Making Petrographic Thin Sections

This is a description of how to make thin sections using the facilities in the Union College Geosciences Department. Thin section facilities in other places will probably have somewhat different equipment, supplies, and procedures, but these instructions may be helpful in any case. Making thin sections is both an art and a science. We have sufficient equipment so that the art aspects are relatively minor, but every rock is different and techniques can best be learned by perseverance and practice.

SUMMARY INSTRUCTIONS

- 1. Cut the slab.
- 2. Mark the slab and cut the chip (some call this a 'billet' or 'block'). Size should be about 25 x 40 x 10 mm.
- 3. Label one side of the chip, and (optional) impregnate the other side with C-D epoxy (Buehler EpoKwick FC).
- 4. Hand-grind the chip with 400 grit, then 1000 grit to make the section side flat.
- 5. Frost a glass slide with 1000 grit. If you don't do this the rock may peel off the glass.
- 6. Wash the chip and slide by scrubbing with a clean sponge and detergent.
- 7. Cement the chip to the slide using E-F epoxy (Buehler Epoxycure 2).
- 8. Scrape off excess epoxy from the glass slide, especially the back, and label the slide using a carbide or diamond scribe.
- 9. Trim off the chip using the trim saw.
- 10. Grind the section on the thin section machine to no thinner than \sim 50 μm .
- 11. (Optional) Surface impregnate the machine-ground section using C-D epoxy (Buehler EpoKwick FC).
- 12. Hand-grind the section using 600 grit.
- 13. Finish hand grinding using 1000 grit to give a uniform 30 µm final thickness.
- 14. Wash the section.
- 15. Put on the coverslip.
- 16. Remove excess Canada balsam with a razor blade (if there's a lot of it), and then acetone. Use acetone in a hood.
- 17. Label the section top using something easily visible.
- 18. Be sure the room and equipment are clean when you leave.

CUTTING THE ROCK CHIP

https://muse.union.edu/hollochk/kurt-hollocher/equipment/diamond-rock-saws/

Slabbing big rocks \rightarrow large diamond saw

- 1. Check that the large diamond saw has enough water in its tank. The pump top should always be covered.
- 2. Decide where you want to cut the rock. This is not as trivial a matter as you might think. Different cuts through an anisotropic rock will show the fabric differently.
- 3. Mount the rock in the saw vise. Securely clamp it so it sticks out on the saw side of the vise by at least 3 cm. Use wood blocks to help clamp oddly shaped rocks. You should be able to yank on the rock without making it move. If the rock shifts during cutting it can damage the

- blade (expensive) or burn out the drive belt (makes a big stink and sets off the fire alarm). Also remember that the blade cuts a 3 mm thickness of rock.
- 4. Using the ratchet wrench, position the vise so that the first (outside) cut is at least 2 cm from the vise. Roll the vise so that the rock is almost touching the blade. Engage the vise feed clutch.
- 5. Close the lid and turn on the saw. Make sure that coolant comes out on BOTH sides of the blade within a few seconds. If this doesn't happen, either the pump is broken, the reservoir is out of water, or something is clogged. Shut off the saw and fix the problem. Running the saw without coolant will ruin the very expensive blade. Don't wander off while the large saw is running. If the blade does jam, turn the saw off as quickly as possible.
- 6. In general, you can run the electric vise drive mechanism at its top speed (highest voltage, ~12 volts). For bigger or tougher rocks, especially chert and similar, slow it down (lower voltage, say ~9 or 6 volts). Listen to the blade. If it starts to slow down, reduce the vise drive speed (lower voltage).
- 7. After the cut is complete, turn off the saw and lift the lid. **Don't remove the rock from the vise yet!** Disengage the vise feed clutch and roll the vise back along the rails. Using the ratchet wrench, move the vise so that a second (inside) cut through the rock will produce a slab 5 to 15 mm thick.
- 8. Move the vise up to the blade again, engage the clutch, and make the second cut. When finished, remove the rock and your slab. To prevent rust, always leave the saw lid open when it's not in use.

