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METALLOGRAPHIC EXAMINATION OF POROUS MATERIALS

MASTER

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UNION CARBIDE CORPORATION
NUCLEAR DIVISION
OAK RIDGE Y-12 PLANT

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ABSTRACT

A metallographic procedure that allows a delineation between the original porosity and mechanical "pull-out" porosity of a porous material has been developed.

CONTENTS

SUMMARY

A procedure was developed to examine the original structure of a porous material. This procedure consisted of impregnating the pores with a low-melting alloy and preparing the specimen for photomicrography by standard metallographic grinding and polishing techniques. By this procedure, a differentiation can be made between the original porosity of the porous material and mechanical "pull-out" porosity due to the grinding and polishing operation.

INTRODUCTION

It was desired to examine the microstructure of porous materials without damage due to metallographic grinding and polishing operations.

Since most porous materials are friable, a method was required which would preserve the original structure, and prevent a "pull-out" of grains during the grinding and polishing operation.

This report describes the method which was found to accomplish these objectives successfully.

METALLOGRAPHIC EXAMINATION OF POROUS MATERIALS

PREPARATION PROCEDURE

The developed process consists in impregnating the pores of the material with a low-melting alloy.

Sample Preparation for Cerrobend Impregnation

1. Place the sample of the porous material in the bottom of a small stainless steel cup (about 1" ID and 1" deep with walls about 1/8" thick). Secure the sample to the bottom of the cup with a small amount of epoxy cement.

2. Place a sufficient quantity of Cerrobend alloy[a] (MP, 70° C) in the cup to furnish enough liquid alloy to cover the sample. Place the cup in a large stoppered test tube fitted with a side arm which is connected to a vacuum pump.

3. Immerse the tube in a heated oil bath (~ 140° C) and turn on the vacuum pump. After the alloy melts and covers the sample, continue the evacuation for a few moments to insure complete removal of gas from the pores of the sample.

4. With the molten alloy covering the sample, turn off the vacuum and remove the sample cup from the tube.

Pressure Impregnation of Pores

The application of vacuum will not force the liquid alloy into the pores of the sample. Therefore, it is necessary to apply considerable pressure to the liquid alloy to force it into the fine pores of the sample.

1. Place the cup on a hot plate and add small bits of the Cerrobend alloy to bring the level of the molten alloy to the brim of the cup. When the cup is completely filled and the alloy temperature is above 150° C, place the cup in a basket and lower it into a laboratory pressure vessel (Figure 1).

2. Close the vessel and immediately raise the pressure to 15,000 psi, while the alloy in the cup is still molten. Hold the pressure at this level until it is certain that the alloy has cooled below its melting point. (This operation will require five to ten minutes in the cold oil of the vessel.)

(a) Supplied by Cerro Sales Corporation, 300 Park Avenue, New York, New York.

