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INFILTRATION OF METALLOGRAPHIC SPECIMENS WITH METALLIC ALLOYS

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OAK RIDGE Y-12 PLANT
OAK RIDGE, TENNESSEE

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**INFILTRATION OF METALLOGRAPHIC SPECIMENS
WITH METALLIC ALLOYS**

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ABSTRACT

A simplified technique has been devised for infiltrating porous and particulate materials with a metallic alloy for metallographic processing. This technique has been found suitable for processing production quantities of metallographic specimens for microexamination and electron microprobe analysis.

CONTENTS

SUMMARY	5
INTRODUCTION	6
INFILTRATING METALLOGRAPHIC SPECIMENS	7
Discussion	7
Conclusions	10
REFERENCES	13

SUMMARY

A metallographic technique was developed which provides for the infiltration of porous and particulate materials with a low-melting-point metallic alloy. The mounting operation can be performed on a standard metallographic mounting press with one slight modification, namely: inserting two high-temperature seals into the mold cavity to constrain the molten alloy. This procedure offers the following advantages over conventional thermoplastic mounting materials:

1. Electrical continuity is maintained through the mount to the specimen surface which is essential for electrolytic etching and anodizing of micrometer-size particulates.
2. Mechanical pullout of material is avoided.
3. The metallic mount is stable in an electron beam, and causes no contamination in an electron microprobe.
4. No additional grounding is required for electron microprobe analysis.

INTRODUCTION

An anodizing technique was required at the Oak Ridge Y-12 Plant^(a) for providing phase analysis of 25-micrometer nominal-diameter particles. Conventional thermoplastic mounting materials could not be used because of their electrical insulating properties. An alternative to the thermoplastic materials was copper diallylphthalate. However, this material was found to be unsatisfactory due to poor bonding of the particles which resulted in the loss of a large percentage of the particles during the grinding and polishing operation.

At the same time, a search was being conducted for a mounting technique for electron microprobe specimens which provided electrical continuity for grounding purposes and also prevented mechanical pullout. This report describes a method which completely satisfies these requirements.

(a) Operated for the US Atomic Energy Commission by the Union Carbide Corporation's Nuclear Division.

INFILTRATING METALLOGRAPHIC SPECIMENS

DISCUSSION

Metallographers have used low-melting-point alloys for encapsulating specimens for many years. The most commonly used alloys are combinations of lead, tin, and bismuth and are known as Wood's metal after its originator.⁽¹⁾ The approach used in the past has been to melt the alloy and simply pour it around the specimen to be examined, resulting in a specimen mount which is conductive and stable in an electron microprobe beam. However, any porosity inherent in the specimen remains unfilled and makes these areas subject to being mechanically dislodged by grinding and polishing the metallographic specimen. This procedure likewise was unsatisfactory for the encapsulation of micrometer-size particulates due to the fact that the particles floated in the dense alloy.

With the application of pressure, any porosity or particle interstices can be effectively infiltrated with the molten alloy. The size of the pore opening or particle interstice which can be filled with the alloy is governed by the following relationship:⁽²⁾

$$D = - \frac{4\sigma \cos \theta}{p},$$

where:

p represents the pressure exerted on the liquid,

D the diameter of the smallest opening filled at pressure, p ,

θ the contact angle between the liquid and the material being infiltrated, and

σ the surface tension of the alloy.

The contact angle is 140 degrees and the surface tension is about 480 dynes/cm for mercury and ceramic materials. Using the values, which should be a reasonable approximation, the equation reduces to the following:

$$D = \frac{213}{p}.$$

At a pressure of 5,315 psi, all porosity larger than ~ 0.04 micrometer is filled with the molten metal.

It seemed obvious that pressure infiltration would yield a desirable metallographic mount. However, most of the available pressurizing equipment (isostatic presses or high-pressure autoclaves) would be much too cumbersome and complicated for use on a production basis. Attention was then directed to a conventional mounting press (Figure 1) which is normally used with thermosetting or thermoplastic mounting materials. The press comes equipped with a slip-on, interchangeable, thermostatically controlled heater and a cooler. Since this type of press was designed for compression molding materials, seals had to be provided to



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Figure 1. METALLOGRAPHIC MOUNTING PRESS.

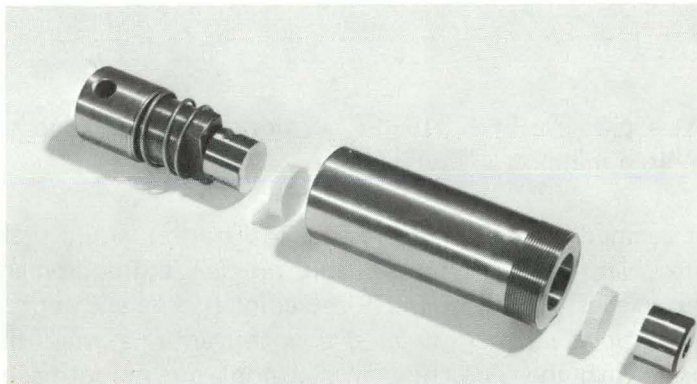
rubber gaskets were formed slightly oversize (1.005" D) in a aluminum mold that contained 100 holes 1.005 inch in diameter by one-quarter inch thick. A catalyst was added to the rubber, poured in the mold, and allowed to set overnight. These gaskets performed satisfactorily and could be used several times before failure occurred. Before each use, both gaskets are inspected for tears and cuts, which would result in pressure loss, and were replaced when required.

