Grinding and Polishing of Glass Samples for Cross Section Determination of Water Uptake

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Tired of grinding and polishing pieces of glass for days on end? Then this guide is just for you. Designed and written by REU students with weeks of experience on the cross sectioning of glass samples, this guide will show you how to make your own **polished 150-200 µm** thick slices with efficiency and quality assurance. The simple steps below will allow you to make not one, not two, but **FOUR** cross sections, see Image 1, **in one day**[1], saving you time and effort. We can't make the process exciting, but the time you will save with these steps will permit you to focus on the more interesting aspects of your research. Ok, enough jibberjab…

Image 1: Finished cross section product

A) Mounting Sample in Epoxy

1. Cut 5x5x1 mm samples in thirds using wire diamond saw/ wax mount. Try to make the cuts parallel to the edges since the cuts will determine the final edges of the cross section. Take the **middle piece** of each sample to use for the cross section so that you do not get any edge effects. Save the other two pieces in case you screw up. (Since you can't keep track of which edge was an original edge of the 5x5x1 mm sample, you need to make sure you grind at least 0.5 mm from both edges if you end up using the other two pieces.)
2. Clean glass piece(s) with acetone in ultrasound to remove wax.

[1] This does not include the initial wire cutting step and final ultrasonic washes. Including those steps, expect a good day and a half for the entire procedure.

3. Place glass piece(s) in plastic sample holders such that the clip is holding the polished faces of the piece. The piece should be flush with the flat side of the sample holder so that we do not take a slanted cross section. The clip should also be holding the sample near one end, so that if the glass happens to crack where it's held by the clip during grinding, there will still be a large piece remaining.
4. Kimwipe down the inside and bottom of the epoxy molds (short ceramic tubes) and spread a small amount of vacuum grease on these surfaces.
5. Place each ceramic mold on top of a **clean** glass slide. (Dirty slides will make removal of sample difficult after the epoxy hardens.) Place samples & holders inside ceramic molds so that the sample is flush with the glass plate. You may want to place the holders slightly off center- this will help you keep track of what side of the sample you are looking at later. It is best to prepare multiple molds (4 is good) to save time and epoxy mix in the next steps.
6. **Mix epoxy** (Citrofix) with curing agent according to instructions (2:1 ratio of powder to liquid).
7. Pour epoxy into molds, enough to cover the glass but not too much as to cover the sample holder, which only increases the amount of epoxy you have to grind through to reach the glass, see Image 2.

Image 2: Making epoxy mount

8. Allow epoxy to cure for about 10 minutes.
9. Remove the epoxy-sample mount from mold and glass slide. This should be fairly simple if the molds and slides were adequately cleaned

B) Grinding first side to proper depth

1. Make (or reuse) **glass slide mount holders**: To make an ideal glass slide mount holder, take a glass slide and break it in half. For each piece, break off the two corners on the broken edge of the piece such that the slide will fit on top of the round metal sample holder used for polishing (see Step C1). Do NOT break the other two corners. The two pieces when their flat edges are placed together should be able to cover all the vacuum holes in the grinding machine and be held firmly by the vacuum (see Image 3 and Step B5). This will allow you to grind two samples simultaneously.

