Grain Picking and Trimming Procedures: Plastic Capsules

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I. BEFORE BEGINNING

Before starting the picking process, make sure that the temperature and humidity settings in the room are at the appropriate levels. The temperature should be roughly 70°F and the humidity near 50%. The humidity is the more important factor for picking. If it is too low (10 percentage points off or more) then there will be too much static to pick safely; conversely, a very high humidity will prevent grains from statically attracting to the needle. It will be possible to pick at non-ideal conditions, but there is a much larger chance of failure. Both humidity and temperature are important factors in the microtoming procedure as the newer SPI diamond knives have much shallower water reservoirs than older knives; reservoir evaporation becomes an issue if temperature and humidity are off of ideal.

Prior to using plastic capsules, we used bullet-shaped gelatin capsules. These have smoother interiors, however it was much more difficult to position the picked grain at the capsule minimum.

II. SUPPLIES FOR PICKING

- Aluminium block mount.
- Large plastic capsules.
- 2M NaOH solution.
- Syringe: 1cc, 31 gauge, filled with resin (no air bubbles).
- Tungsten micromanipulator needle.

III. CAPSULE PREPARATION

1. Identify a good capsule; bottom of the capsule should be smooth and free of pock marks. Make sure to focus on the "square" as this is the bottom of the *inside* of the capsule. If you focus on a circular region, this is the outside of the capsule. Note: some black bits that look like holes are actually dirt - if you are unsure, blow out the capsule with nitrogen and then examine again.
2. Use a marker to draw a line on the capsule just above the top of the aluminium block.
3. Use the tip of a razor blade to cut off the top of the capsule; slowly cut in a circle around the line.
4. Once the top of the capsule is cut away, remove any plastic threads or dangling bits from the rim of the capsule with tweezers.
5. Label the side of the capsule with a marker.
6. Using the microscope as an aid, place a red dot on the outside of the capsule directly above the center of the square. This will help later in identifying where the center of the capsule bottom is when picking grains. Note: Sometimes the center of the inside of the capsule is not aligned with the center of the outside.
7. Place the capsule in the aluminium mount. For orientation, place the label facing in the same direction as the mount number - also use this orientation when taking pictures under the microscope.
8. Blow out the capsule with nitrogen.
9. Check the capsule under the microscope to make sure there is no dirt. Take a picture of the capsule bottom.
10. Add one drop of resin to the bottom of the capsule. A good way to do this is to place a Kim wipe underneath the block mount, place the first drop from the syringe (squeezing gently) on the wipe, then drop the second into the capsule.

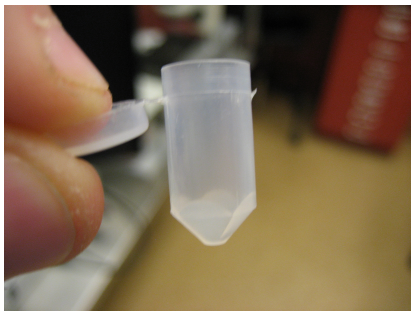

FIG. 1. A plastic capsule.

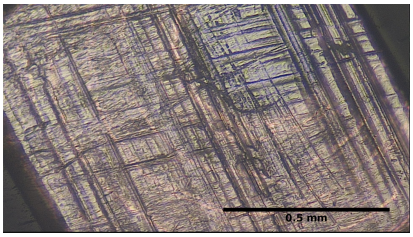

FIG. 2. Example of a poor-quality capsule (interior bottom surface). Poor surface are characterized by large gauges and irregularities. This capsule is too rough for picking.

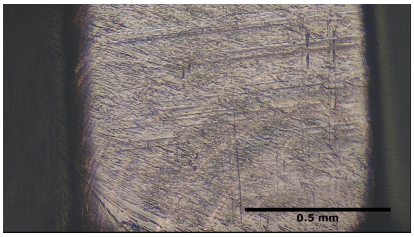

FIG. 3. Example of a higher-quality capsule.

