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A technique for mounting and polishing melt inclusions in small (<1 mm) crystals

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ABSTRACT

We have developed a method for mounting and polishing small crystals (<1 mm) to expose melt inclusions for analysis. Individual crystals are mounted in epoxy on one end of a 3.2 mm × 1.3 cm polycarbonate rod. The rod is inserted into a two-piece polishing tool with an adjusting screw to control the amount of sample exposed for grinding and polishing. Thus, individual crystals can be ground and polished so that features of interest (e.g., melt inclusions) are exposed on the polished surface.

INTRODUCTION

The study of melt inclusions has become an important tool for investigating petrogenetic processes because inclusions provide information not easily obtainable from studies of whole rocks (Schiano and Bourdon 1999). Preparation of crystals for analyses of contained melt inclusions requires special care because melt inclusions, reentrants, and hourglass inclusions in individual crystals occur at different depths beneath the polishing surface. Therefore, grinding and polishing may expose a melt inclusion in one crystal while melt inclusions in other crystals either are destroyed by polishing or remain unexposed beneath the surface. Further polishing would destroy any exposed melt inclusions and it may not be possible to distinguish between true inclusions and reentrants, especially if the sample was not observed before grinding and polishing. Some workers have avoided this problem by removing crystals from the epoxy (or other glue) as melt inclusions become exposed. These crystals are then placed into a separate mount containing only crystals with exposed melt inclusions. However, if the individual crystals are less than ~1 mm, removing the crystals and remounting them is difficult. We desired a technique that allows individual crystals to be ground and polished to different depths, and which allows the grinding progress to be controlled and monitored carefully so that pre-selected inclusions can be exposed individually for analysis.

Our technique was developed to study melt inclusions in <1 mm long zircon crystals (Thomas et al. 2002). The technique is applicable to other minerals in which melt inclusions are rare or in which selection of specific melt inclusions is necessary. The technique described here allows the maximum number of inclusions to be exposed for analysis. Similarly, the technique described here may be used to prepare small polished crystals for experiments in the hydrothermal diamond anvil cell (cf., Schmidt et al. 1998; Darling and Bassett 2001).

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MATERIALS

The polishing tool is a 2.5 cm diameter × 2.9 cm long piece of 316 stainless steel with a 3.3 mm diameter hole drilled through its center parallel to the long dimension (Fig. 1). The hole is threaded from one end to a depth of 0.7 cm and fitted with a threaded adjustment screw 2.9 cm long. This adjustment screw allows the amount of sample exposed beyond the surface of the sample polisher to be controlled precisely during grinding-polishing.

Before grinding and polishing, individual crystals are mounted in epoxy on a clear polycarbonate rod 3.2 mm in diameter and ~1.3 cm long (McMaster-Carr cat. no. 8571K11) (Figs. 1–3). After final grinding and polishing, the rods containing the polished crystals are inserted into a brass probe mount that holds 12 rods (Fig. 2). The probe mount is 2.54 cm in diameter and 1.3 cm thick with twelve 3.3 mm diameter holes drilled through the cylinder around the outer diameter (Fig. 2). The sample rods are held in place with small setscrews (2.3 mm) tightened with an Allen wrench (Fig. 2). The sample rods are placed into the probe mount by placing the probe mount face down on a flat surface, inserting the rods (sample-side down) and tightening the setscrews so that all the polished samples are secured at the same height.

CRYSTAL MOUNTING AND POLISHING PROCEDURE

Polycarbonate rods (~1.3 cm long) are inserted into the polishing tool and ground so that both ends are flat and perpendicular to the long axis of the rod, using 600 grit paper (Buehler Carbimet paper disks no. 30-5778-600) with water. Before inserting the rod into the polishing tool, a drop of water is placed in the hole. The surface tension of the water keeps the rod from falling out of the polishing tool. The polishing tool is placed on the 600 grit paper and the screw is continuously adjusted during grinding so that the end of the polycarbonate rod is in contact with the paper. The rods (including those containing attached crystals, as described below) are ground until the end