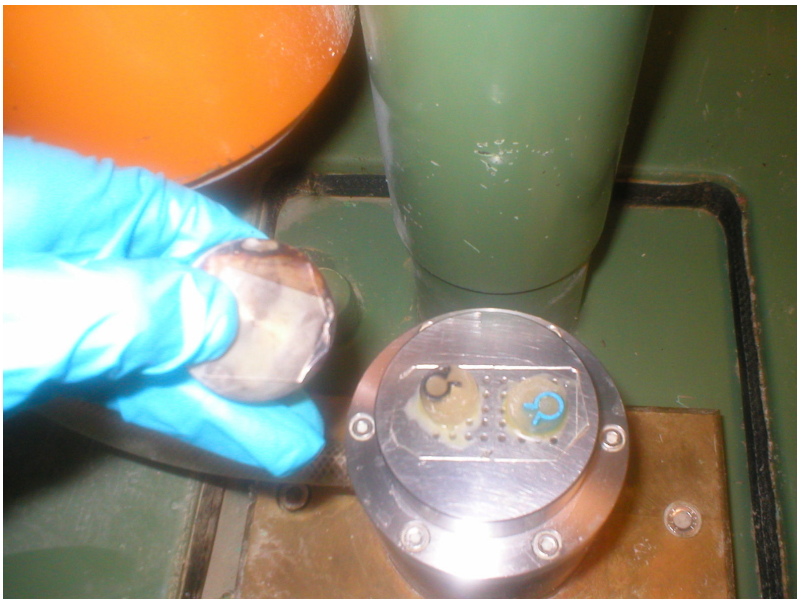

Image 3: Two glass slide mount holders fit on the grinding machine while one fits on the polishing metal cylinder

2. Affix the epoxy mount to slide with glue: Place the flat/flushed side of the epoxy mount down on the glue side so that the long edge of the sample is parallel to the long edge of the slide. This is very critical for obtaining a flat surface during grinding at a later stage. Press the mount down onto the slide to ensure the mount is flush with the slide. A good way to do this is to place a flat piece of metal on the top of the epoxy mount and a heavy piece of metal on top of that, see Image 4.
3. Prepare for grinding on the left GMN machine, which has a coarser grinding wheel than the right GMN machine. Loosen the lever behind the center column, then adjust the height of the grinding wheel above the sample mount. Raise the wheel by pumping the lever on the right side of the machine. Lower it by turning the handle on the right side. Be careful to avoid any sudden contact to the sample. The wheel must remain out of contact with the sample; check this by moving the wheel across the sample. When the wheel is in place, tighten the lever on the back of the main column.
4. Turn on the vacuum pump without any tubes attached. Check which of its connections is the sucking one and then attach the proper tube to it and close

the separation funnel used for collecting grinding fluid. Turn on the grinding fluid circulation pump.

Image 4: Heavy metal pieces press mount flat

5. Place two samples on the grinding machine and turn left the vacuum selector switch (just under the table surface under the right machine) to vacuum. The right switch should be off vacuum. Check that the samples are firmly stuck in place on the sample platform.
6. Turn on the grinding solution flow and the grinding wheel rotation. The grinding solution flow should be enough to produce a mist when the grinding wheel is spinning. Any more than this just increases the chances of flooding (and the need for unnecessary cleaning).
7. **Begin grinding**: Each pass should be made at moderate even speed, approximately several seconds to pass over each sample and several seconds to pass back across. One unit on the grinding dial equals 10 µm and one complete turn grinds off 1 mm. Grind off about **5 increments (50 µm)** for each pass. Continue grinding until you have reached the top edge of your glass sample(s).
8. The gear on the central column has only a limited range of motion, at some point it may reach the bottom of its track. At this point, turn off the grinding wheel and grinding solution. Then turn the gear repeated in the other direction until it stops and then line up the grinder again using the coarse adjustment (Step 3). Then continue grinding.
9. The liquid trap connected to the vacuum pump may fill up with time. Empty it before the liquid level reaches the exhaust tube by disconnecting the vacuum pump and turning the nozzle at its base.
10. When you have ground through the epoxy to the top edge of the sample(s), measure the thickness of each. Continue grinding until the sample(s) is(are) **1 to 1.1 mm thick**, grinding off about 3 increments per pass.

11. Switch to the right GMN grinding machine, which has a finer grinding wheel attached. To do this switch use the vacuum levers under the table (see 5).
12. Use the coarse adjustment to place the grinding wheel slightly above the sample. Lock the coarse adjustment.
13. Make very gentle passes with the grinding wheel off to find the surface of the sample. If you are gentle, the sample will be fine if the wheel touches it. Find the point where the grinder first is stopped by the sample and back up 10 increments.
14. Start the grinding wheel and grinding fluid as with the other machine and grind of increments of 1/2 to1 per pass. Never use increments more than 2 per pass (because this will result in non-flat surfaces which are not acceptable for the intended IR sample analyses). Grind off a total of about **10 increments (100 µm)**.