The metallic charge can be placed in the mounting press in either of two ways: (1) a pre-formed cylinder can be placed in the mold and then allowed to melt, or (2) molten metal can be ladled from a hot plate directly into the mold. The latter method has been used almost exclusively because it eliminates the need for a hold time in which to permit the alloy to melt. The specimen, if solid, can be placed

contain the molten metal at moderate pressures. Gaskets at the ram and closure assembly were used to seal the system.

A fluorinated hydrocarbon was chosen as the first candidate for the sealing material because of its excellent resistance to thermal decomposition and its ability to resist the adhesion of various materials. Cylindrical discs (1/4" T x 1.005" D) were machined for use as gaskets on either side of the molten metal. Since the gaskets were slightly larger than the 1.00-inch-diameter mold cavity it was anticipated that they should contain the molten alloy. In practice, the gaskets performed rather erratically resulting in a loss of metal to the interior of the press on occasion. Figure 2 is a photograph of the mold assembly; Figure 3 gives a sectional view of the mounting press with the gaskets in place.

A material was sought which could be repeatedly heated and pressurized with no loss of efficiency as a seal. Silicone



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Figure 2. MOUNTING PRESS MOLD ASSEMBLY.

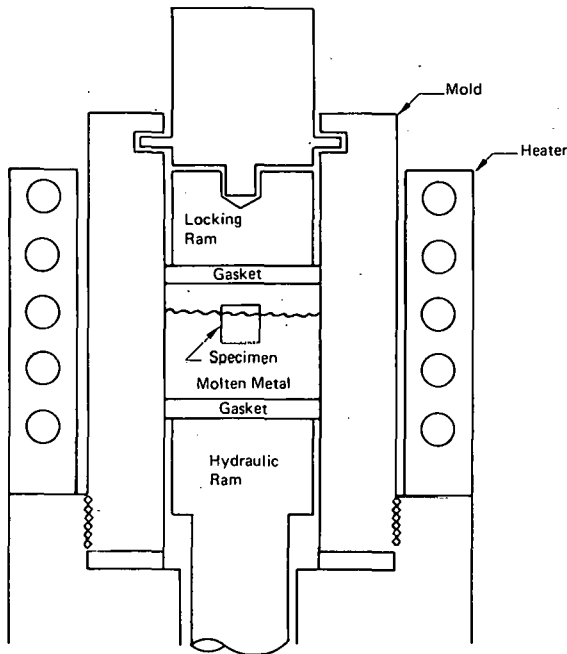


Figure 3. PRINCIPAL COMPONENTS OF THE MOUNTING-PRESS MOLD ASSEMBLY.

on top of the molten metal where it will either sink or float, depending on its density. It may be desirable to fix the specimen in such a manner as to view its edge. In this case the specimen must be supported in some manner. If the sample is in powder form, pouring the powder loosely into the molten metal is unsatisfactory for two reasons: (1) there would be a high scatter of material across the entire face of the sample, making it undesirable for rapid particle analysis and microprobe analysis; (2) if a variety of phases were present in the powder specimen with various densities, a segregation could result. A thin-walled aluminum or copper ring approximately one-quarter inch in diameter and one-quarter inch in height was found to be an adequate container for the powder when the bottom was sealed with cellophane tape. The ring should be tapped slightly to remove any trapped air and provide a close

compact, but not to a point where settling of the particles would cause extensive segregation. The ring containing the powder and sealed at the bottom acts like a boat floating on the liquid metal in the press. Upon applying pressure, the tape breaks and the liquid metal flows in under that pressure allowing very little particle movement and providing complete infiltration.

Any of several alloys can be used as the mounting material. The one which has been used most frequently is Cerrocast which melts at 270° F. This alloy was chosen because of its relatively high hardness and its unique property of expanding upon changing from a liquid to a solid. The expansion on solidification causes each particle to be tightly bonded in the matrix, thereby minimizing particle loss during processing. Table 1 lists the properties of several of the many alloy combinations that are available.

