# Flat Polishing for TEM sample preparation

#### Things you will need to buy or borrow



YINQE has lots of copper tubing, but you are expected to buy your own consumables: grids, epoxy, and sandpaper. You are also expected to buy your own small tools: tweezers, diamond scribe, and clippers.

1. Suppose you have grown or deposited a thin film on a wafer (typically, a silicon wafer.) Create a sandwich of wafer/glue/wafer, keeping the film of interest in the middle of the sandwich. The glue should be a thin epoxy, such as Epotek 110 or MBond 610.

You could use the **diamond saw** to slice 2.5mm wide strips of the sandwich, or you could cleave the wafer into narrow strips first, and then make a narrow sandwich.

Refer to the section below, "Making a sandwich the fast way"

Save some epoxy for step 5.

- 2. Harden the epoxy on a hotplate at around 150C for one hour. The small hotplate in the TEM prep room gets to 150C at its lowest setting.
- 3. Place the wafer sandwich strip in a 3mm copper tube, then balance the tube on end, on a glass cover slip.
- 4. Place the cover slip and tube on the 150C hotplate (lowest setting)
- 5. Fill the tube with epoxy, using a toothpick to drip it inside. Some of the epoxy will come out the bottom, hardening onto the cover slip.
- 6. Leave the tube on the hotplate overnight.



## Slice the bologna

- 1. Clamp the cured tube and cover slip in the saw, with the cover slip sticking out to the right.
- 2. Cut the cover slip off the tube, using ~ 100 rpm and a moderate pressure from the counter-weight.
- 3. If your wafer sandwich is at the other end of the tube, then unclamp the tube and turn it around. Cut a fresh slice from this end.
- 4. Turn the micrometer four full turns to make a 1mm thick slice. Cut again. Were you able to catch the slice with a paper towel? If not, you'll have to fish it out of the oil pan. What fun!
- 5. While you are here, cut a bunch more. Sing the Oscar Meyer song for good luck.



#### Mounting the sample

- 6. Crystal bond wax melts nicely at 150C, so hopefully you left the hotplate on. Place the T-shaped round sample holder on the hotplate, then put a small flake of wax on it. Let the wax melt, then push your sample into it. Use just a small flake of wax. You don't want wax all over the holder.
- 7. Place the sample holder into the polishing puck, and retract the center mount until the sample is below the surface of the puck.



sample toward polishing wheel



## Ready to polish

- 1. **SAFETY** REMOVE OR TIE BACK LOOSE CLOTHING, NECKLACES, TIES ETC. LONG HAIR SHOULD BE TIED BACK OR FIXED UNDER A HAT.
- 2. Place the aluminum platen on the polishing wheel, and affix a sheet of 600 grit sandpaper.
- 3. Turn on the water and set the rotation to about 100 rpm. Place the puck on the platen, holding the puck by the side. Do not push down on the puck.
- 4. On the puck, turn the knob clockwise, which lowers the sample toward the sandpaper.
- 5. You will feel the sample start to catch on the sandpaper.
- Turn the sample slowly while polishing, to make a more even polish. Gradually dial the puck thickness knob, polishing down until you get to the 150 – 200 micron mark.
- 7. Switch to 1200 grit sandpaper. You will find it convenient to use two aluminum platens so that you can switch grits easily.
- 8. Polish down to around the 500 micron mark.
- 9. Flip the sample over: Place the sample holder on the hotplate to melt the wax (lowest power setting). Pull the sample off and flip it over.
- 10. Put the sample back in the puck and back off the dial a bit.

- 11. Switch platens to the 600 grit paper again, and place the puck on the polishing wheel.
- 12. Adjust the knob until you feel the sample catch on the paper.
- 13. Polish until you get to around the 700 micron mark.
- 14. Switch platens to the 1200 grit paper.
- 15. Keep polishing until the copper ring is removed completely. Be sure to turn the sample as you polish. The dial indicator on the puck might get close to zero or even become negative, and so the copper ring is a better indicator of the stopping point.

If all goes well, your sample will be about 20 microns thick - ready for the ion mill.

## Mounting the sample on a TEM grid

#### Placing your sample on the grid

- 1. Place the polishing puck on the hotplate (lowest setting) and remove the thin sample. Soak the sample in acetone to remove the remaining wax.
- 2. Prepare some Epotek 110 or MBond epoxy
- 3. Hold the grid down with two pieces of semi-tacky tape or post-it notes, affixed just on two edges.
- 4. Using a broken toothpick, single-hair brush, or sharp needle, place tiny dabs of epoxy on the TEM grid, on opposite sides of the hole. Tiny dabs, not big gobs!
- 5. Place your sample in the middle of the grid, spanning the hole. Vaccum tweezers might be good for this operation. If the glue wicks over your sample then you've used too much. (Start over)
- 6. Place the grid on a hotplate to cure for about one hour (or whatever).
- 7. Using the nail clippers, remove any exess sample material that might be hanging beyond the 3mm diameter TEM grid.

## Making a sandwich the fast way

- 1. Suppose you have deposited an interesting film on a (100) silicon or GaAs wafer. Those wafers are very easy to break into neat rows. The flat of a wafer is along one of the crystal planes. Another crystal plane is perpendicular to the flat.
- Using a diamond scribe, make a small scratch ~2mm long, perpendicular to the flat (or parallel to the flat)



3. Grasp the wafer with tweezers on either side of the scratch, and crack the wafer by pushing the scratch UP.

The picture here shows #6 Dumont tweezers, which are great for this job; but you could also simply use your fingers.

Some people like to break the wafer over the edge of something. That works too, but not as well.



- 4. Cleaving with tweezers works great when the pieces are big, but if you want 2.5mm wide strips, then we have to change tactics. Flip the wafer over and use a diamond scribe to make a series of light scratches 2.5mm apart on the backside of the wafer.
  - If you have an automated scribing machine or a table scriber, then your lines will be neater and more uniform. But actually, they don't need to be perfect. Just try to keep the scratches more-or-less along the crystal plane.



 Now for the fun part. Put two cloths or paper towels under the wafer, so the surface is soft. Flip the wafer back to the front side. Place a glass rod or thermometer parallel to the backside scratches.



6. Using two hands, roll the glass rod over the wafer while pressing down firmly. The wafer should break neatly as the rod rolls over each scratch.

These strips should fit inside a 3mm copper tube. Check that now.



7. Mix the epoxy according to directions, and then use a toothpick to spread some drops over the (film side of) the wafer. Press two wafer strips together and spread the glue by wiggling the wafers a bit. The film and glue are the meat of the sandwich.

For a toasted sandwich, clamp the wafers with binder clips and leave it on the hotplate overnight to cure.

Directions from epoxy manufacturers specify a complex sequence of baking temperatures. You can ignore that.



Just leave the sandwich on a hotplate at 150C (the lowest setting) for several hours, or overnight.

Now the sandwich is ready to become a burrito, by mounting it inside a copper tube.