IV. NEEDLE PREPARATION

1. Will need approximately 2cm of needle exposed. Shape this nearly vertically with an angle of 30 degrees or so on the tip. You want the needle to be tall enough to reach the bottom of the capsule. The tip should be angled so that only the very end touches the bottom of the capsule and sample mount. Need the tip top be angled so that you are able to focus on the end of the needle under the microscope, but so that the elbow in the needle doesn't bump the higher sides of the pyramid when inside the capsule.

2. Remember to wear gloves! NaOH is caustic [and sticky when it starts to dry].

3. Connect the needle to the circuit through the NaOH. Dip the needle repeatedly in the solution. You should see tiny bubbles foaming from the needle when dipped. The voltage dial should be set anywhere between 1-5%. The end of the needle should be a few microns thick.

4. If the entire needle is somewhat dirty, may need to dip the entire bit in NaOH so that dirt doesn't fall off into the resin while picking.

V. PICKING FIBRES

1. When it is time to begin picking the fibres, turn off the big AC unit. Then turn off all of the clean-benches.

2. Before putting the fibre slide or the mount on the sample stage, find the tip of the needle, center it on the screen and bring it into focus with 5× objective. [Note: Currently the 5× and 20× objectives are not completely aligned and parfocal. This is not a huge issue except that you may want to have the needle tip slightly above center on the screen in 5× so that it is centered on 20×.]

3. Bring the needle tip above the plane of focus (roughly 3 seconds on [Fine], middle speed) and set this as [Pos. 1]

4. Put the mount and fibres on the stage (I usually put the mount in back and the fibres in front because you will have to switch out the fibres for the sample later) and bring the carbon fibres into focus.

5. Search around the slide until you find a good fibre (maybe 75μm) and bring it to the center of the screen.

6. Drop the carbon fibre below focus by roughly $\frac{3}{4}$ of a minor revolution.

7. Bring the needle tip down into focus.

8. Raise the carbon fibre back up into focus using the minor-z knob until the fibre touches the needle. Move the needle and stage around slightly until the fibre sticks to the needle. The first fibre is always the most stubborn.

9. Once the fibre is stuck to the needle, move the sample stage below focus. Draw the needle above focus for about 10 seconds on [Fine]. This will give you roughly 3-4 seconds of vertical space above the surface of the resin when you are focused on the bottom of the capsule.

10. Move the sample stage and bring the bottom of the capsule into focus. Lower the needle until the tip touches the surface of the resin, then remove the needle. You can easily tell when you hit the surface. Bring the capsule below focus until you find the carbon fibre; ensure that the fibre is sinking. Check that the fibre reaches the bottom of the capsule then lower the sample stage fully.

11. Hit [Pos. 1] to reset the needle and repeat.

12. Once all three fibres have been deposited in the resin, you may use the needle to rearrange them on the bottom of the capsule. You may have to do this again once the grain is picked. Go slowly and be careful while dropping the needle through the resin until it and the bottom are in focus. The needle will easily scratch and gouge the bottom of the capsule, creating places where it is easy to lose the grain.

VI. PICKING GRAIN

1. Procedure is similar to picking carbon fibres.

2. Navigate on mount using 5× objective and then pick grain using 20×. I always keep the magnification module set at 1× since the 1.25×, 1.5×, and 2× can be blurry. You begin to reach the limit of optical resolution.

3. Make note of the grain size using the transparency scale. Scale for 400× uses 10× eyepiece, 2× magnification module, 20× objective. This will help you differentiate the grain once in the capsule from carbon bits or dirt. Side note: the 3CCD camera has an intrinsic 3× magnification because of it's $\frac{1}{3}$" chip size so true magnification as viewed on the screen in actually 1200× (or possibly more).

4. Make sure no resin is stuck to the needle tip. Lightly touch the tip to a clear area of gold foil, resin (if any) will wick off to foil. You don't want to have this happen as you try to pick your grain otherwise it will never stick to the needle. This also helps reduce excess static on the needle.

5. Always try to pick grain with very tip of needle or position to that grain is on the underside of the needle. If the grain is on top it may jump further up when dipped in the resin.

