APPLICATIONOTE

Silicon plan view specimen preparation for transmission electron microscopy

Proper preparation of samples for ion milling increases milling process speed and improves the quality of the resulting transmission electron microscope (TEM) images.

This application note describes how to prepare a silicon wafer that is 100 µm thick, 3 mm in diameter, with a 1 µm finish on both sides.

Materials

- Fischione Model 160 Specimen Grinder
- Fischione Model 170 Ultrasonic Disk Cutter
- Fischione Model 200 Dimpling Grinder
- Mechanical grinding/ polishing machine
- Digital measuring system
- Glass plate
- Hot plate
- Glass beakers
- Bulk silicon (approximately 750 µm thick)
- Crystalbond™ 509 mounting adhesive (melting point 121° C)
- Grinding paper, 400 (22.1 µm) and 600 (14.5 µm) grit (see Figure 1)
- Cubic boron nitride (CBN) or silicon carbide paste, 600 grit
- Diamond paste (6, 3, and 1 µm)
- Diamond paste diluent (e.g., MetaDi diamond paste)
- Colloidal silica drops
- Ethanol
- Acetone
- Deionized water

Parallel grinding and polishing

This section explains how to produce a 3 mm diameter disk from bulk silicon wafer with 1 µm finish on both sides of the specimen that is suitable for dimple grinding.

Preparing to cut the specimen

1. Turn on the hot plate and set it to 140° C.
2. Use a digital measuring device to measure the specimen thickness, e.g., 750 µm.
3. On the Model 170 Ultrasonic Disk Cutter, mount a 3 mm cutting tool on the transducer using the supplied copper washer; use the supplied wrench to tighten the washer (see Figure 2).

Grit Number	Size (µm)	Grit Number	Size (µm)
P60	269.0	60	268.0
P80	201.0	80	188.0
P100	162.0	100	148.0
P120	127.0	120	116.0
P180	78.0	180	78.0
P240	58.5	220	66.0
P280	52.2	240	51.8
P320	46.2		
P360	40.5	280	42.3
P400	35.0	320	34.3
P500	30.2	360	27.3
P600	25.8	400	22.1
P800	21.8		
P1000	18.3	500	18.2
P1200	15.3	600	14.5
P1500	12.6	800	12.2
P2000	10.3	1000	9.2
P2500	8.4	1200	6.5
P4000	5.0		

Figure 1. Grinding paper grit and surface quality reference.

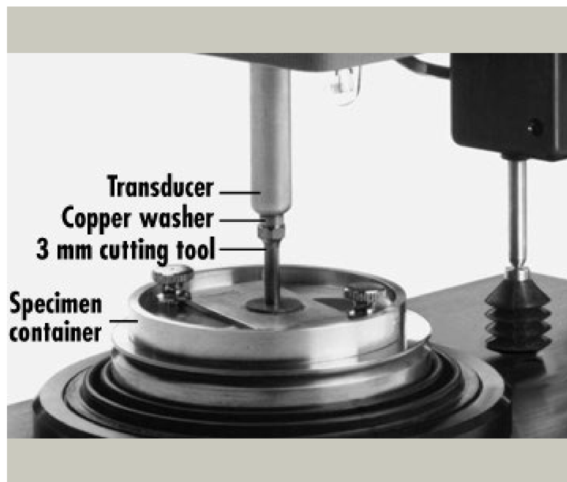

Figure 2. Mount 3 mm cutting tool on to the disk cutter.

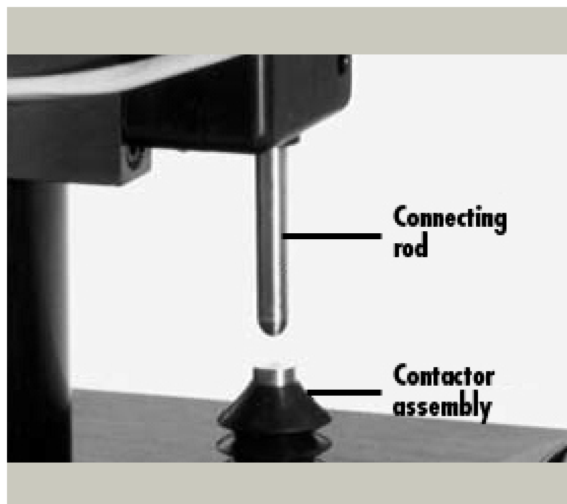

Figure 3. Add the 10.54 mm extender to the indicator contactor assembly.

Part Number	Height (in)	Height (mm)
008-0071	.175	4.45
008-0072	.255	6.48
008-0073	.335	8.51
008-0074	.415	10.54
008-0075	.495	12.57

Figure 4. Extenders supplied with the Fischione Model 170 Ultrasonic Disk Cutter.