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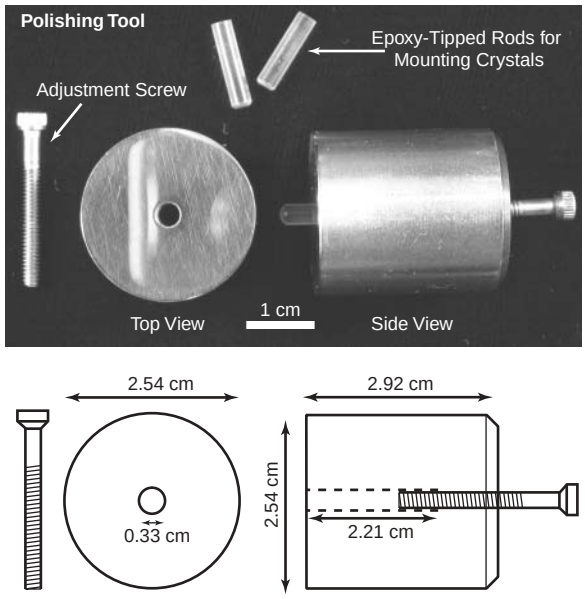

FIGURE 1. Photograph (**top**) and line drawing (**bottom**) of the polishing tool, adjusting screw and epoxy tipped rods for mounting crystals. In the photograph, the body, adjustment screw and two sample rods are shown to the left, whereas the assembled polishing tool with a rod and the adjustment screw inserted is shown to the right.

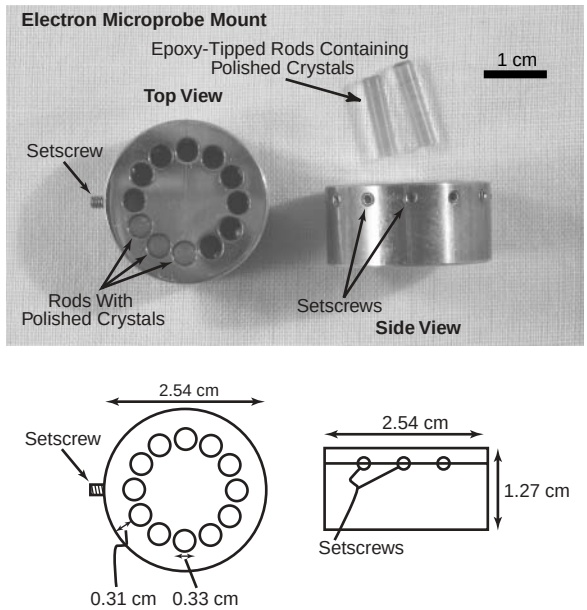

FIGURE 2. Photograph (**top**) and line drawing (**bottom**) of the electron microprobe mount and epoxy tipped rods containing polished crystals.

is flush with the much larger metal surface of the polishing tool. This procedure assures that the surface is flat and perpendicular to the long axis of the rod. After cleaning and drying the rods, ~200 µL of epoxy (Buehler Epoxide resin no. 20-8130-032 and hardener no. 20-8132-008) is applied to one end of each rod with a dissecting needle. Experience has shown that the best results are obtained if Buehler Epoxide resin and hardener are used because most other brands leave an excess of hardener after curing causing inadequate setting. The epoxy is mixed and allowed to cure for ~30 min prior to applying it to the rod tips to ensure that the resin and hardener have adequately mixed and reacted. Allowing the epoxy to cure for ~30 min before applying it to the polycarbonate rod increases its viscosity. This increased viscosity prevents the epoxy from flowing down the side of the rod when the droplet is applied. After the epoxy has hardened, epoxy tipped rods are ground to produce a flat surface (Fig. 3b) following the same procedures described above to produce flat surfaces on the rods. After grinding, the epoxy on the rod tips is ~25–50% of its original thickness. The epoxy tipped rods are cleaned, dried, and stored until needed to mount individual crystals. This first application of epoxy provides a base on which to mount the crystals and prevents them from becoming detached from the polycarbonate rod during polishing.