Slabbing small rocks \rightarrow small diamond saw

- 1. For fist-size rocks and smaller, the small diamond saw can be used. The main points are to make only straight cuts, use moderate, even pressure on the blade, and make sure you don't push the blade to one side or the other.
- 2. Make sure there is enough water in the saw coolant tank. Water should at least cover the pump top.
- 3. Decide where to cut the rock. Turn on the saw using the switch on the motor.
- 4. Make sure water comes out on both sides of the blade. If this doesn't happen, either the pump is broken, the reservoir is out of water, or something is clogged. Shut off the saw and fix the problem.
- 5. Hold the rock firmly, keeping your fingers out of the plane of the blade, using either just your hands or hands and the metal guide tool. The spinning blade won't cut your skin if you gently touch it, but it will if you press hard enough. It will cut fingernails, though. The spray guard should only be high enough to let the rock underneath it.
- 6. If you make two cuts for the chip top and bottom, make them as parallel as possible.

All rocks → trimming the slab to make a chip

- 1. On the cut slab, use the thin section chip template (rectangular aluminum plate with a rectangular hole in it) and pencil to mark the slab area you want for the thin section. Several chips can be marked at once on a big slab. Remember that the trim saw blade makes cuts about 1.5 mm thick.
- 2. Make sure the small trim saw has enough coolant in its tank. Turn on the saw and make sure coolant sprays from the blade on both sides. Put your slab flat on the saw table (break or trim off any rock protrusions if necessary). Align one side of your marked area with the blade and

- slowly push the slab into the blade to make your cut. Hold the slab firmly and make the cut as straight as possible. Let the saw cut at its own pace and don't force it. When finished, make the other cuts to complete the chip.
- 3. Rinse the chip with water and let it dry. Label the back with the sample number using an indelible marker, paint, or a piece of notecard paper with a pencil label (Appendix C).

PREPARING THE CHIP AND GLASS SLIDE

- 1. If the sample is porous, friable, or has big cracks, you may want to impregnate the sample with epoxy first (see Appendix B).
- 2. Clean off a piece of plate glass and find a flat area. On the flat part, make a slurry with some 400 grit SiC and a little water. Using a circular or figure-8 motion and with moderate pressure, slide the chip around on the grit slurry to grind away the saw marks and plucked and damaged layer of the chip. Be sure to use as much glass plate area as possible, including edges, to slow grinding hollows in the glass.
- 3. Grinding can take 1 to 10 minutes. You will have to practice a bit to get the water/grit ratio correct. Check the flatness and quality of the chip surface frequently by cleaning and drying the chip, and holding it up at a small angle (few degrees) up to a light, so that the surface looks shiny. Continue grinding until the chip is shiny and flat across its whole surface.
- 4. Wash the chip and plate glass. Prepare another slurry on the glass using 1000 grit SiC, and grind the chip for about 1 minute, again using a flat part of the glass, and using as much of that flat part as possible. The result should be a flat and smooth reflective surface, visible when held at a low angle to a light.
- 5. You also need a glass slide to glue the chip onto. On a flat part of a piece of plate glass, grind the glass slide using 1000 grit SiC. You only need to grind enough to frost it (make it hazy) across the whole surface.

CEMENTING THE CHIP

- 1. Porous samples may fist have to go into the ultrasonic cleaner to remove grinding debris. Then, scrub the ground sides of the chips and slides with a clean soapy sponge, rinse thoroughly, and put them ground side down on some paper towels to drain. Use more paper towels to soak up water drops, then transfer them to dry paper towels ground-side down (usually keeps off more dust than ground-side up). If the samples are porous, put them on a hot plate or in a drying oven at whatever temperature you think best to dry them out, but <100°C.
- 2. Cover a smooth metal plate or tray with new aluminum foil and put the chips on it, smooth side up. Don't touch the ground surfaces! See Appendix C if you're planning on putting on paper labels at this stage.
- 3. Mix a small batch of E-F epoxy (2 ml or so, Buehler Epoxycure 2, see Appendix A).
- 4. Use the stick that you mixed the epoxy with to put an epoxy blob onto the ground rock surface. Gently put a glass slide onto the epoxy, making sure that the ground sides are together.
- 5. Use a rounded pencil eraser to work out the bubbles by pressing gently in the middle of the slide and moving the slide in small circles. Press the eraser progressively from the middle to each corner and edge to slowly work the bubbles out from between the slide and the chip. If