4. Place the specimen plate on the hot plate.
5. Obtain enough Crystalbond adhesive to adhere a cleaved piece of silicon wafer.
6. Place the wafer polish-side down into the adhesive.
7. Press the wafer until the adhesive can be seen all around the specimen.

   If you did not use enough adhesive, then repeat the process. Be certain to press the specimen into the adhesive until all the air is removed between the specimen and the adhesive; air under the specimen may cause the specimen to break.
8. Remove the specimen plate from the hot plate and place the specimen plate in a safe place to cool. A metallic block is ideal for cooling.
9. Fix the specimen plate on the specimen container with thumb screws.
10. Add the 10.54 mm extender to the indicator contactor assembly. The choice of extender sizes will vary depending on the specimen thickness; however, the extender allows the measurement range to be extended (see Figure 3).

Cutting the specimen

1. Lower the cutting tool to the surface of the specimen.
2. Ensure that the left-hand switch is in the middle position (between AUTO and CONT).
3. Put the power switch in the ON position.
4. Press the switch to CONT position for a few seconds and then raise the cutting tool.
5. Loosen the locking knob and rotate the microscope into position.
6. Tighten the locking knob.
7. Confirm that the microscope is centered on the cutting tool.

If the microscope is not centered, then adjust the microscope (see the *Model 170 Ultrasonic Disk Cutter Instruction Manual,* document number 008-0141).

8. Reposition the specimen area for cutting; ensure that the area of interest is centered.
9. Loosen the locking knob and rotate the cutting tool into position.
10. Tighten the locking knob.
11. Add water into the syringe and connect the syringe to the disk cutter.
12. Add some CBN or 600-grit silicon carbide paste to the surface of the silicon.
13. Use the syringe to put a drop of water on the end of the cutting tool.
14. Lower the cutting tool to the surface and push down the specimen so that some spring pressure is applied.
15. Set the dial indicator to the zero position (make sure the dial clamp is free). Make sure to place the appropriate extender on the disk cutter's indicator contactor assembly; otherwise, the reading will be incorrect.
16. Switch to AUTO cutting and watch the dial indicator to monitor the cutting progress.
17. Pause the process and adjust the water and clamping pressure, as needed.
18. Once cutting has stopped (cutting exceeds specimen thickness of 750 µm), switch to CONT mode and cut for an additional 10 to 20 µm and/or until the light is a solid red color.
19. Raise the cutting tool and position a new area ready for cutting and repeat the procedure.
    a. If the disk cannot be found, use the syringe to stream water through the cutting tool to remove the disk.
    b. If the water fails to dislodge the disk, toggle the switch between CONT and the center position a few times to try to dislodge the disk.
    c. If toggling the switch fails to dislodge the disk, remove the cutting tool to extract the specimen and then reattach the cutting tool.

Mounting and grinding the specimen disks

1. When cutting is complete, remove the specimen plate and put it on the hot plate.
2. When the adhesive has melted, remove the bulk silicon and extract the 3 mm disks.
3. Put the disks into acetone for 10 minutes.
4. Place a specimen grinder platen on the hot plate and then add small piece of Crystalbond adhesive.
5. Press a 3 mm disk on the platen with the polished side down.
6. Allow the adhesive to cool.
7. Use a digital measuring device to confirm the thickness of the adhesive.

   The adhesive thickness is the total height above the platen, minus the specimen thickness, e.g., 750 µm.
8. Place the platen in the Model 160 Specimen Grinder.
9. Turn the black knob counter-clockwise to retract the platen into the grinder.
10. Place the specimen grinder on a hard, flat surface.
11. While applying downward pressure on the specimen grinder, turn the black knob clockwise to advance the platen.

    When the knob will no longer turn or becomes very difficult to turn, the platen surface is flush with the wear plate of the specimen grinder.
12. To set this position as zero, rotate the numbered flat plate until the 0 reading on the plate aligns with the marked line on the black knob. Do not turn the black knob at this time, only the numbered plate.

13. Turn the black knob clockwise 25 µm to advance the specimen.
14. Place the specimen grinder on a standard grinding wheel with a selected abrasive (typically 600-grit SiC) to remove material. No downward force is needed due to the weight of the specimen grinder.
15. When the desired material is removed, verify that the specimen is flush with the specimen grinder's wear plate.
16. Confirm the specimen thickness by removing the platen from the specimen grinder and using the digital measuring device. The specimen grinder is only a guide to thickness removal.

Advancing to 200 µm thickness

1. Reinsert the platen into the specimen grinder and advance the specimen until 200 µm remains.
2. Remove the platen and use the digital measuring device to confirm the specimen thickness.
3. Clean the specimen grinder and platen in water.
4. Reinsert the platen into the specimen grinder and rezero the specimen.
5. Place the specimen grinder on a standard, 6 µm lapping wheel to remove material in 25 µm increments. No downward force is needed due to the weight of the specimen grinder.
6. When the desired material is removed, verify that the specimen surface is flush with the specimen grinder's wear plate.
7. Remove the platen and use the digital measuring device to confirm the specimen thickness.

