

mixture, where the identification based on the powder pattern is often difficult.

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REFERENCES

- ANDREW, R. W., M. L. JACKSON AND K. WADA (1960) *Soil Sci. Soc. Amer. Proc.* **24**, 422-423.
- BISCAYE, P. E. (1964) *Amer. Mineral.* **49**, 1281-1289.
- BRINDLEY, G. W. (1961) G. Brown, ed., *The X-ray Identification and Crystal Structure of Clay Minerals*. Mineralogical Society, London, 85 and 262.
- HIGASHI, T. AND S. AOMINE (1958) *Soil Sci. Plant Nutr. (Tokyo)*, **7**, 167-177. [in Japanese]
- KELLER, W. D., E. E. PICKETT AND A. L. REESMAN (1966) *Proc. Intern. Clay Conf.* **1966**, 75-85.
- WADA, K. (1961) *Amer. Mineral.* **46**, 78-91.
- (1965a) *Amer. Mineral.* **50**, 924-941.
- (1965b) *Clay Sci.* **2**, 101-110.
- AND S. AOMINE (1966) *Soil Sci. Plant Nutr. (Tokyo)* **12**, 151-157.
- WEISS, A., W. THIELEPAPE, W. RITTER, H. SCHÄFER AND G. GÖRING (1963) *Z. Anorg. Allgem. Chem.* **320**, 183-204.

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HIGH PRESSURE EPOXY IMPREGNATION OF POROUS MATERIALS
FOR THIN-SECTION AND MICROPROBE ANALYSIS¹

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The need for preparing thin sections and microprobe samples from porous and friable rock specimens such as altered pyroclastics and manganese nodules led to devising a successful method of high pressure impregnation with a thermally-resistant epoxy resin. The method described below provides thorough penetration and rigid cementation of materials which could not be treated satisfactorily by conventional means. In brief, the process consists of encapsulating small specimens in epoxy-filled rubber bags after vacuum treatment to remove trapped gases, followed by compression in a cylindrical die at 12,000 lb/in² to drive in

¹ Contribution from Scripps Institution of Oceanography, New Series.

the resin. After curing, samples may be sliced, ground, and polished by conventional means.

Although we have used this method so far only for a few rock types, it suggests itself as being superior to current methods used for mounting friable rock and mineral specimens intended for thin section and microprobe work. It also appears suitable for impregnation of unconsolidated aggregates composed of clay-silt and sand-size grains.

EQUIPMENT AND MATERIALS

The compression cylinder used was a standard 1 inch diameter molding die for metallographic polished sections. Pressure was applied to the die plungers with a laboratory press of suitable capacity.

The selection of mounting media is critical in microprobe applications because only media with high thermal resistances can be employed for objects smaller than about 40μ because part of the polished medium surface will be exposed to the electron beam during scanning procedures. Even in specimens where a stationary beam can be focussed within an area of a few square micra it is difficult to avoid inadvertent exposure of the mounting medium to the stationary beam. In both cases, most ordinary organic impregnation and mounting media decompose more or less rapidly, leading to contamination of the optical system and other components in the vacuum chamber.

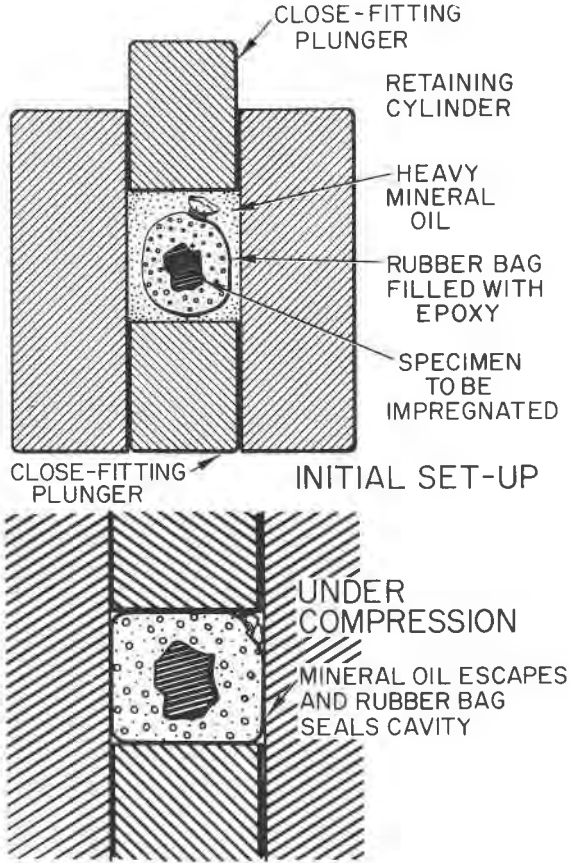
Arrhenius *et al.*, (unpublished) selected Epon 828 (Shell Chemical Co.) as having satisfactory thermal properties for microprobe application; this medium was therefore selected for the impregnation process described herein. Epon 828 is catalyzed by Shell Chemical Curing Agent "Z". Pot life is long enough to permit preliminary preparation of equipment and thorough impregnation after specimens are introduced in the pressure apparatus.