C) Polishing of the first side

1. Mount the sample on a **metal polishing cylinder** (on counter, round, several centimeters high) using glue. Place the glass slide/sample on top of glue. Apply some downwards pressure with some metal pieces as in step B2 to ensure slide is flat on the cylinder. Be sure to use a cylinder that fits in an available **donut** (found with cylinders) without much excess space.
2. Place **1200 grit paper** onto the right polishing wheel. Turn on polishing wheel and water and completely wet the surface of the wheel.
3. Place metal cylinder with the sample in the donut, which will hold it upright during polishing.
4. Locate the direction of the scratches on the sample from the GMN machine. You should be able to see them with your bare eyes. (The giant burr made from the grinding chips will be on one end of the scratches.)
5. With the wheel off, place donut with cylinder onto the wheel. Orient the cylinder such that the **direction of polishing is perpendicular to the scratches** (align the scratches pointing towards the center of the wheel.)
6. Polish sample by turning on wheel to **300 rpm**. Make slow large circular motions with the donut. Do not have the water drip while polishing unless the wheel becomes very dry. The drier the wheel the faster (and coarser) the polishing. Stop after a minute to check the sample. Continue polishing until you can no longer see scratches from the grinding machine with your bare eyes. Then polish for a minute using a lot of water (a continuous flow) and while rotating the sample so it is polished in all directions.
7. Wash donut and cylinder with water in ultrasound for a minute to remove grit.
8. Turn on the left polishing wheel, which has a polishing cloth. Wet the surface of the wheel. Drip some **alumina powder suspension** (0.3 µm grain) on the wheel; a line from the center to the edge of the wheel is plenty. Turn off water so powder is not washed away using the wall valve (machine is leaky!).

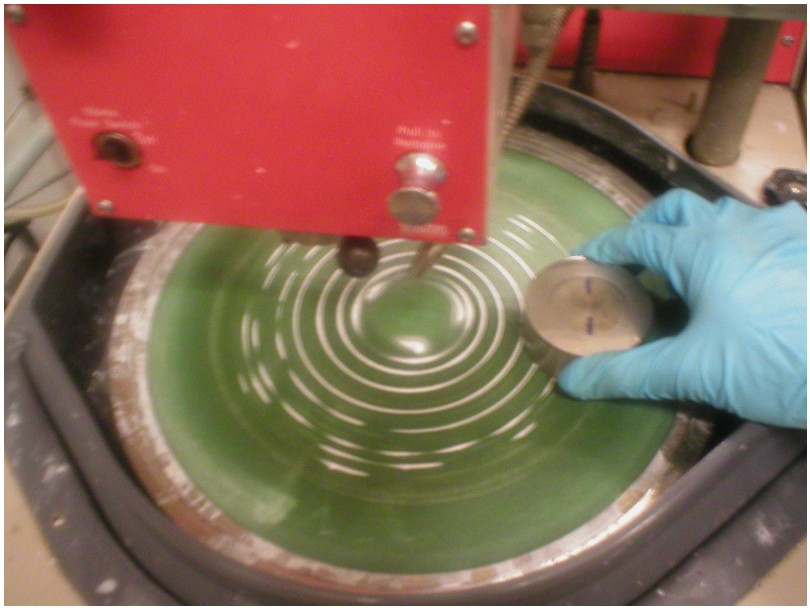

Image 5: Grinding with alumina powder

9. Polish sample at 300 rpm for two or three minutes using no water, again making circular motions with your hands. **Hold on tight!** This wheel grips the sample a lot harder and will jerk the donut from your hand if you are not careful. Rotate the sample to polish in all directions. Polish for an additional minute or two with the water at a slow continuous flow. This will wash away alumina grit embedded in the surface of the sample. (See Image 5)
10. Wipe sample with a kimwipe and examine the surface under a **microscope**. The surface will be essentially featureless when sample is completely polished. If scratches remain, repeat Steps 8-10. If a lot of really small dots remain, polish a final time using no alumina powder and a lot of water.
11. If alumina powder polishing does not seem to be polishing the sample well after several repetitions, change the polishing cloth. The green felt type works really well.
12. Note: You should polish two samples at the same time to save time. Before doing this make sure you can grip both donuts firmly with one hand and be able to hit the stop button with the other. You may want to try this at 150 rpm before knocking it up to 300 rpm, especially on the really jerky polishing cloth wheel.

