Letters

A method for the rapid mounting and polishing of ceramic materials for microstructural examinations

The present note reports a technique by which a variety of ceramic materials may be impregnated, mounted and polished within approximately 1 h.

To prepare polished sections of poorly bonded materials, it is necessary to impregnate the sample with a suitable binder to prevent damage occurring during the grinding and subsequent lapping stages - otherwise the presence of "pull-out" material during the lapping stage severely damages the whole section. Epoxy-based resins are excellent for this purpose, provided the material has relatively large interconnected pores which allow the resin to readily penetrate the sample. However, in those cases where the pore system is fairly fine, these resins are too viscous to be effective in any vacuum impregnation technique. In addition, other binders, such as the much lower viscosity acrylic-based materials, either require a relatively long curing period or they set with a mass of entrapped bubbles that retain grinding debris during the subsequent stages, leading to contamination of the polishing laps and severe scoring of the section.

These limitations are avoided by the use of a low viscosity binder which is rapidly cured at

elevated temperatures under a high isostatic pressure. In this manner, samples may be satisfactorily impregnated and mounted within 15 min.

The technique for impregnating and mounting the samples follows conventional procedure in part. Equal volumes of each component* are mixed and stirred for approximately 5 min until the mixture thickens. This viscous material is then used as a thin layer to seal the temporary joint between a clean glass slide and a piece of glass tubing 4 cm diameter by 1.5 cm high. The sample is placed within the enclosed area and freshly mixed (very fluid) material is poured into the mould. The mount is then transferred to a small heated pressure chamber and subjected to a temperature between 85 and 100° F at a nitrogen gas pressure between 1.38 and 3.07 MPa (200 and 450 psi) for a minimum of 10 min. Satisfactory results are obtained at 90 to 100° F for 15 min at 3 MPa. Under these conditions, the methacrylate readily impregnated the sample and is rapidly cured. The high gas pressure prevents the material boiling and also collapses any bubbles present in the mixture during the pouring stage. Fig. 1 indicates that cracks in the sample are readily penetrated.

The mounted samples are subsequently roughly finished on a $30 \,\mu\text{m}$ fixed-diamond, nickel-plated lapping wheel, followed by hand lapping on a glass plate with an aqueous slurry of 1000-mesh

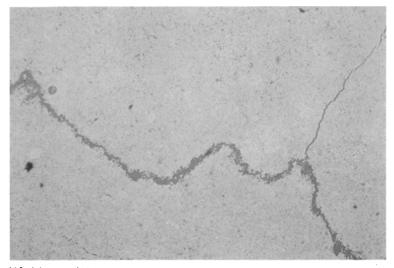


Figure 1 Polished section of a synthetic hollandite showing cracks ranging in width from 2 to $100 \,\mu\text{m}$ completely penetrated by plastic. (\times 56.25).

*'Quickmount' (available from Fulton Metallurgical Products Corp., 4716 Ellsworth Avenue, Pittsburgh, Pa. 15213) and methyl methacrylate (available from Eastman Kodak Co., Rochester, N.Y. 14650).

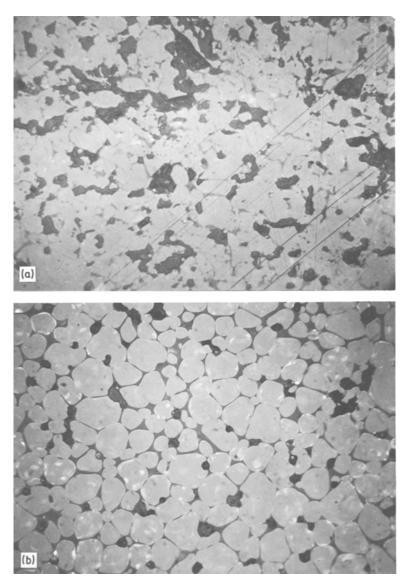


Figure 2 Polished sections of a commercially available silicatebonded zirconia refractory (\times 56.25) (a) Polished on successive lead laps using loose diamond powder and a 1:1 mineral oil: kerosene lubricant. Final polish with 0 to 1 μ m diamond powder, (b) Lapped on glass plate with 1000-mesh Carborundum powder and polished on a Politex-Pix lap with Syton.

Carborundum grit until a uniform matt finish is obtained. Any resisual abrasive is removed using an ultrasonic bath. The total time for these stages is approximately 15 min.

The samples are given a final polish on a synthetic cloth lap* rotated at 150 r.p.m. and impregnated with a commercially available colloidal silica polishing fluid[†]. After 10 min, this final polishing stage is completed. A comparison of the finish produced by this method and by conventional polishing of a silicate-bonded zirconia is shown in Fig. 2.

*Politex-Pix available from MicroMetallurgical Ltd., Thornhill, Ontario. †Syton available from Monsanto Canada Ltd., Toronto, Ontario. 906

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