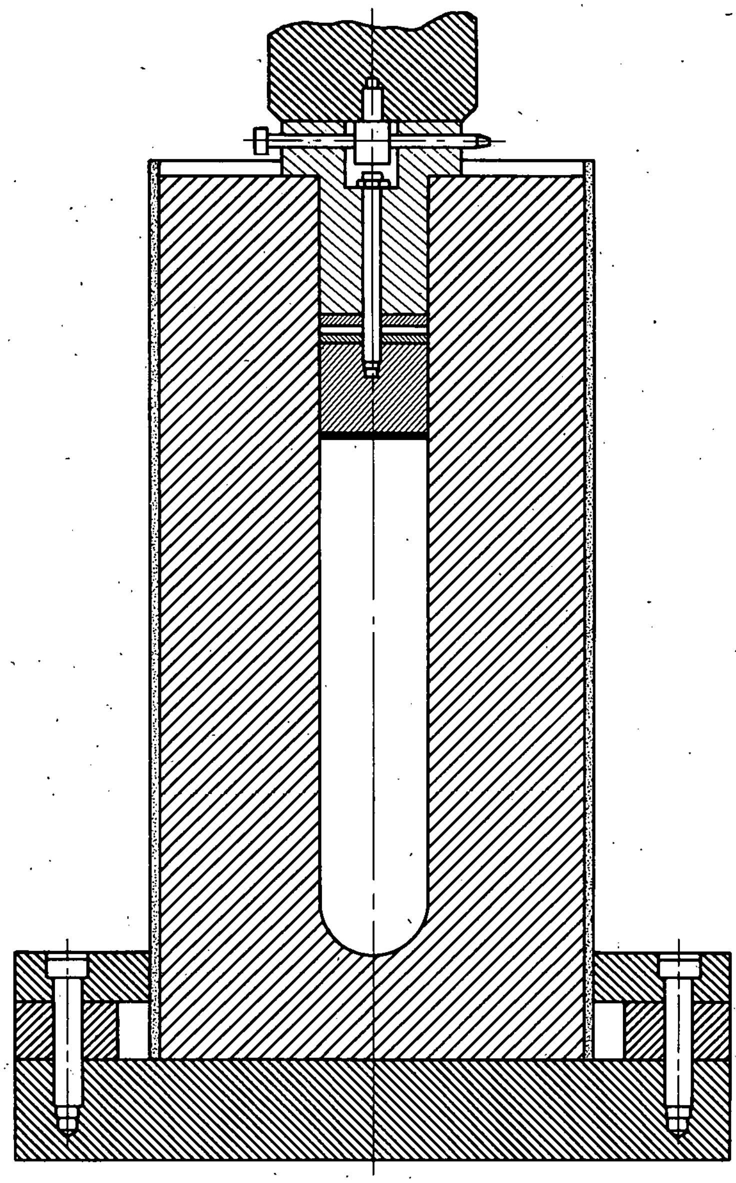


Figure 1. CROSS SECTION OF LABORATORY ISOSTATIC PRESSURE VESSEL.

3. Remove the cup from the pressure vessel and place it on a warm hot plate. As soon as the alloy melts, pour it from the cup, remove the sample from its epoxy glue mooring, and quench the sample in water or allow it to cool to room temperature on a steel plate. The pores of the sample now contain the solid alloy, and the sample can be mounted, ground, and polished.

Mounting

It has been found that an epoxy mounting medium is better for porous materials because no pressure is required as with other media; therefore, there is no danger of crushing the sample.

1. Use a hollow steel cylinder as a casting mold, which has as its inside dimensions those which are desired in the mount. Coat the inside of the cylinder with a mold release agent, such as MS 122 fluorcarbon[b] or a similar material. This step prevents the cured epoxy from sticking to the walls of the cylinder.

2. Place a piece of adhesive-coated sheet, such as Plain-Vu[c] (a self-laminating plastic sheet), across the bottom of the open cylinder. Grind the sample flat on the surface that is to be polished. The adhesive on the sheet enables the sample to be placed in any desired position, and will hold the sample in this position while the epoxy sets.

3. With the sample in place, pour the epoxy-mounting medium into the cup until the cup is full. After the epoxy has set, remove the mount from the mold and the plastic sheet from the mount. The mount is now ready to be ground and polished.

Grinding and Polishing

Porous materials have many properties which cause them to resemble ceramic materials; and, therefore, most of them can be ground and polished by methods similar to those used for ceramic materials.[1] No one method will be effective for all ceramic and ceramic-like materials.

1. Grind the mounted specimen using an Automet[d] head, starting with 240-grade paper, followed by 400-grade, and, finally, a 600-grade paper.

2. Rough polish the specimen using either an Automet or Whirlimet head[d], first with a 30-micron diamond disc with lapping oil, then with diamond paste with lapping oil. Use first the 15-micron diamond paste followed by 6-micron and then 1-micron paste, all on silk cloth or Texmet paper[d].

3. Place a final polish on the mounted specimen using a Vibromet polisher[d] with silk cloth, nylon cloth, or Texmet paper and α-alumina abrasive, starting with 1-micron alumina, followed by 0.3 micron, and, finally, 0.05 micron abrasive.

This method can be used for nearly any ceramic-like material by varying the time spent with each abrasive and by omitting those abrasives that do not contribute appreciably to the final finish or to a decrease in the time required to achieve the desired finish.

