# PREPARATION OF GRAIN MOUNTS FOR EPMA OR SIMS ANALYSIS

#### Samantha Engwell

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#### **Equipment required**

- Struers SpeciFix-20 resin (7g) and hardener (1g) (or an approved alternative)
- Tweezers.
- Pipettes, polystyrene weighing boats and lollipop sticks for mixing resin
- Silicon carbide grinding papers with a range of grades (1200, 2500, 4000 grit) and a flat plate (glass) to support paper.
- 6 µm 3 µm 1 µm diamond laps, compounds and polishing lubricant.
- One-inch round and 5 mm deep aluminuim rings or 1" ID Teflon ring.
- A glass plate ( ~ 3 by 2 inch in size)
- Brown parcel tape.
- Vacuum chamber and/or pressure chamber.
- Ultrasonic bath for cleaning.
- Solvents for cleaning (Pet Ether, Decon90, water).

#### **Mount Preparation**

Attach a piece of brown parcel tape to the glass plate with the sticky side facing out, the tape can be secured and made taught using more tape and securing on the back of the plate.



Press the aluminium ring on to the tape so that are edges are secure and there are no gaps underneath from which the resin could seep. Run a small bead of epoxy around the outside of the ring to ensure a seal to the tape.



#### Sample mounting

Arrange the grains in lines on the tape within the aluminium ring using pointed end tweezers if the samples are quite large or a hair or needle if the grains are small. Be careful not to mix samples. Keep the grains to the centre of the mount. This is critical for isotopic ratio measurements.



#### **Resin Mixing**

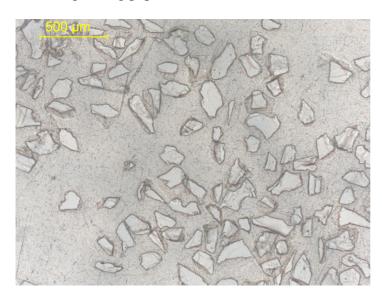
Make up a batch of resin in a polystyrene weighing boat. Each individual mount will require ~4 grams of resin. The resin should be mixed using a lollipop in the <u>proportion</u> 7:1 resin to hardener **by weight** (e.g. 14g resin to 2g hardener) **for SpeciFix**. Do not introduce bubbles while mixing, but ensure the two components are thoroughly mixed. You should continue mixing for at least 2 minutes. Do not try and mix small quantities because the errors in weighting may not produce the correct ratio mix.

After thoroughly mixing the resin and hardener, place in a vacuum chamber and evacuate to 20inHg - 250 Torr. The resin will bubble gently. Too high a vacuum will cause the resin to boil vigorously. After a few minutes, when bubbles have stopped appearing gradually reduce the vacuum and remove the resin from the chamber. Without stirring and using a disposable pipette, pour the resin into the aluminium ring ensuring it is filled to the top. Either return the mould to the vacuum chamber and evacuate to the same value as before for about 5-10mins, or leave under pressure in the Buehler pressure (~2.5Bar) chamber. The sample should be left to cure overnight. Do not cure at high temperature as this causes the tape to shrink. Remove the sample from the vacuum/pressure chamber.

## \* Leave the sample for at least 2 weeks before grinding \*

#### Grinding

The sample must be ground to produce a flat surface before polishing. This should be done progressively and carefully to produce a uniform, flat surface, with minimal loss of material. Grinding should be done sequentially through the 800, 1200 and 2500 grades. Ensure the papers are wet. A small amount of washing up liquid can be added to the water to help the block move easily over the grinding paper.



Photograph showing what mount should look like after grinding at 2500 grade.

### **Final Polishing**

Samples should be polished using the  $6\mu m$ ,  $3\mu m$  diamond and finally the  $1\mu m$  polishing lap for ~3min for  $6\mu m$ , 4-8min for  $3\mu m$  and 3-4mins for  $1\mu m$ . Cleaning between each stage is critical. View the finished product using a high powered reflected light microscope. If there are still small scratches on the surface polish for a further 3-4 minutes or until scratch free.