- the rock is porous (e.g., vesicular basalt, pumice), bubbles will get trapped in the holes and getting them all out will be impossible. Don't worry about it.
- 6. Move the slide so that it overhangs the chip roughly equally on three sides, with a larger overhang at one end for the sample number.
- 7. Repeat steps 4–6 for the rest of the chips. When finished, move the tray of samples into a drying oven to cure at 40° C.
- 8. When the epoxy has cured, take the warm chips one at a time from the oven, peel off the aluminum foil, and immediately use a sharp razor blade to trim excess epoxy from the edges and bottom of the glass slide. This is important because otherwise the chip will not lie flat in the thin section machine chucks. You can trim off warm or hot epoxy much more easily than cold epoxy.
- 9. Use a diamond or carbide scribe to write the sample number on the back of the slide, under the wide area of glass at one end. Wash off all of the epoxy trimmings.

TRIMMING THE CHIP

- 1. Turn on the coolant and vacuum pumps and set the valves so that water and vacuum are directed to the cutoff saw. Make sure there is coolant flow to both sides of the blade. Open the vacuum release valve and put the glass side of the section onto the vacuum chuck. Ordinarily the micrometer is preset to cut the chip about 0.5 mm from the slide. If not, you can adjust the chuck position.
- 2. Turn on the saw. Slowly and with moderate and even pressure, move the chuck toward the blade to cut off the chip. Ease off on the pressure near the end of the cut. When the chip falls off, bring back the chuck, open the vacuum release valve, and remove the section.
- 3. Repeat steps 1 and 2 for the rest of the chips. Turn off the saw before trying to get your trimmed chips out of the saw tank. Rinse off all of your thin sections and leftover chips.

MACHINE GRINDING THE THIN SECTION

- 1. You will use the cup wheel thin section machine to grind the sections to about 50 μm thickness at their thinnest place. The grinder is too rough on samples to grind the section to their final 30 μm thickness. Also, the grinding wheel never grinds the section perfectly flat, so there will always be thicker and thinner places. Our equipment is nowhere near good enough to produce a finished section. The problems include faster grinding at the first corner that touches the cup wheel and poorer grinding everywhere else, ground chip surfaces that are not perfectly flat (caused by dished glass plates used during chip grinding), and variable glue thickness between the glass slide and rock surface.
- 2. The thin section machine micrometer has 100 divisions around the ring, and each division increment represents 2 μm of chuck movement. The ring is adjustable, so the numbers usually don't mean anything in terms of specific thin section thickness. You will have to periodically check section thickness with a polarized light microscope as you grind, and use that to decide when to stop grinding.
- 3. Set the vacuum and coolant valves so that they go to the thin section machine. Turn on the vacuum, coolant, and the machine. Put a thin section in the vacuum chuck (vacuum release valve open). Using the micrometer, back the chuck away from the cup wheel until the thin section barely misses the wheel. Move the chuck back and forth, advancing the chuck with

- the micrometer by 5 increments for each 5 passes or so, until the thin section is about 100 μ m, (50 ring increments) of being done (done meaning 50 μ m thick at the thinnest part). After each time you check the thickness, you may reverse the section orientation on the chuck. Sometimes that helps make the section grind to a more even thickness, though sometimes it makes things worse. I don't know why.
- 4. Continue advancing the chuck more slowly and with more passes, such as 1 increment per 10 passes, but the specifics aren't really important and each rock may be different. Cherts you have to advance more slowly, but for shales and pumice you can go faster. For granites and such 1 and 10 are OK. Check section thickness frequently, especially checking each corner. Stop when the thinnest part is about 50 μm thick (quartz and feldspars have interference colors that are near the top of the first order).
- 5. Remove the section from the machine and rinse it off. Repeat steps 2–4 for all remaining sections. Once you get an idea of the approximate micrometer end point for a few slides, you can save time by not having to check the thickness of the others until you get within 50 or 100 increments ($100-200 \mu m$) of the final chuck position.
- 6. When all sections have been ground on the thin section machine, turn off the water and vacuum pumps, and the machine. Empty water out of the flask next to the vacuum pump.