Since the mounting material is very soft, rounding of the edges of the specimen can occur unless special techniques are used to minimize this effect. Diamond abrasives have been found to yield superior results; however, alumina abrasives are satisfactory if hard (short napped) polishing cloths are used. Grinding is accomplished on an Automet apparatus using 120-grit progressively through 600-grit silicon carbide abrasive paper. The polishing is done on nylon cloth first with 0.3 micrometer alumina abrasive and water followed by a light polish using 0.05 micrometer alumina abrasive with water as a carrier. When diamond is used, the rough polishing proceeds using 6-micrometer diamond with nylon cloth followed by 3-micrometer diamond on nylon cloth. Final polishing takes place on nylon cloth using 1/4-micrometer diamond abrasive in an oil carrier.

Table 1
 PROPERTIES AND COMPOSITION OF SEVERAL "CERRO" ALLOYS

Properties	Cerrolow	Cerrobase	Cerrosafe	Cerrocast
Melting Temperature (° F)	117	281	No definite melting point	
Temperature Range (° F)	117 - 117	281 - 281	160 - 190	281 - 338
Density, 20° C (gms/cc)	8.9	8.7	9.4	8.2
Tensile Strength (lbs/in ²)	5400	8000	5400	800
Brinell Hardness	12	22	9	22
Coefficient of Thermal Expansion (10 ⁻⁵ /° C)	2.5	1.5	2.4	1.5
Volume Change, Liquid to Solid (%)	-1.4	-1.5	-2.0	+0.5
<u>Composition (wt %)</u>				
Antimony	-	-	-	-
Bismuth	44.7	55.5	42.5	40.0
Cadmium	5.3	-	8.5	-
Indium	19.1	-	-	-
Lead	22.6	44.5	37.7	-
Tin	8.3	-	11.3	60.0

Figure 4 is a photomicrograph showing the appearance of metallic particles which have been infiltrated with Cerrocast alloy, processed using alumina abrasives, and subjected to a surface anodizing treatment. Various colors are produced in the individual particles and are the result of the anodic film buildup which is a function of the composition of the particles. In this photomicrograph, the tungsten particles appear blue in color while the aluminum particles are white.

A photomicrograph of a porous material infiltrated with Cerrocast alloy is presented in Figure 5. This specimen represents friable materials which normally give rise to rounding of particles and pore openings during metallographic processing. The bonding action of the Cerrocast alloy prevents this type of defect from occurring. In this figure, taken using sensitive-tint illumination, the carbon appears dark while the Cerrocast appears lighter. The Cerrocast metal enhances the contrast between the graphite and porosity which makes it possible to run porosity analyses using automated image analysis techniques.

CONCLUSIONS

A technique was developed for mounting porous materials or particulates down to one micrometer in diameter in a metallic matrix. Using this technique poses no problems for mounting and processing quantities of metallographic specimens for optical examination or electron microprobe analysis. Expansion of the metal matrix upon solidification provides for minimal pullout in both particulates and porous materials. In addition, the metallic mount provides a conductive path which will facilitate anodizing, electropolishing or etching, and electron removal during microprobe analysis without contaminating the electron beam column.

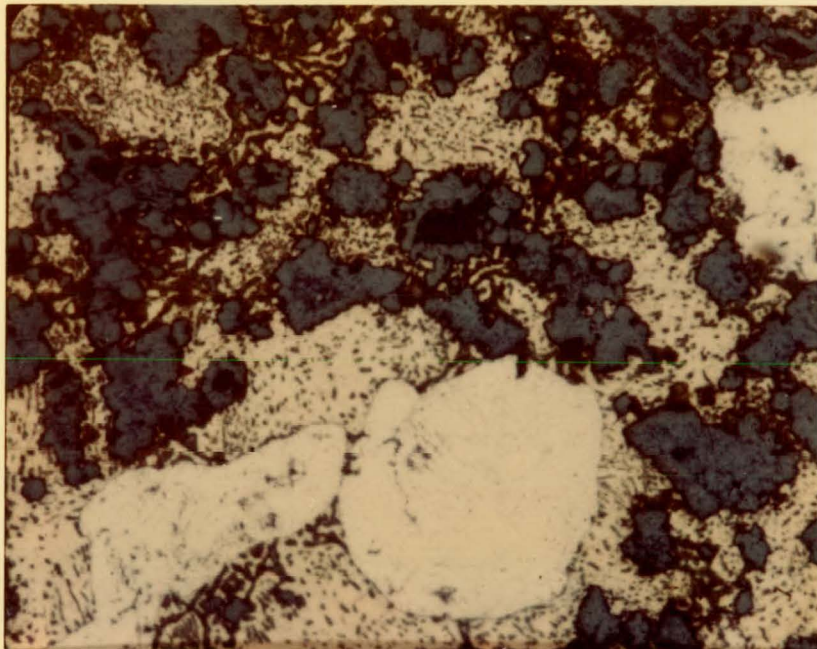


Figure 4. ANODIZED PARTICLES OF TUNGSTEN AND ALUMINUM IN CERROCAST ALLOY. (600X)



Figure 5. POROUS CARBON MOUNTED IN CERROCAST ALLOY. (Sensitive Tint Illumination; 500X)

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