To identify crystals containing melt inclusions, the crystals are immersed in refractive index oils (e.g., Cargille Type A, cat. no. 16482) and observed with a binocular microscope (Fig. 3a). Selected crystals are cleaned in an appropriate solvent (e.g., toluene) to remove the oil prior to mounting in epoxy. To mount the crystal to the epoxy tipped rods, a small droplet of epoxy (<10 µL) is placed on the pre-flattened surface and spread to the appropriate thickness (thinner than the crystal to be mounted) with a dissecting needle or cotton-tipped swab. This thin layer of epoxy fixes the crystal to the surface prior to the final application of epoxy. Using a binocular microscope, a single crystal is placed onto the end of the rod containing the thin layer of epoxy, and a final drop of epoxy is added to cover the crystal. As above, epoxy should be mixed and allowed to react for ~30 min prior to application. After curing, the rods are placed into the polishing tool (Fig. 1) and ground with 1200 grit polishing paper (Buehler Carbimet paper disks No. 30-5778-012) and water. Grinding progress is monitored through repeated grinding and observation under a binocular microscope using reflected light until the crystal is exposed on the surface (Fig. 3c). After exposing the crystal surface, the rod containing the crystal is removed from the polisher and placed "on end" on a microscope slide and observed under a petrographic microscope using both transmitted and reflected light. The rod acts as an optical fiber and transmits light to the crystal. If additional grinding is needed to expose the melt inclusion, the sample is further thinned with 1200 grit paper until the melt inclusion (or any other feature of interest) is exposed. Once the desired level within the crystal is reached, final polishing (Fig. 3d) is achieved using 0.3 µm aluminum oxide-coated polyester film (Buehler Fibrmet sheets no. 69-3154) and water. It was found that crystals were less likely to break during this final polishing step if a paper towel was placed beneath the aluminum oxide-coated polyester film, rather than placing the film directly on a hard surface. Each sample is archived until sets of samples are ready for microbeam analysis.

Depending on the types of analyses to be conducted, the steps described below may vary. In our case, we generally first perform electron microprobe analyses followed by secondary ion mass spectrometry (SIMS) analyses. Samples from archived