(b) Supplied by Miller-Stephenson Chemical Company, Los Angeles, California.
(c) Supplied by Elgin Label Company, Carr Adhesive Division, Cleveland, Ohio.
(d) Supplied by Buehler, Ltd., Evanston, Illinois.

DISCUSSION

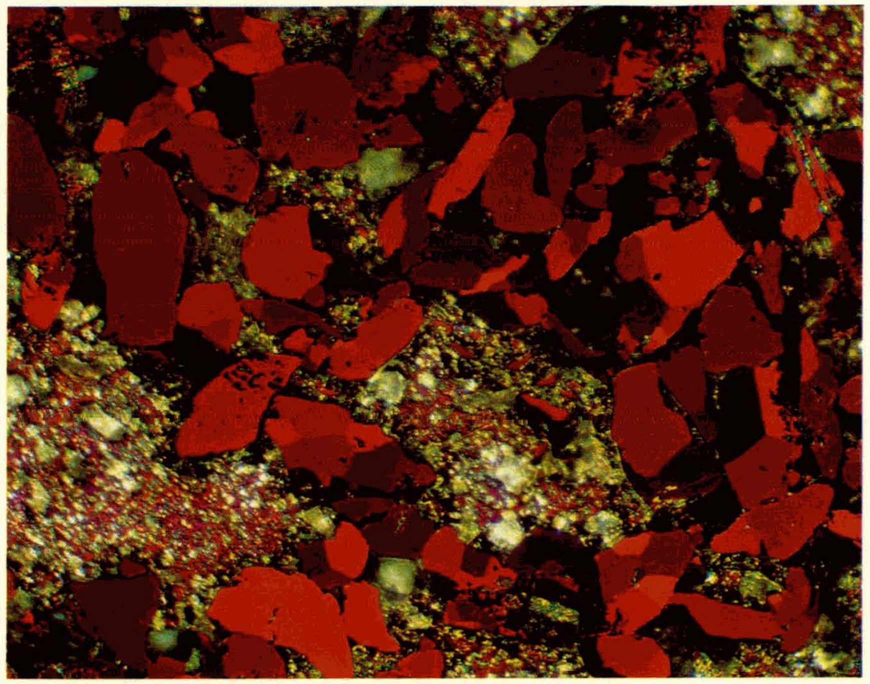
In the procedure for sample preparation for Cerrobend impregnation, the heavy-walled steel cup is used to insure having a high heat capacity, so that when filled with the molten alloy and placed in the pressure vessel, the heat present will keep the alloy molten while it is being forced into the pores under pressure. The sample is held to the bottom of the cup with epoxy cement to insure that it is completely immersed in the molten alloy during impregnation. During the pressure impregnation of pores, the cups filled with molten alloy are handled carefully during placement into the pressure vessel. It is not necessary to encapsulate them in a plastic sheath for pressing. The oil, pressing directly on the surface of the molten alloy, causes no difficulty. Elimination of the need for encapsulation saves a considerable amount of time.

For materials containing fine porosity, it is occasionally found desirable to heat the oil in the pressure vessel to about 150 - 160° C before inserting the sample basket. When hot oil is used, the alloy cools slower, producing larger grains which are more easily photographed and distinguished from the matrix material.

There are other Cerro alloys which have low melting points and might be used in place of the Cerrobend. Cerrotru alloy[a] (MP, 138° C) has been used and produces results very similar to the Cerrobend; but, since the Cerrobend has the lower melting point (70° C), it is more easily handled. There are also several other metals and alloys which have low melting points, but their use does not appear to offer any particular advantage. Several porous materials (beryllium, tungsten, carbon, fireclay crucible, and silica brick) were impregnated with Cerrobend alloy by the developed procedure. Photomicrographs of the final polished specimens are presented in Figures 2 (a) - (d) and 3 (a) - (d).