Advancing to 150 µm thickness

1. Reinsert the platen into the specimen grinder and advance the specimen until 150 µm remains.
2. Remove the platen and use the digital measuring device to confirm the specimen thickness.
3. Clean the specimen grinder and platen in water.
4. Reinsert the platen into the specimen grinder and rezero the specimen.
5. Place the specimen grinder on a standard, 3 µm lapping wheel to remove material in small increments. No downward force is needed due to the weight of the specimen grinder.
6. When the desired material is removed, verify that the surface is flush with the specimen grinder's wear plate.
7. Remove the platen and use the digital measuring device to confirm the specimen thickness.

Advancing to 125 µm thickness

1. Reinsert the platen into the specimen grinder and advance the specimen until 125 µm remains.
2. Remove the platen and use the digital measuring device to confirm the specimen thickness.
3. Clean the specimen grinder and platen in water.
4. Reinsert the platen into the specimen grinder.
5. Place the specimen grinder on a standard, 1 µm lapping wheel to remove material in small increments. No downward force is needed due to the weight of the specimen grinder.
6. When the desired material is removed, verify that the surface is flush with the specimen grinder's wear plate.
7. Remove the platen and use the digital measuring device to confirm the specimen thickness.

Advancing to 100 µm thickness

1. Reinsert the platen into the specimen grinder and advance the specimen until 100 µm remains.

2. Remove the platen and use the digital measuring device to confirm the specimen thickness.
3. Clean the specimen grinder and platen in water.
4. Rinse the specimen grinder and platen in ethanol.
5. To extract the specimen, place the platen in acetone for approximately 30 minutes or more to dissolve the adhesive.

Dimple polishing

This section explains how to dimple polish a parallel-polished 3 mm specimen with a starting thickness of 100 µm to a final thickness of 10 µm (surface finish of < 1 µm).

Mounting the Specimen on the Model 200 Dimpling Grinder

1. Adjust the counter weight to 50 g.
2. Switch on the dimpling grinder and allow it to initialize with the dimpling wheel lowered.
3. Set the GRINDING WHEEL SPEED and the SAMPLE ROTATION SPEED to approximately 40% of the total speed.
4. Lift and park the dimpling wheel.
5. Loosen the setscrew on the dimpling grinder's magnetic platen holder.
6. Select a grinding wheel (stainless steel) and place it on the dimpling grinder, holding it in place with the wheel locking nut.
7. Select a glass platen and place it on a hot plate. Because you need to observe the specimen during the dimple grinding process you will use the glass platen holder, which allows the dimpling grinder lamp to shine through the platen so that you can gauge the translucence of the specimen.
8. Place a small amount of Crystalbond adhesive on the platen surface and allow it to melt.
9. Place the specimen over the adhesive and apply gentle pressure to minimize the adhesive layer thickness.

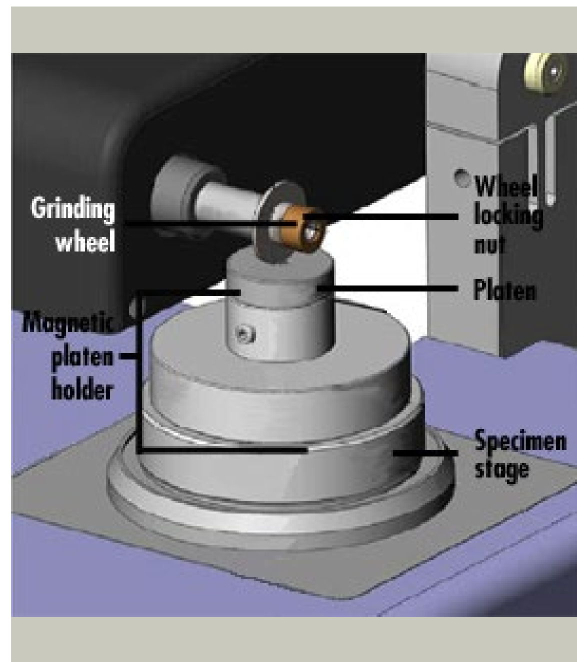

Figure 5. Model 200 Dimpling Grinder schematic.

10. Remove the platen from the hot plate and allow it to cool.
11. Ensure that both the platen holder and magnetic platen holder are completely clean.
12. Install the platen into the magnetic platen holder and tighten the setscrew. Ensure that the setscrew is aligned with the slot in the platen before tightening (see Figure 5).
13. Center the magnetic platen holder to the specimen stage using the centering ring.
14. Mount the microscope.
15. Check that the specimen disk is centered.