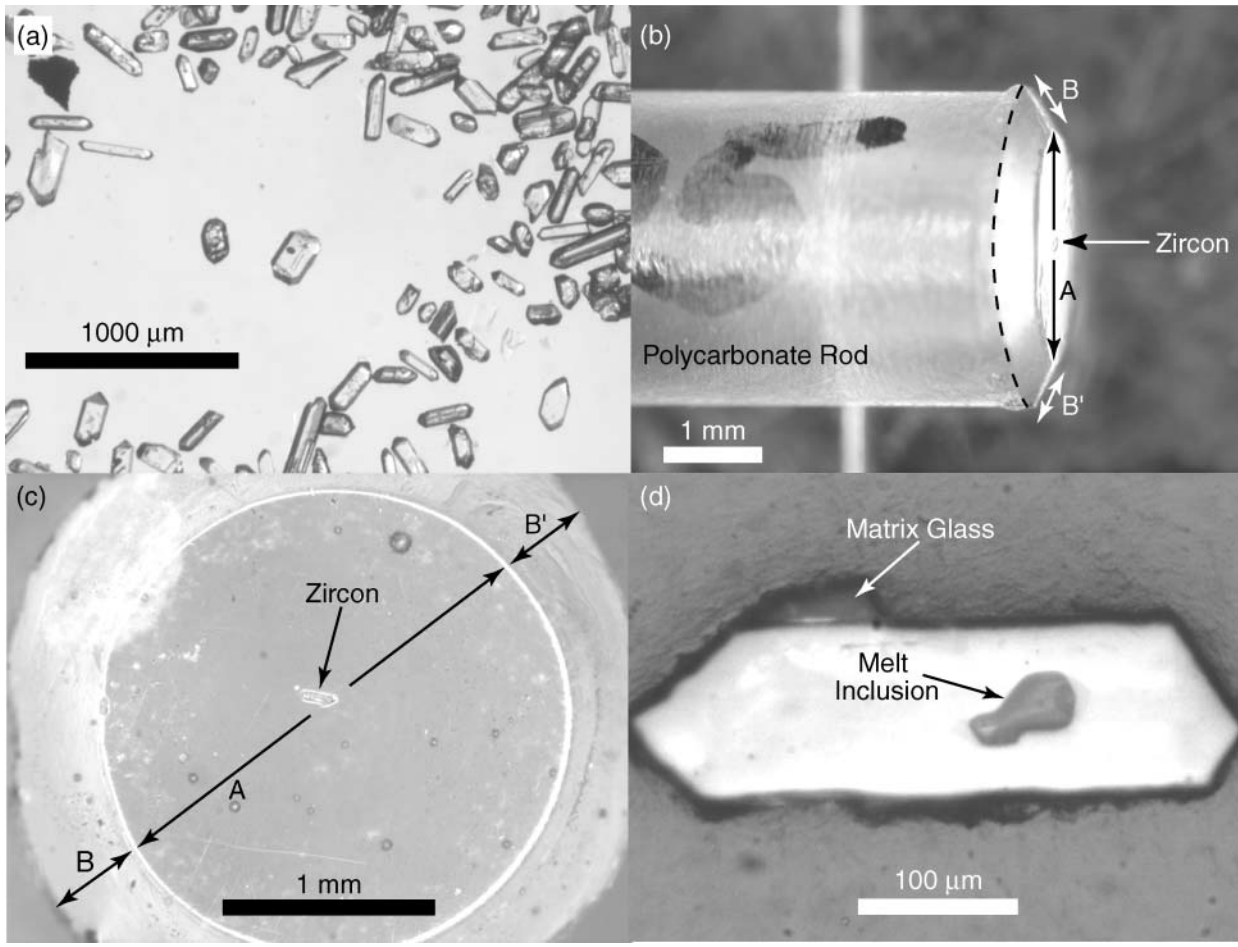

FIGURE 3. (**a**) Zircon crystals in refractive index oil (n = 1.515). Two crystals in the center containing melt inclusions have been separated from the other crystals. (**b**) Side view of a polycarbonate rod containing a zircon crystal in epoxy. The dashed line shows the boundary between the polycarbonate rod and epoxy. Line A denotes the diameter of the polished surface of the epoxy, and lines B and B' denote the edges of the polished epoxy droplet. Lines A, B, and B' are the same as those shown in (**c**). (**c**) Reflected light end view of a polycarbonate rod containing a zircon mounted in epoxy and exposed at the surface. The bright ~0.4 mm annulus surrounding the polished surface is the sloping edge of the original epoxy droplet (labeled B and B'). (**d**) Reflected light image of the same crystal as shown in (**c**) at higher magnification, showing the polished zircon crystal, melt inclusion, and matrix glass.

sets were selected for electron microprobe analyses and placed into the probe mount (Fig. 2) as described above. Samples that gave desirable results and which contained sufficiently large melt inclusions (>30 µm) were selected for further analysis using SIMS.

The mount used for electron microprobe analyses is not normally used to hold the samples for SIMS analyses because the polished crystals are not all at exactly the same height. This occurs because tightening the setscrews causes slight random displacements in the Z-direction. Minor height variations are not generally a problem during electron microprobe analyses because the stage has an adequate range of Z-direction movement. However, slight vertical variations from one crystal to the next are problematic during SIMS analyses due to the primary ion beam angle of incidence and low range of Z-direction movement. Prior to SIMS analyses, the sample rods are permanently cast in epoxy (Fig. 4). A 2.54 cm (outside diameter) standard aluminum ring form is placed on double-sided

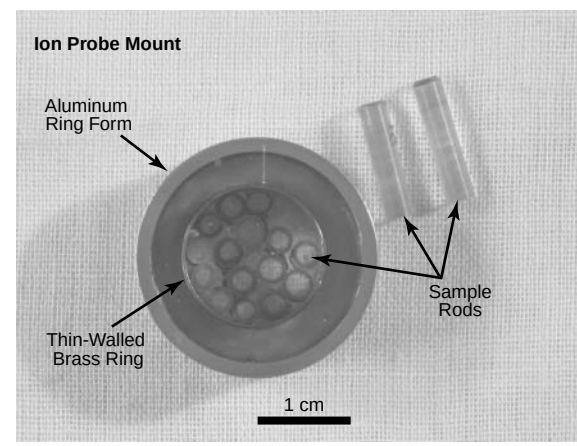

FIGURE 4. An ion probe mount with a 2.54 cm (outside diameter) standard aluminum ring and an internal 1.52 cm diameter thin-walled brass ring. The sample rods are contained within the internal ring in a close-packing configuration (~15 sample rods).

tape. A 1.52 cm diameter thin-walled brass ring is placed in the center of the larger ring, and the sample rods are placed inside this ring in a close-packing configuration (~15 sample rods) with the crystal-side placed against the tape. Both rings are filled with epoxy and allowed to cure prior to removal from the double-sided tape. Minimal (if any) polishing may be necessary to reduce minor height variations from one crystal to the next prior to SIMS analyses. We note that the ion probe mount described above may be inappropriate for H analyses of melt inclusions owing to the large volume of epoxy used (Dunbar and Hervig 1992). Additionally, other resins may be preferable to Buehler for high vacuum applications.

The technique described above was developed through trial and error, and has been found to be an effective means of exposing individual melt inclusions contained within small (as small as 50 μm) crystals. An added benefit of this technique is that undesirable samples may be culled and those giving the best results may be included in a final epoxy mount for further analysis. This procedure assures that all (or most) of the samples in the final mount will be suitable for SIMS analysis, minimizing the amount of time that has to be spent changing mounts. Finally, we note that the technique described here may also be suitable for preparing individual, small crystals for many other applications.

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