6. Once grain sticks to needle, keep needle and grain in focus. Lower sample mount.

7. Move the stage so that the capsule is underneath the needle and grain. Always move the sample stage, NOT the needle. It is best to keep the grain in view.

8. Raise the sample stage and center needle over the red dot. Continue raising stage until the needle dips into the resin, then lower slightly exposing the needle. If the grain remains on the needle, repeat. Once the grain is no longer on the needle, raise the needle and focus on the resin surface to find the grain.

9. Grains sink slowly in the resin (may take 15-20 minutes). If the grain remains on the resin surface use the needle to break the surface tension in a nearby area. Don't put the needle too close to the grain - you don't want the grain to stick back to the needle.

10. The grain may also need to be coaxed down into the right area. Do this by submerging the needle tip near the grain on the side of the direction you wish to move the grain (again without touching). Moving the needle away will drag the grain in that direction. When you are far enough away, raise the needle and repeat as necessary.

11. When the grain sinks to the bottom of the capsule it is best to position the carbon fibres around the grain in lieu of repositioning the grain with the needle itself. It is too easy for the grain to stick to the needle again, though it will be more difficult to dislodge a second time.

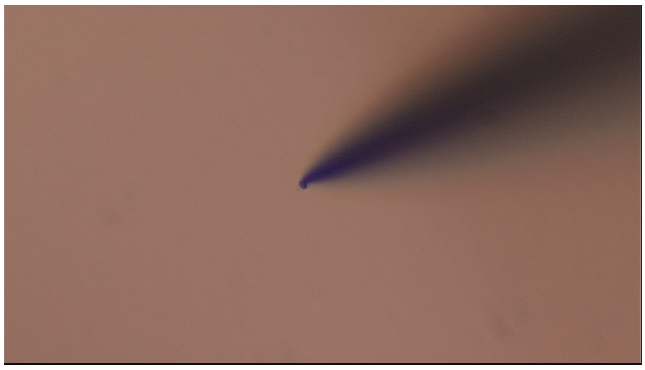

FIG. 4. 3μm SiC grain on micromanipulator needle.

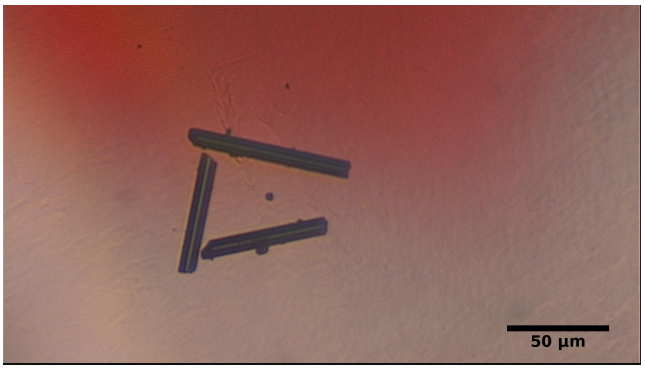

FIG. 5. 3μm SiC grain lying on the bottom of a resin-filled capsule. Grain is surrounded by carbon fibres. You can see part of the red dot on the opposite side of the capsule in the upper middle of the image.

VII. CURING

1. Once finished picking the grain, slowly add resin drop by drop to the capsule [dropping resin down side of capsule may be best]. Make capsule roughly 3/4 full.

2. Check position of fibres and grain under microscope. If the fibres and grain have moved, you may need to reposition them. Take another picture if things have moved.

3. Place sample in vacuum oven for at least 24 hours at 70^{o}C [The digital heater/thermometer will read 170^{o}F $\approx$ 77^{o}C, but the analogue thermometer will probably read 66^{o}C. The analog thermometer is more accurate.]

VIII. EPOXY RESIN BLOCK TO HEX SCREW

Supplies: 1 foil weigh boat, 2 aluminium jig blocks, 1 packet of epoxy with popsicle stick, wooden dowel cut in half, razor blade, small square of weigh paper, 1 hex screw.
Wear latex gloves to prevent getting oils on the hex screw and to prevent skin exposure to epoxy.