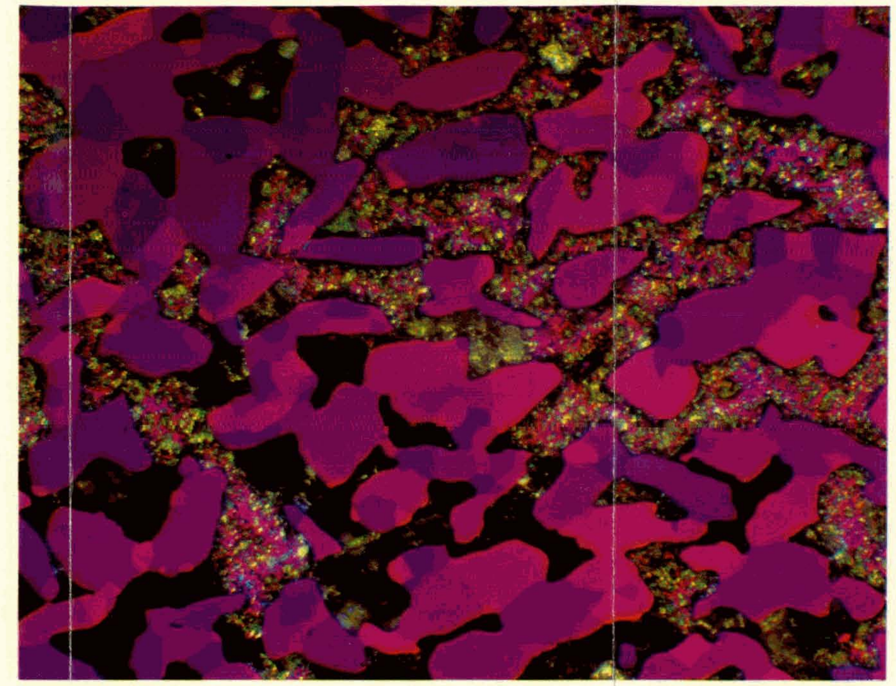
These photomicrographs, shown in color, were taken with a metallograph which had a rotatable 1/2 λ plate mounted in the vertical illuminating turret plate. This plate and its use has been described by Jackson and Calabra.[2] When the 1/2 λ plate is rotated, a vivid color variation in anisotropic materials is produced from the black and white of polarized light to the pale blue and pink of the sensitive tint. Exceptional color contrast is produced in many of the common materials encountered in metallography. The phases in the figures, which are some shade of blue or pink, are the matrix material; while, in every case, the dark olive-green phase is the Cerrobend alloy.

There are many short cuts which can improve and shorten the described procedure without altering the end result. However, the process that is outlined will achieve the desired impregnation of the sample which, when polished, will yield satisfactory photomicrographs showing: (1) the original porous structure, (2) any pullout by the absence of the low-melting alloy in the cavity, and (3) a natural pore by the fact that the alloy is in the pores.



CYN 1433

(a) Beryllium – 0.57 gm/cc, 69 Percent Porosity



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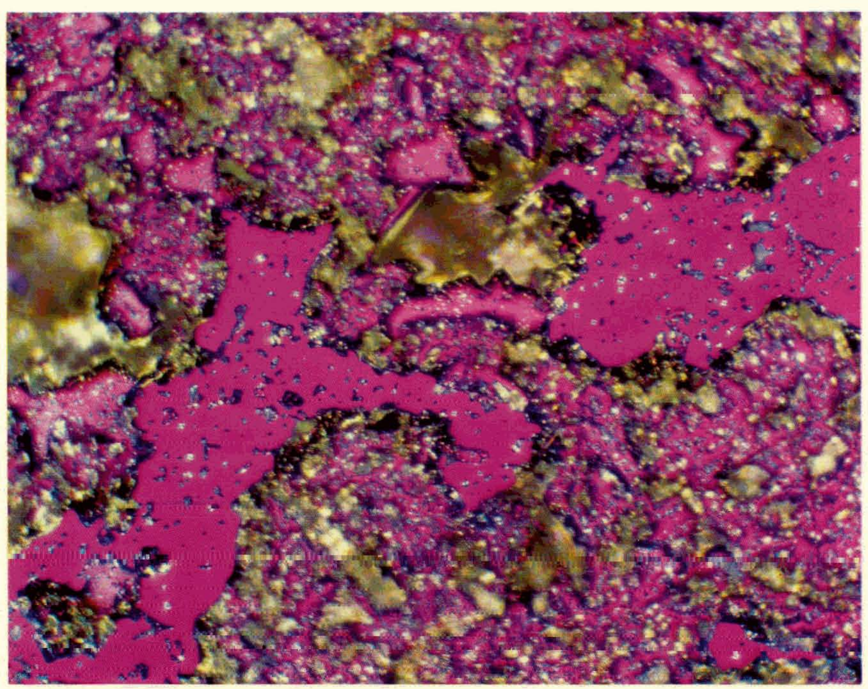
(b) Beryllium – 1.042 gms/cc, 44 Percent Porosity