HAND GRINDING

- 1. On a flat piece of plate glass, make a water slurry with some 600 grit SiC. Check with a microscope the section thickness in different parts. The middle may be the thickest, but it could be a corner or an edge. Put the section rock side down into the slurry and, while pressing on it with one or two fingers, move the section in circles or figure-8's to grind away the thickest parts. Use all of the glass plate surface, especially the edges and corners to slow dishing of the glass surface. The places where you press the hardest will grind away the fastest. You can actually hang part of the section off the glass plate edge, so that part will not be ground at all.
- 2. Rinse the section with water and check its thickness at frequent intervals. Always try to grind the thick parts to make the whole section even. Continue until the section has reached a constant thickness and quartz and feldspar has 1st order yellow or yellow-orange interference colors. Repeat steps 1–2 for all other sections.
- 3. Wash the plate glass and make a new slurry with 1000 grit SiC. Grind the section as before, and check the section thickness often. It's finished when the whole section has quartz and feldspar have interference colors of 1st order white to pale-yellow. Repeat this step for all other sections. If you grind too much away, don't fret too much. Thin sections are objects of scientific research, not usually subject to art criticism especially by people who don't make thin sections.
- **4.** Optional: Warm up a hot plate to about 60-70° C. Put a thin section on a smooth and flat part of the hot plate, and use a SHARP razor blade (blade horizontal) to peel away all of the epoxy from the edges of the rock section. Peel away enough so the rock part is slightly smaller than a coverslip so the rock edge will later be embedded in Canada balsam (n ~1.54) when the coverslip is put on, rather than epoxy (n ~1.56–1.57). Use an actual coverslip for comparison. This allows the refractive index of edge minerals to be compared with the Canada Balsam (Becke line method), allowing rapid discernment of minerals that might otherwise be hard to distinguish, such as untwinned plagioclase and orthoclase. A wadded-up

paper towel can protect your fingers from the hot section. This is a pretty arcane procedure that almost no one does anymore. It's also somewhat dangerous, what with a hot surface and razor blades and all, so be careful if you actually want to do this step. Have band-aids ready.

MOUNTING THE COVERSLIP

- 0. If you are definitely going to use the section for microbeam imaging or analysis, you'll be polishing the sample and won't need a coverslip. Skip this section. If you know you'll never, ever want to analyze the mineral surfaces or polish the sample, you can put the coverslip on with epoxy, just like you glued the glass slide on the rock chip. That's easy, but the coverslip will never come off. If you want to do optical microscope examination but *might* want to do microbeam imaging or analyses in the future, mount the coverslip with Canada balsam as described here. Balsam is a sticky pain to use, but it's easy to remove the coverslip.
- 1. Wash and scrub the thin sections thoroughly using detergent and a clean piece of sponge. Rinse them well and put them rock side down on paper towels, soak up water drips, and transfer them to new paper towels to finish drying.
- 2. Set a hot plate to about 100° C. Put a slide on the hot plate rock side up on a small piece of new aluminum foil, and let it heat up for a minute. While holding the slide in place with a pencil eraser, smear some Canada balsam onto the section, leaving it relatively thick in the middle. The idea is to wait for solvent to evaporate until strings of balsam pulled from the hot blob turn brittle after they cool. That means the balsam has lost enough solvent to put the coverslip on. Only the first section needs to be done by looking at gooey strings, the rest can be done by timing based on the first.
- 3. Carefully put a coverslip on the section and work out the bubbles by gently pressing the coverslip with a rounded pencil eraser, while moving the coverslip in small circles. Work the bubbles from the center outward, while also working out as much balsam as possible. Don't press too hard or you'll get vapor bubbles that you can't get rid of. The aluminum foil should catch all of the balsam that slobbers over the edge.
- 4. When done with the bubbles, center the coverslip over the rock, and take the section off the hot plate to cool. Repeat steps 3–4 for the other sections.
- 5. When all of the coverslips have been mounted, put them one at a time on a smooth, flat surface and use a razor blade to scrape away most of the excess Canada balsam. Be very careful not to get the razor under the coverslip or it will peel up. Don't cut yourself, either. If the coverslip peels up just a little, put the section back on the hot plate and use a pencil eraser to press the gap closed as the slide heats up.
- 6. Go to a fume hood away from all heat sources or sparks, and use acetone and Kimwipes to wash off the rest of the Canada balsam. If you use too much acetone you will dissolve too much balsam and undermine the coverslip.
- 7. Your section is now complete! Optionally label it on the top wide end with India ink, pencil, or something else, to make it easier to read the section label (Appendix C). Also optionally, you can cover the ink with a thin layer of epoxy, varnish, or colorless fingernail polish to protect it.

