

   a. Thin to approximately 200 µm with 600-grit SiC paper.

   b. Thin to final thickness in steps using diamond lapping films in the following order: 6 µm, 3 µm, 1 µm. Remove at least 25 µm per step.

      Total specimen thickness at this point does not matter, but for safe removal of damage, final thickness after polishing one side of the disk should be approximately half of the starting thickness.

2. Remove the disk from the specimen grinder.
3. Remove the bulk material from the back side of the disk using either the Model 160 Specimen Grinder or the Model 200 Dimpling Grinder.

   Model 160 Specimen Grinder

   a. Turn over the disk and place it on the specimen grinder platen, polished-side down.

   b. Thin to approximately 200 µm with 600-grit SiC paper.

   c. Thin to final thickness using diamond lapping films in the following order: 6 µm, 3 µm, and 1 µm.

      Remove 25 µm of specimen thickness with each step. Aim for a final disk thickness of < 100 µm.

   d. Remove the specimen from the platen and use a micrometer to measure the specimen thickness. Adhere the specimen to the platen after measuring.

   e. Remove the specimen platen from the specimen grinder and place it on the Model 200 Dimpling Grinder. Continue on to step 4.

   Model 200 Dimpling Grinder

   a. Turn the disk over and place it on the specimen platen polished-side down and then place the platen on the dimple grinder.

   b. Set the counterweight to 50 g.

   c. Using the flattening wheel, remove specimen material with diamond polishing paste in the following order: 6 µm, 3 µm, and 1 µm. Remove at least 25 µm of specimen material per step.

      Aim for a final disk thickness of < 100 µm.

   d. Remove the specimen from the platen and use a micrometer to measure the specimen thickness. Adhere the specimen to the platen after measuring and return the platen to the dimpling grinder.
4. Select the dimpling wheel you wish to use and place it on the grinding wheel spindle. Refer to *Model 200 Dimpling Grinder Instruction Manual* (document number 006-0358) for information on the choice of grinding wheels and grinding parameters.
5. Zero the grinding wheel on the top of the specimen.
6. Set the counterweight to 50 g, if you have not done so already.

7. Dimple the specimen using 1 µm diamond paste until the specimen is 30 µm thick.
8. Clean the dimple wheel well.
9. Continue to remove material using 1 µm diamond paste until the specimen is 20 to 25 µm thick.
10. Check the condition of the specimen with the microscope.
11. Clean the dimple wheel well and dimple the specimen until the first sign of transmitted light shows through the specimen (approximately 10 µm) and the specimen is deep red or brown in color.

    Transmitted lights and colors will only be seen in plan view or cross-section specimens that contain Silicon.
12. Clean the dimple wheel well and add more 1 µm diamond paste.
13. Dimple the specimen until the specimen is dark orange in color (approximately 7 to 8 µm).
14. Continue dimpling carefully with 1 µm diamond paste until the specimen is bright orange in color.
15. Remove the specimen platen from the dimpling wheel.
16. Carefully remove the specimen from the platen by either:

    — Placing the specimen on the hot plate and removing the specimen when the Crystalbond adhesive becomes molten.

    — Soaking the entire specimen platen in acetone until the specimen falls off.

    The second option is the safest and requires approximately 30 minutes.
17. Soak the specimen in acetone to remove any remaining Crystalbond adhesive.
18. Rinse the specimen several times.

Ion Beam Milling

1. Mount the specimen on the Model 1050 TEM Mill Dual-Post Specimen Holder.
2. Insert the specimen holder into the TEM Mill stage and close the load lock.
3. Set the milling parameters:

    — Set both the left and right ion beams to 4 kV

— Set one beam angle to +10°; set the second ion beam angle to -10°

— Set the specimen rotation to ±35° rocking

— Set the rotation offset to 90°

4. Start the ion million operation; continue the milling operation and add additional milling steps at lower voltages, as required, until the specimen is perforated.