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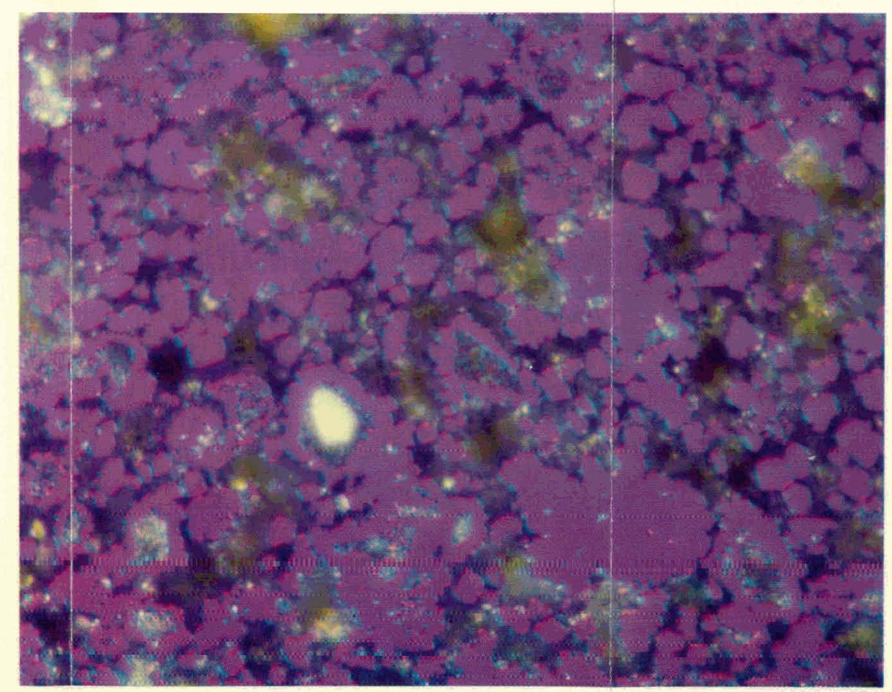
(c) Tungsten – 93 Percent Porous



CYN 1436

(d) Tungsten – 50 Percent Porous

Figure 2. MICROSTRUCTURE OF POROUS METALS IMPREGNATED WITH CERROBEND ALLOY. (Specimens Polished, Not Etched, Then Photographed in Sensitive Tint at 250X; Phases in Some Shades of Red or Purple are the Matrix; Cerrobend is a Shade of Olive Gray-Green)