APPENDIX A: MIXING EPOXY

We currently use two kinds of epoxy. **C-D epoxy (Buehler EpoKwick FC)** is low viscosity and relatively fast-curing, good for sealing cracks, impregnating porous samples, sealing surface damage on thin sections after finishing with the thin section grinding machine, and for grain mounts that won't be exposed to an electron beam. **E-F epoxy (Buehler Epoxycure 2)** is higher viscosity but stronger and slower-curing, that is relatively resistant to electron beam damage. It's used for gluing chips onto glass slides, and for grain mounts that will be exposed to an electron beam.

- 1. For each kind of epoxy, the mixing ratio is 4 parts resin (C or E) and 1 part hardener (D or F) by volume. If mixing by weight (easier for large volumes, look at the bottle labels). You can only use C with D and E with F. You can't exchange one resin or hardener for another. There are containers with oral syringes next to the epoxy bottles. Each syringe should be labeled C, D, E, or F, and may only be used for the labeled hardener or resin. Don't use epoxy syringes for any bottle other than the one for which they are labeled.
- 2. Take one of the small aluminum evaporating dishes and blow out any dust.
- 3. Remove the cap from an epoxy resin bottle, and the cap from the end of the proper syringe. Fully depress the plunger on the syringe. Stick the end of the syringe into the hole in the bottle, and invert the bottle until all bubbles have risen out of the cap area. Extract the amount of epoxy resin you want, and squirt it into the middle of the aluminum dish. Pull back the syringe plunger all the way to the end, recap the bottle and syringe, and put them away.
- 4. Repeat Step 5 for the proper hardener, remembering that the volume ratios are 4:1, resin:hardener.
- 5. Use a wooden stick to thoroughly mix the epoxy parts together. The bottle instructions recommend two minutes of mixing, with repeated tilting of the aluminum dish to get different, less well mixed parts to flow over each other. Try not to trap bubbles as you stir. The epoxy is now ready to use.

The two epoxy components make a somewhat hazy mix at normal room temperature, suggesting that they are not completely miscible. The mix becomes clear at 40°C, so it's best to cure both types of Buehler epoxy in a drying oven at 40°C.

APPENDIX B: EPOXY IMPREGNATION

Porous or friable rocks can be surface or vacuum impregnated with epoxy so they hold together when cut or ground, and so grinding grit doesn't get into all of the little holes.

Surface impregnating

- 1. Rinse off the chip and dry it out in an oven or on a hot plate at 40° to 100° C for as long as necessary or appropriate for your samples. When dry, take them out and let them cool.
- 2. Mix an appropriate amount of C-D epoxy (Buehler EpoKwick FC).
- 3. Spread the epoxy on the thin section side of the block (top) and let it soak in for a few minutes. If it completely soaks in, keep adding more until it stops or until it starts flowing out the bottom of the chip.

