TEM SAMPLE-PREPARATION PROCEDURES FOR THIN-FILM MATERIALS

Initial Set-Up:
Heat up a hot plate to around 150-200 °C

Plan view
Mounting/Grinding/Dimpling/Polishing:
1) Cleave a square-ish piece of sample. Place the piece, film side down, on a glass slide on the optical microscope stage.

2) Pick out a Cu grid (usually Pelco 1GC10H or 1GC12H aperture grids) and place next to the sample. Ideally, the aperture sample should just cover the aperture when centered on the grid.

3) Prepare some silver paste (epo-tek H20E, 1:1 hardener:resin).

4) Using a pin or toothpick, place some small drops of glue on the edges of the grid. You will need to hold the grid down with high-quality tweezers.

5) Pick up the sample from with the tweezers and place on the grid, film side down. Try not to slide the sample around, so the glue stays just on the edges.

6) Pick up the glass slide and place on the hot plate. Push the sample/grid around occasionally so it does not adhere to the glass. The glue should harden in one or two minutes.

7) Mount the sample/grid on a dimpler platen with CrystalBond (clear) wax. First heat the platen on the hot plate. We have two dimplers, which use different types of platens:

a) The VCR D500 Dimpler uses a metal platen with a glass circle in the middle. Put a circular cover slip on the platen with wax.

b) The Gatan Dimple/Grinder uses clear glass, cylindrical blocks. These are also compatible with the Gatan Model 623 Disc Grinder.

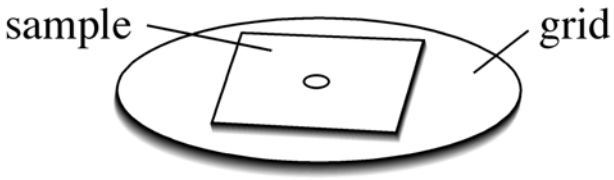
Center the grid on the platen with more wax.

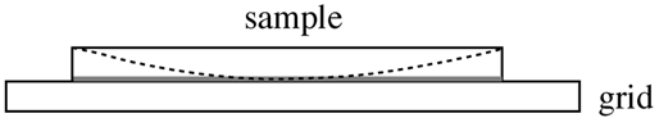
8) Grind the sample to about 100 μm total thickness. That is the sample should be about the same thickness as the grid it is attached to.

9) Dimple to near perforation. It's usually OK if you get a small hole.

10) Place the platen on the hot plate. When the wax is soft, push the sample off the platen with a toothpick and quickly immerse in acetone. Cover and let sit for at least several minutes to remove the wax. Do not allow the acetone to evaporate with the sample immersed, as this will leave a residue on the sample that is hard to remove.

Pull the sample out of the acetone from the edges with high-quality tweezers and immediately drop onto a lint-free wipe or filter paper to absorb the acetone. It helps to turn the sample over a couple of times to quickly dry both sides.





Store the sample in a grid holder or small vial. Be sure to label the sample.

Ion Milling:
Mill from the substrate side with rotation and low angle. Reduce the angle and current as you go.

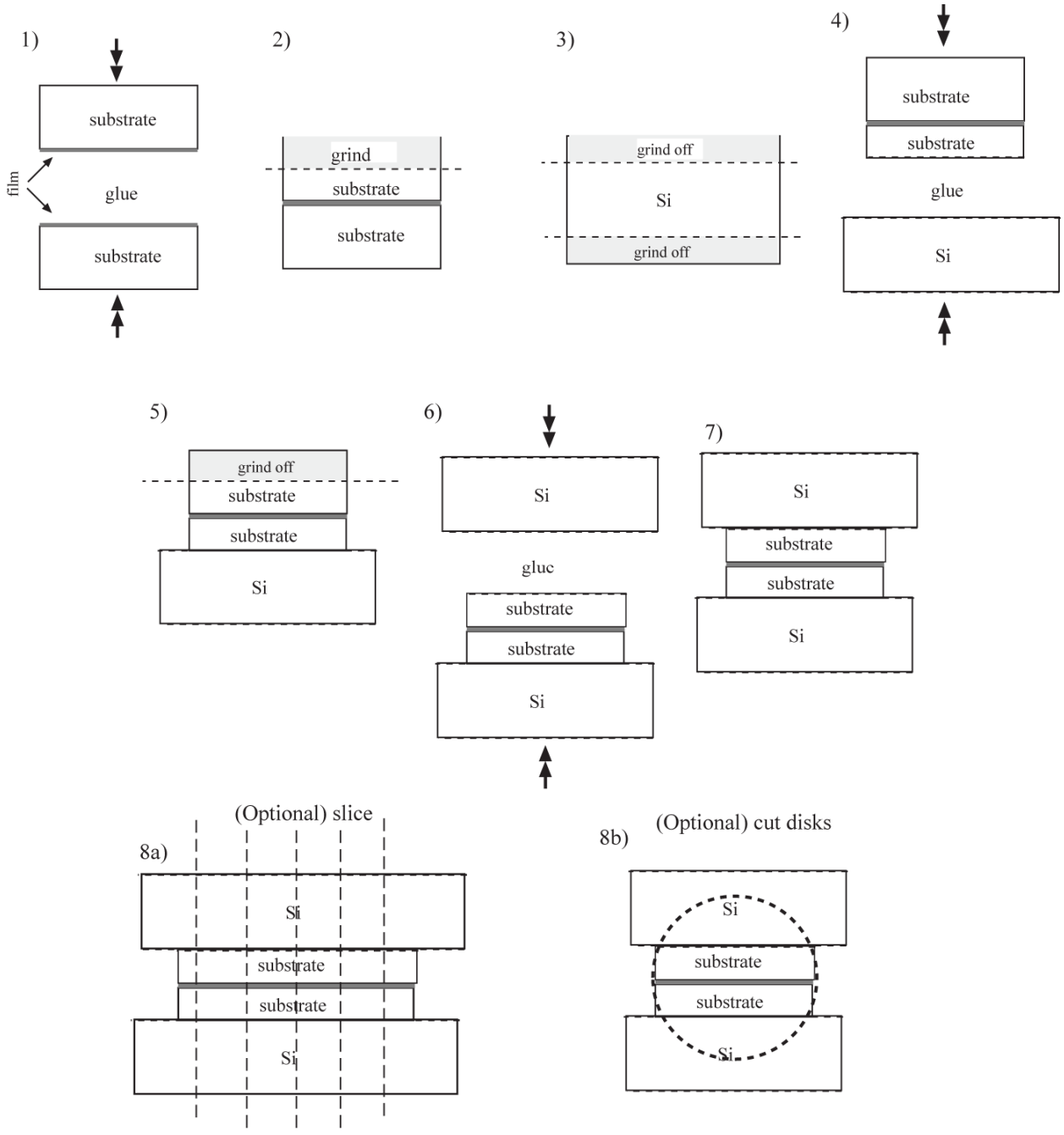
Cross Section
Gluing/Mounting/Grinding/Dimpling/Polishing:
1) Cleave two narrow strips (roughly 1.5 mm x 4 mm) off of the edges of the wafer. Rather than taking them both from the same edge, it is conventional to take one strip from each of the two orthogonal <110> edges. Also cut two pieces of scrap Si to about 2 mm x 5 mm.