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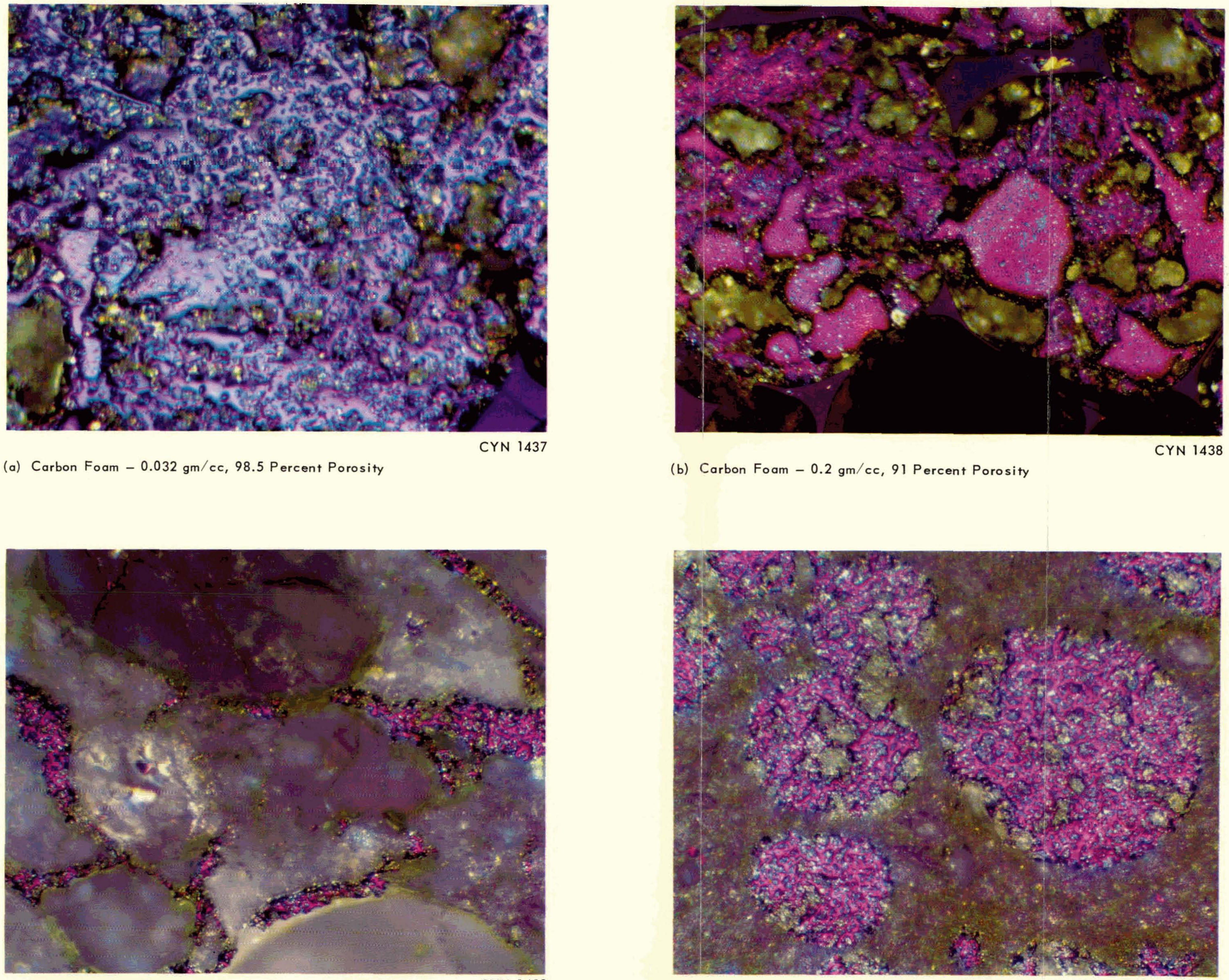

CYN 1437

(a) Carbon Foam – 0.032 gm/cc, 98.5 Percent Porosity

CYN 1438

(b) Carbon Foam – 0.2 gm/cc, 91 Percent Porosity

CYN 1439

(c) Fire-Clay Crucible Body – 2.124 gms/cc, 22 Percent Porosity

CYN 1440

(d) Silica-Fire Brick – 0.858 gm/cc, 40 Percent Porosity

Figure 3. MICROSTRUCTURE OF POROUS CERAMICS IMPREGNATED WITH CERROBEND ALLOY. (Specimens Polished, Not Etched, Then Photographed in Sensitive Tint at 250X; *Matrix is in Each Case Some Shade of Purple; Cerrobend is the Olive-Gray-Green Phase*)

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REFERENCES

(1) Procedure Guide for Ceramic Materials; Buehler, Ltd; Evanston, Illinois (1965).

(2) Jackson, R. J. and Calabra, A. E.; "Advances in Metallography", Technical Papers of the Twentieth Metallographic Conference, p 100; Dow Chemical Company-Rocky Flats Division; Golden, Colorado (1966).