- 4. Wipe off excess epoxy from the chip top with a paper towel.
- 5. Cure the epoxy in an oven or hot plate at about t 40°C.

Vacuum impregnating

- 1. This procedure is usually reserved for very soft or friable samples. It's a messy pain, so don't be too enthusiastic about doing every sample this way. Be sure the samples are dry.
- 2. Get out the plastic vacuum bell jar and make sure the gasket, gasket slot, and bottom rim of the top are clean and free of dust and epoxy. Connect the bell jar to the vacuum line.
- 3. Use the switch on the thin section machine to turn on the vacuum pump, and turn the valves so that the vacuum pump is drawing air from the bell jar. Turn the stopcock on the bell jar so that the vacuum pump only evacuates the vacuum line (vacuum pumps don't like to pump large volumes of air).
- 4. Mix enough C-D epoxy (Buehler EpoKwick FC, see Appendix A) for one or a few samples. Put them in cups or dishes and make sure they are completely covered with epoxy. Put the samples into the vacuum jar and put on the lid.
- 5. Open the bell jar stopcock so that the vacuum pump draws air out of the bell jar. Bubbles in the epoxy will immediately swell and burst, and bubbles of air will come streaming out of pores in the rock. After a minute or two, the air pressure will become low enough so the epoxy boils. Let it boil for a few seconds, but not enough to overflow, then turn the stopcock and let air back into the bell jar. Air pressure forces liquid epoxy into the pores of the evacuated sample, hopefully cementing everything together. Let the samples sit at atmospheric pressure for a minute or so, and repeat the evacuation–pressurization cycle two more times. Each cycle takes 2 or 3 minutes.
- 6. Remove the samples from their epoxy baths and remove excess epoxy especially from the side that will be ground down flat (the top side). Cure the epoxy on a hot plate or drying oven at 40° C. When cured, you may want to cut a flatter surface to grind using the small diamond saw.
- 7. WARNING! Large masses of epoxy will heat up from their own exothermic heat of reaction. Although the C-D epoxy is supposed to cure in 2 hours, that's at room temperature. Self-heating can cause it to cure in minutes. Don't mix more epoxy than for the number of samples you can handle at one time (start with 1). If the epoxy starts to cure before you are done, just leave the samples to finish curing, then use the small saw to cut off the excess to get you back to the chip stage again.

APPENDIX C: LABELING

On the thin section

When finished, the back of the thin section should already have its identifier etched on the glass slide with a carbide or diamond scribe. The front side should also be labeled to make finding the right thin section easier. Best is India ink, protected with a thin film of epoxy or colorless fingernail polish. Glued-on paper labels (mostly in the olden days) or pencil (cover with epoxy or colorless fingernail polish) on the frosted glass are OK. Worst is a felt- or fiber-tip marking pen. These fade over time, can become smudged and blurry, and usually aren't very high contrast.

On the chip

Felt-tip marker labels are almost universally used to label chips, but they can be a disaster especially on dark or porous samples. First, epoxy dissolves most marker ink, blurring out the label even on non-porous, light-colored samples. Second, epoxy makes the rock even darker, so dark marker ink can become essentially invisible. White paint splotches work OK as a background for India ink or pencil labels, but avoid ball point pen because some of those inks are also soluble in epoxy. White paint pens work fine though the tips wear out fast and they tend to clog up, so have some new ones on hand.

What I usually do is write the sample numbers in pencil on white notecard paper, then I cut the numbers out as little paper rectangles. When I epoxy glass slides onto the chips, I first put a blob of epoxy on the back of the chip and plop it down on the upside-down label piece for that chip. Then I put the glass slide on. For usual non-porous rocks, this works fine. The paper is encapsulated in epoxy, the pencil label stays legible indefinitely, and the thick white paper is a good background even against black samples. Do the gluing on a new piece of aluminum foil, so the epoxy doesn't leak and smear around everywhere.