Digital Computer Laboratory
Massachusetts Institute of Technology
Cambridge, Massachusetts

SUBJECT: POLISHING SPECIMENS OF FERRITES

To: David R. Brown

From: F. E. Vinal

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Abstract: Phase relationships in ferrite bodies and correlation with loop squareness and other electrical and magnetic properties have raised questions about polishing specimens for photo-micrographic and thin-section study. This memorandum points out some of the technique differences for handling ceramic-type materials over the somewhat more familiar metallographic techniques.

Techniques for polishing ceramic-type specimens must be somewhat modified over those used for metallographic specimen preparation. Characteristics of ceramics which necessitate the modification are primarily their hardness, porosity, if any, and notoriously low tensile strengths as compared to metals. The standard practice with ceramic materials of this character is to impregnate them with resinous material before polishing. This technique will permit the specimen to be polished to a high degree without the loosening of grains at the surface of the specimen. The general technique consists of dipping the specimen, then permitting the resinous material to penetrate and harden within the pores. The penetration will be dependent upon the size and number of the pores and of the surface tension and viscosity of the resin. This technique does not give complete impregnation but it should penetrate well below the surfaces and may be sufficient for rough examination.

A good deal of our interest, however, will not lie in surface examination but rather in sectional examination; and in order to apply the same techniques, it will be necessary for the resin to penetrate the specimen completely or surface impregnate a sectional specimen. More precautions are required in the complete impregnating technique and this is usually accomplished by placing the specimens in a shallow dish in a vacuum desiccator. Through the top center hole is placed a dropping funnel by means of which the impregnating resin is permitted to flow on to and completely cover the specimen after a vacuum has been drawn on the desiccator. Before the resinous material hardens, a short period is allowed for any trapped air to work out of the power and then normal atmospheric pressure is admitted to the desiccator which tends to force the resin well into the pores. Various materials have been used for the impregnation--a combination of Canada Balsam and Shellac, high-purity phenol formaldehyde mixtures for polymerization and methyl methacrylate type materials. After impregnation, small specimens are then mounted in a larger block of plastic by well-known techniques to facilitate handling on the lap. If simple polished surfaces are required, a specimen mounted near the bottom of the plastic block is polished in a manner much in analogous to those of metallographic specimens. However, because of the hardness and abrasive nature of ferrite materials, the grinding medium used in the lapping must be harder than those used for polishing most metallographic specimens. Fine grain Carborundum is the starting abrasive usually followed by diamond dust or a diamond impregnated wheel. Sometimes this is sufficiently fine to complete the polishing of the specimen; but if a higher finish is desired, various very fine polishing mediums are available of alumina. The impregnating resin is often tinted with an organic die to differentiate it clearly from colored cyrstals and matrices in the ceramic. 0.5 grams of crystal violet dissolved in 100 ml of resin should give a satisfactory tint.

Among the **impregnants** commercially available, the Buehler Co., manufacturers of metallographic polishing equipment offer a "light colored" bakelite resin but a clear or tinted clear plastic is to be preferred. The most promising impregnant is a methyl methacrylate monomer. This thin, volatile liquid will polymerize, by linkage through the vinyl grouping in its structure, under the influence of heat or light or catalysts. It should therefore penetrate pores and be polymerized in situ without the necessity of removing solvents as would be required for phenol-formaldehyde or Bakelite type of resins such as is supplied by the Bakelite Corp. as their number BL-3128. The methyl methacrylate monomer (see Information Bulletin number X-28 of the DuPont Polychemical Department) has the further advantage of being physically and chemically compatible with the more familiar granular methacrylate compression molding powder used for block mounting specimens. The monomer must be freshly prepared as used by removing the inhibitor added to prevent polymerization during shipment or storage. This is done by vacuum distilling the monomer at 61° C and 200 mm pressure, or lower temperatures and lower pressures, through a short, packed column. The distilled monomer may be stored for short periods at a temperature not over 40° F (refrigerator). The monomer is polymerized by heating to the temperature of a water bath under reflux for four hours. A similar procedure and information sheet may be obtained from Rohm and Haas Co. entitled "Embedding Specimens in Plexiglass."

It should be pointed out, however, that the polishing of specimens is probably only a half-way point as far as interest and information obtainable is concerned with ceramic-type materials. Petrographic and ceramic materials are usually examined in thin sections as well as surface examination of a larger mass because most of the ceramic grains and matrices have considerable transparency when sections of a few mils thickness are prepared. It is, therefore, possible to see through the specimens and place the grains of the various phases with relation to each other or a matrix, if such is present; or if no matrix is present, observe the character of the material and the sintoring which has taken place. It is also usually possible through the use of oils of known refractive indexes to determine what the refractive index is of various phases; and in the case of better known materials, these data are of considerable value for purpose of identification of the various phases. Since ferrite thin sections will contain crystals, etc., which are strongly colored, it might be well to consider taking photo-micrographs of particularly interesting specimens in color. The Ektacrome process is well suited for this as cut film is readily available and the process has the added advantage that excellent color transparencies and prints may be developed by any novice who can follow directions. The use of the polarizing microscope frequently gives informative birefringence colors showing domains and other structural features in ferroelectric materials and to a lesser extent for ferromagnetic materials. These birefringence patterns may also be well recorded with the Ektachrome process.

Signed Francis E. Vinal
Francis E. Vinal

Approved DRB
David R. Brown

FEV/jk

cc: L. Gold
J. H. Epstein
D. Epstein
G. Economos
J. H. Baldrige
D. Wickham
R. Adler
J. Sacco
H. F. Priest
S. A. Kulin