D) Grinding the second side

1. Find another glass slide in the appropriate shape (see step B1) for each sample. Glue on the new slide and heat the glue on the old slide to remove sample/ epoxy mount. Quickly flip the sample/ epoxy mount over and place on the new side. Quickly place a piece of weighing paper on top and then apply **downwards pressure** using metal weights as in step B2. This is EXTREMELY important as the thin layer of epoxy will warp due to the heat and needs to be flattened. If this is not done properly, your sample will likely

break away from the epoxy mount during grinding and may be lost. During this step try to minimize the epoxy's exposure to heat to minimize the warping. (The weighing paper will prevent your sample from sticking to the metal weights.)

2. **Measure the thickness** of the sample. It should probably be **800 - 1050 µm** at this point.
3. Use the left **course grinder** (Part B) to grind sample down **to 500 µm**. Grind at most 2 increments per pass.
4. Use the right **fine grinder** (Part B) to grind sample down **to 300- 350 µm** using ½ increments (5 µm) per pass. Do NOT grind further than this or else you will fracture your sample.
5. As before, you can grind two samples at the same time. Make sure you measure the thicknesses of both samples as they may differ.

E) Polishing the second side

1. **Mount slide** onto metal cylinder as in Step C1. Remember to put metal weights on top during cooling to prevent epoxy from warping.
2. Polish sample using **1200 grit paper** as in Part C - 300 rpm and no water. Check the thickness of the sample after each minute of grinding. The grinding will take off roughly 20 to 30 µm per minute when no water is used, however this is extremely variable depending on how dry and new the paper is. If you want to polish off material at a slower rate use water while polishing. Continue polishing until your sample thickness measures **180 to 230 µm.** Due to the glue on the slide the measured value maybe a little off from the actual thickness of the sample.
3. Switch to the **alumina polish** wheel and polish sample as in Steps C8 to C10.
4. As before, you can polish two samples at the same time. Be sure to have a firm grip on the donuts and keep track of the thicknesses of both samples which may polish at different rates.
5. "Melt" the glue and remove sample from the slide.
6. Wash sample in ultrasound using the same procedure as cleaning samples cut by the diamond saw (acetone x 2, ethanol, water). Transfer the sample between beakers in an uncluttered area. It is really hard to find cross section samples when dropped.

F) Addendum on FTIR measurement:

- **First, make sure that your sample is plane-parallel in the direction perpendicular to its long edge and parallel to the water diffusion direction by using a digital indicator. If it is not, you need to go back and do some more machining. Starting any measurements with non-parallel samples is a waste of time because one cannot produce any useful data using such samples.**

- Place sample on metal plate with the small **slit** through the middle.
- **Purge** using the glass ring on the microscope to remove background water noise. A rapid purge rate (about **9 LPM = liter per minute**) produced the best results. Rather than retaking the background when the background water noise gets large, you may want to change the purge rate to counteract the noise (increase purge rate when the noise is positive and vice versa).
- **Focus** the beam on the **center** of the sample for the best resolution.
- The position numbers on the joystick are in **4 times** what they are in micrometer. Thus move 400 units to go 100 μm.
- Measurements / backgrounds should be taken in the **middle of the slit** to maximize the signal. Before taking the first background, scan in the x direction until the signal is maximized to find the middle of the slit. Since the slit is crooked, you will need to change the x-coordinate to stay in the middle of the slit as you traverse in the y direction. This is not that important, but if you are anal retentive, move about **7** units to the left for each 400 units upwards.
- Take spectra at every 10 μm near the edges of the sample and every 100 μm near the middle for a good profile.
- Due to the thinness of the cross section, your samples may produce thin film **interference patterns**, which will show up as sinusoidal wave patterns on the FTIR. The best way as of Aug 2004 to deal with this is to use the **Fourier Self-Deconvolution** function in the Manipulations menu. This function smoothes out the spectrum by removing higher frequency oscillations. Modify the parameters so that the sinusoid pattern is gone but not too much smoothing is done. Deconvolute all of your spectra using the same parameters, even if you have spectra without interference problems. The 'good' spectra will only be modified a negligible amount. Some good parameters to try: Adjust Parameter Tab: Line shape = **Lorentzian**; **Deconvolution Factor = 1; Noise Reduction Factor = 0.3.**