1. After curing, the resin should fill roughly $\frac{1}{4}$-$\frac{1}{2}$ of the capsule.

2. Capsule should sit in one Al jig. Screw hex screw into other Al jig so that hex screw head sits inside of the jig.

3. Trace circumference of blank capsule on weigh paper and cut out circle. This will provide a barrier so that the jigs don't stick together in case epoxy leaks out of the capsule. Place over top of jig containing the capsule.

4. Mix epoxy with popsicle stick in foil weigh boat.

5. With end of dowel apply roughly two drops of epoxy to inside of capsule. Do not overfill.

6. Nest jig with hex screw on top of the capsule and lightly turn screw until it stops/reaches the resin block. Do not overscrew.

7. Press jigs together and hold for 5+ minutes until epoxy has set. Epoxy outgasses as it dries so you must keep the jigs pressed together firmly.

8. Once set, remove the screw from the jig and place screw/resin block/capsule in kiln for a few hours.

9. Carefully slice away capsule from resin block with a razor blade.

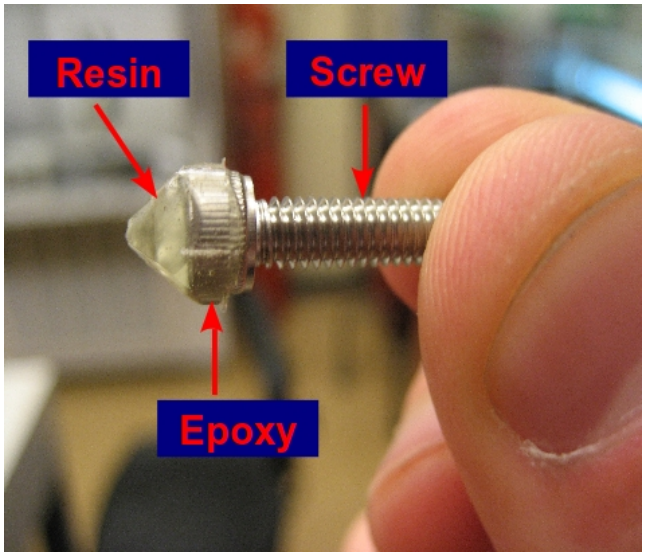

FIG. 6. Resin block epoxied to a hex screw. The tip of this resin block has already been trimmed with a glass knife to form a "Mayan pyramid".

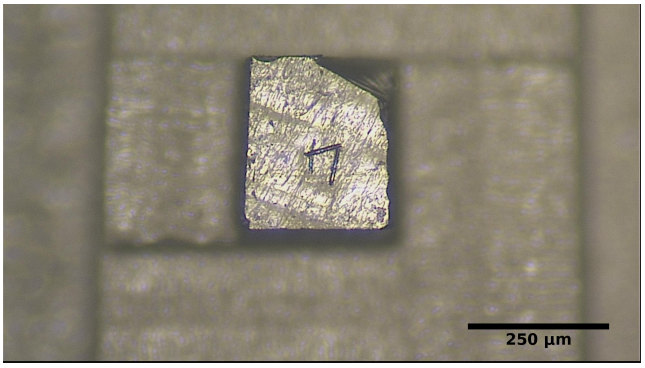

FIG. 7. Top-down view of a trimmed resin block. The top of the pyramid is roughly 250μm square. Carbon fibres are clearly visible.

IX. TRIMMING CAPSULE TIP

At the microtome station:

1. Fully retract knife holder and lock in position. Adjust knife holder angle to 8° for glass knives.
2. Remove chuck from microtome and secure screw in the chuck.
3. Place the plastic shield on the microtome arm and then secure chuck back to microtome. Level the sample arm. Be careful not to bump the sample.
4. Select a glass knife and place in knife-holder. Inspect the cutting edge (corner). Corner and edge should be completely flat with no chips or horns, see Fig. IX. Bad knives should be discarded in a glass recycling or sharps box.
5. Secure good knife in knife holder. Unlock knife holder stage and advance to roughly 1cm from the resin block. Lock the knife holder stage.
6. Focus eyepieces on the knife edge.
7. Rotate resin block so that the knife edge is parallel to the lower edge of the resin block top. Adjust the pitch of the resin block so that its entire top surface is vertically aligned.
8. Locate carbon fibres. Adjust knife position as to leave the carbon fibres centered in a 250μm square (at full magnification this is one square on the reticle.
9. Very slowly advance the knife towards the sample (turn main knob clockwise) while moving the sample up and down in slicing motions. Do not actually advance the sample! Only move sample up and down in the same plane by rotating sample advance knob back and forth. It is very very important that you only advance the knife!
10. When the knife is very close you will see its reflection in the resin block's surface. As you begin trimming, you may need to adjust the pitch of the resin block. Always retract knife before adjusting the sample.
11. Shave off resin from one side, retract knife, and rotate sample 90°, repeat.
12. You should make 2 steps or more in the "Mayan pyramid". Each step should be roughly as tall as it is wide.

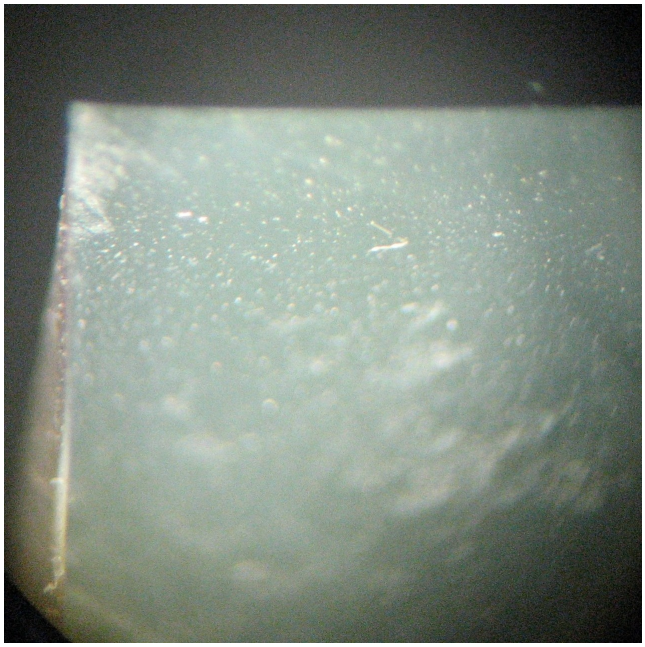

FIG. 8. Example of a "good" glass knife. Cutting edge is flat and unchipped. Image taken under max microtome magnification.

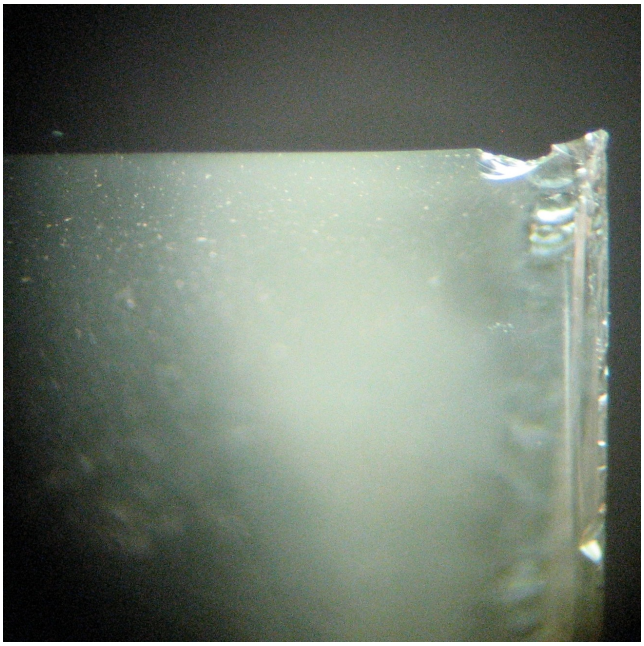

FIG. 9. Example of a poor-quality glass knife. Cutting edge has both a horn and chips out of it.