PROCEDURE

The sample is thoroughly dried. A suitable portion of the mixed resin, 10–15 ml, is heated to about 60° in a temperature-controlled oven. When this temperature is reached the viscosity markedly lowers, and the sample, previously heated to this temperature also, is immersed in the resin. To preserve the lowered viscosity of the resin, it is helpful to also warm the die and plungers to the same temperature. The resin container, suitably a polyethylene cup, is immediately transferred to a bell jar and evacuated until bubbles cease to emerge from the sample. If very porous, the sample should be subjected to another cycle of warming followed by evacuation.

The specimen and resin are then transferred into a rubber bag, a finger

of a rubber glove being suitable for this purpose, or a small surgical finger cap. Enough resin is added to cover the specimen entirely. The bag is closed with loops of twine, tied securely just below the epoxy level to exclude air. The excess twine and rubber are cut off and the bag is inserted in the press die into which a few milliliters of mineral oil have been



previously added. More oil is poured in until the bag is just covered. The bottom plunger of the die assembly is moved upward until the oil level reaches the top of the die cavity; at this point the upper plunger is inserted, excluding as much air as possible. The die is placed in the press and the plungers compressed at about 12,000 lb/in². In the type of die used, the oil is squeezed past the plungers until the rubber bag seals the cavity; at this point the pressure rises sharply and thereafter drops only slowly. Readjustment of pressure may be necessary in the event of

leakages in the apparatus. At the end of an hour, the impregnation is complete for specimens of about one to two cubic centimeters.

After impregnation, the die assembly is dismantled. Because the rubber bag may pinch along the plunger-cylinder interfaces, the plungers may have to be pushed out forcibly by using a smaller diameter plunger. The bag commonly breaks during the extraction process and care must be exercised to avoid excessive skin contacts with the resin. All die parts should be cleaned immediately with acetone or ethyl methyl ketone before polymerization sets in. The specimen is removed from the bag, the excess epoxy wiped off, and the specimen placed upon a suitable substrate to cure for the time and temperature specified by the manufacturer of the resin. In the case of Epon 828, overnight curing at about 60° is sufficient. If the nature of the materials being impregnated permits, another curing for several hours at about 100–120° serves to harden the resin still more. However, direct polymerization at the higher temperature mentioned results in excessive internal strain in the mount. If the specimen is to be cut later by precision sawing it is advantageous prior to epoxy polymerization, to place the specimen upon a slip of plywood or other suitable substrate which will later permit clamping in the feed mechanism. Small specimens of hyaloclastites and manganese nodules mentioned above were found to be completely impregnated, enabling slices as thin as a few millimeters to be cut on a precision saw. While this method cannot succeed in impregnating extremely dense aggregates of clay minerals, it can fill the very fine cracks formed in such aggregates during drying, thus creating a sort of breccia less liable to disintegrate during subsequent surface preparation.

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KINETIC CONSIDERATIONS IN THE GENESIS OF
GROWTH TWINNING: A DISCUSSION

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Donnelly (1967) criticizes current theories of growth twinning with emphasis on the energetics involved because of the kinetic nature of the phenomenon. It is concluded that the energy differences between the twinned and the untwinned states may be of minor importance, and that growth twins may result because of the more favourable facial arrangement obtained by the twinned configuration. However, similar considerations were put forward by Billig (1954) who showed that the twinning of germanium crystals grown from the melt is not due to a haphazard event and occurs if the actual growth direction deviates appreciably from the