2) Prepare some Epo-Tek 353ND epoxy (10:1 resin:hardener). Test the glue by placing a drop on a glass slide and placing on the hot plate. It should become dark brown and hard, without crumbling. (Too light and gummy, not enough hardener; Too crumbly, too much hardener.) Let it sit for a few minutes before using. It will remain useable for several hours.

3) With a toothpick, generously spread the epoxy on the film side of both sample strips. Then place the two strips face to face and apply mild pressure to make a "sandwich". Place them in the open vise, with some paper strips (to keep the sample from being glued to the vise.) Then gently tighten the vise to apply a constant pressure. Place the vice on the hot plate, with the handle up, and wait for at least 10 min for the glue to harden.

4) Remove the vise from the hot plate, being careful not to burn yourself. Place on a piece of metal to cool. Loosen the vise and pull out the sample.

5) Place the sample on a Gatan platen with clear wax. Carefully grind enough to get a flat, ground surface. Remove from the platen and clean with acetone. Also grind both strips of Si, so they each have one flat, ground surface. Remove and clean.



6) As in 3), glue the ground side of the sample to the ground side of one piece of Si. Then mount on a Gatan platen, with the sample side up. Lightly grind this side to get a flat, ground surface. Remove from the platen, and clean. Then glue the second side of the sandwich to the second piece of Si. Apply glue to all the sides that will be attached. Squeeze to make the glue layers narrow. You will probably have trouble if the faces aren't parallel. I put the whole structure in a vise and apply pressure. Then put the vise on a hot plate at about 150°C for several minutes. The excess glue turns hard and dark brown when ready.

6) Mount the glued construct with wax to a glass slide, and slice with a slow-speed diamond saw into roughly five 0.8-mm-thick sections. This gives you five potential cross sections. Your glue layers have to be strong here, or they will just fall apart.

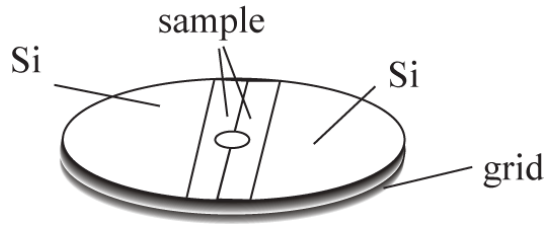
7) Pick one piece and mount on a glass block. Lightly grind, and then polish with 3-mm and then 1-mm diamond slurry, Metadi fluid, and water on a Texmet or nylon pad. Always keep the polishing wheel moving normal to the interfaces. (I spin the wheel — fast!) Reduce the pressure at the end to remove scratches.

8) Prepare some silver paste and obtain a clean Cu grid.. With an optical microscope, put glue on a few spots around the grid. Orient the cross-section with the polished side up, and place the grid with the hole over the region of interest (or vice-versa). Push down lightly. If I get unwanted glue on the sample, I wash with acetone and try again.

9) Flip the sample over, and heat the grid side for about 30 s on a glass slide.

10) Mount the sample on a VCR platen. Thin to about 100 mm (plus the grid thickness). If the sample extends beyond the edge of the grid, chip off the corners with a tweezers.

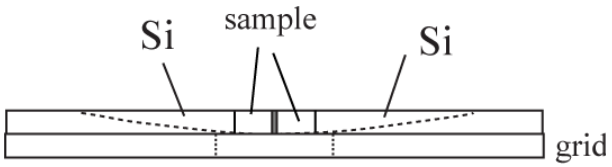
11) Now dimple using 3-mm and then 1-mm diamond, slowly reducing the pressure. You can use light transmitted through the Si to estimate the thickness. Ideally, you can stop before going all the way through. It may not be a total loss if it has a hole; this reduces your milling time, and the associated damage.



12) Heat the platen. Remove the sample and soak in acetone.

Ion Milling:

13) Ion mill from both sides at low angle (<15°) with L-N_2 cooling. I use 3.5-4 kV. Reduce the angle and current as you go. I use a two-sector setting at 10 rpm. Try not to mill more than about 2-3 hours, or the milling damage can get severe. (However, if you mill less than about 1 hour, the polishing scratches will still be present.)


Si
sample
Si
grid