    If not, reposition the specimen disk without the centering ring.
16. Lower the dimpling wheel.
17. Zero on the specimen surface.

    When the zeroing is complete, the wheel will automatically raise 50 µm. If a mark is left on the specimen surface, use the microscope to verify that it is centered.

Dimple grinding the specimen

Advancing to -10 µm

1. If the wheel is raised, lower it into place.
2. Add 6 µm diamond paste and 2 drops of diluent. During the grinding process, monitor the platen surface; keep the surface wet, adding diluent or water as needed.
3. Lower the dimpling wheel.
4. Select the DEPTH-BASED mode.
5. Select a depth of -10 µm.
6. Start dimple grinding.
7. When the process stops and the wheel raises, lift the dimpling wheel.
8. Check that the dimple is centered with the microscope. Adjust if needed.

Advancing to -50 µm

1. Add 6 µm diamond paste and 2 drops of diluent. During the grinding process, monitor the platen surface; keep the surface wet, adding diluent or water as needed.
2. Lower the dimpling wheel.
3. Select DEPTH-BASED mode.
4. Select a depth of -50 µm.
5. Resume dimple grinding.
6. When the process stops and the wheel raises, lift the dimpling wheel.

Advancing to -75 µm

1. Add 3 µm diamond paste and 2 drops of diluent. During the grinding process, monitor the platen surface; keep the surface wet, adding diluent or water as needed.
2. Lower the dimpling wheel.
3. Select DEPTH-BASED mode.
4. Select a depth of -75 µm.
5. Resume dimple grinding.
6. When the process stops and the wheel raises, lift the dimpling wheel.

Advancing to -80 µm

1. Add 1 µm diamond paste and 2 drops of diluent. During the grinding process, monitor the platen surface; keep the surface wet, adding diluent or water as needed.
2. Lower the dimpling wheel.
3. Select DEPTH-BASED mode.
4. Select a depth of -80 µm.
5. Resume dimple grinding.
6. When the process stops and the wheel raises, lift the dimpling wheel.
7. Remove any excess polishing paste from the specimen.
8. Fit the lamp beam block carefully on top of the specimen.
9. Turn the BOTTOM LIGHT control clockwise to the maximum setting. If a red color is not visible, then the specimen is probably not viable. Restart with a fresh specimen because the polishing damage from 3 µm will be present in the specimen.
10. Turn the BOTTOM LIGHT control counterclockwise to turn the light off .

Advancing to -85 µm

1. Add some 1 µm diamond paste, if needed, and 2 drops of diluent. During the grinding process, monitor the platen surface; keep the surface wet, adding diluent or water as needed.
2. Lower the dimpling wheel.
3. Select DEPTH-BASED mode.
4. Select a depth of -85 µm.
5. Resume dimple grinding.
6. When the process stops and the wheel raises, lift the dimpling wheel.
7. Fit the lamp beam block carefully on top of the specimen.
8. Turn the BOTTOM LIGHT control clockwise to the maximum setting. If a red color is visible, skip to *Dimple polishing the specimen*, on page 7.

9. Turn the BOTTOM LIGHT control counterclockwise to turn the light off .

Advancing to -90 µm

1. Add some 1 µm diamond paste, if needed, and 2 drops of diluent. During the grinding process, monitor the platen surface; keep the surface wet, adding diluent or water as needed.
2. Lower the dimpling wheel.
3. Select DEPTH-BASED mode.
4. Select a depth of -90 µm.
5. Resume dimple grinding.
6. When the process stops and the wheel raises, lift the dimpling wheel.
7. Fit the lamp beam block carefully on top of the specimen.
8. Turn the BOTTOM LIGHT control clockwise to the maximum setting. If a red color is not visible, repeat this procedure (*Advancing to -90 µm*); however, set the depth to -93 µm.
9. Turn the BOTTOM LIGHT control counterclockwise to turn the light off .

Dimple polishing the specimen

1. Change the dimpling wheel to a semiconductor polishing wheel.
2. Add colloidal silica drops to the specimen. During the polishing process, monitor the platen surface; keep the surface wet, adding colloidal silica drops as needed.
3. Select TIME-BASED mode.
4. Press START.
5. Polish the specimen for 5 to 10 minutes.
6. Clean the specimen.
7. Check the specimen color under the microscope. The specimen color should change to orange at approximately 8 or 7 µm of Si. The absolute values will vary with light source type.
8. Repeat steps 2 to 6 and polish the specimen for an additional 5 minutes.

   During the polishing process, monitor the platen surface; keep the surface wet, adding colloidal silica drops as needed. After each time gap, check the specimen under the microscope.
9. To extract the specimen, place the platen in acetone for approximately 1 hour to dissolve the adhesive. The polished specimen is delicate; do not use tweezers or the hot plate to remove the platen.

   The specimen is ready to be ion milled.

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Document Number AN001 Revision 00 10/2013