

PREPARATION OF POLISHED BULK SPECIMENS

EQUIPMENT & MATERIALS

Equipment:: Hot plate, oven, scale (opt.), plexiglas plate, aluminum rings, polishing wheel.

Consumables: Silicone grease, E-7 epoxy, toluene, aluminum weighing dish, wood applicator sticks, carbide grinding paper, polishing compounds and cloths.

SAMPLE REQUIREMENTS: Representative sample which will fit into either a 1/2" or 1" Al ring.

SETTING SAMPLE IN EPOXY

- 1) Turn hot plate to $\sim 70^{\circ}\text{C}$.
- 2) Grease plexiglas plate and Al ring with sample. Thoroughly coat the top surface of plexiglas plate and both the bottom and inside of Al ring with Dow Corning silicone grease. Place Al ring on plate with sample centered inside.
- 3) Pre-heat plexiglas plate. Place plexiglas on hot plate which allows the plexiglas to become hot enough to release air bubbles in the epoxy.
- 4) Weigh epoxy in Al weighing dish. E-7 epoxy is in two parts: A - resin and B - activator. By weight (or sight), the mixture should be 3 parts A to 2 parts B. Add 1-2 drops of toluene for lower viscosity.
- 5) Mix epoxy on hot plate. Place Al weighing dish with a finger formed pouring spout on the hot plate. Begin mixing with applicator stick until the epoxy is a translucent yellow and only a few small air bubbles remain.
- 6) Pour into mold. Pour epoxy to the top of the ring.
- 7) Leave plexiglas plate on hot plate until hard. Most of the bubbles should rise to the top. Dislodge stubborn bubbles with a broken applicator stick.
- 8) Cure for two hours at 67°C .
- 9) Remove mounted epoxy sample.

GRINDING: Two step rough (240) and fine (600) grit carbide grinding paper.

- 1) Level back of mount. With the 240 grit cloth and water, grind the back of the sample so it is flat.
- 2) Expose sample. Again with the 240, grind sample until it is sufficiently exposed.
- 3) Fine grind. With the 600 grit cloth and water, grind surface until large scratches are gone.
- 4) Clean sample. Clean surface with dilute micro and small paint brush. Rinse under running water. Dry with kimwipe.

POLISHING: Two step rough and fine polish using a high speed polishing wheel.

- Rough Polish: 3.0 μm diamond paste and Selected Silk polishing cloth.

- 1) Prepare wheel. If necessary, add 3.0 μm diamond paste to silk polishing cloth. Add a few drops of polishing oil to cloth. Rub oil and paste into cloth with a clean glass disk.
- 2) Polish sample. Using the slow speed, turn on motor, spin the wheel while holding sample on cloth. Rotate sample in the opposite direction of spinning wheel for 1-2 minute intervals. Add oil if needed.
- 3) Clean sample. See grinding, step 4.
- 4) Examine surface. Examine surface area under the microscope (3.5x objective) with reflected light to see if the 600 grit scratches are eliminated from the areas of interest.
- 5) Repeat steps 2, 3, and 4 until satisfied.
- 6) Clean sample. See grinding, step 4. Do not transfer paste and oil to next step.

- Fine Polish: 0.3 μm Al_2O_3 powder and Microcloth.

- 1) Prepare wheel. Saturate wheel with water. Spin wheel (slow speed) and remove excess water with kimwipes. Sprinkle Al powder on wheel. Spin wheel again rubbing Al compound into the wheel with a kimwipe. Check wheel preparation with a clean glass disk, rub it around the wheel 2 or 3 times and examine disk. You will see either:
 - 1) A thin water film slowly evaporating - OK.
 - 2) A thicker water film - too much water
 - OR 3) No water at all - too much Al powder.
- 2) Polish sample. As with the diamond paste, use the slow speed, rotating the sample opposite the wheel direction. Do this for 1 - 1 1/2 minute intervals. If the 3.0 μm polish was good and 0.3 μm preparation right this should only take a few minutes.
- 3) Clean sample. See grinding, step 4.
- 4) Examine surface. Use the 16x objective. There should be few if any scratches visible on surface and different compositional phases should be evident if present. Each small division on the ocular scale is $\sim 6.6 \mu\text{m}$.
- 5) Repeat steps 3, 4, and 5 until satisfied.
- 6) Clean sample. See grinding, step 4.