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# **Epoxy Impregnation Procedure for Hardened Cement Samples**

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U.S. DEPARTMENT OF COMMERCE  
National Bureau of Standards  
National Engineering Laboratory  
Center for Building Technology  
Gaithersburg, MD 20899

May 1988

Progress Report



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Research Information Center  
National Institute of Standards  
and Technology  
Gaithersburg, Maryland 20899



## ABSTRACT

A method was developed previously for epoxy impregnation of hydrated cementitious materials for microscopical examination without drying the samples. The impregnation was achieved by sequentially replacing pore solution with ethanol, then the ethanol with epoxy. During subsequent application of the procedure, many specimens were found to be either incompletely impregnated or inadequately cured. Studies were carried out to identify the cause of these problems and to modify the procedure for more reliable impregnation. Contamination with low levels (4 percent) of water or ethanol was found to prevent proper curing. Modifications in the procedure to prevent contamination, including monitoring the replacement of pore solution by ethanol, were shown to provide consistent and reliable impregnation.

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## INTRODUCTION

Hydrated cementitious materials are highly porous, and some or all of the pores are typically filled with an aqueous solution. When preparing a polished section of such a porous material for examination by scanning electron microscope (SEM) or optical microscope, it is often necessary to impregnate first with epoxy, to provide physical support during sectioning and polishing. Epoxy also facilitates the identification of voids and pores using SEM with backscattered electron imaging (BEI), since the low atomic number of epoxy provides contrast with the other constituents of hydrated cement.

In a previous report, Struble and Byrd [1] studied methods of drying and impregnating hydrated cement. For epoxy impregnation, the aqueous solution in the pores must be removed by drying, which causes considerable shrinkage and cracking. Specimens dried and vacuum-impregnated with epoxy were found to contain microcracks regardless of the drying method (vacuum drying, critical point drying, or freeze drying). A method was described in which pore solution was replaced with alcohol and then the alcohol replaced with epoxy without drying the specimen. This method produced well-impregnated samples that were, in general, free of microcracks.

During subsequent application of the sequential replacement procedure to large numbers of specimens, many were found to be inadequately cured, especially around the hardened cement. It appeared from these observations that a contaminant from the specimen was interfering with curing of the epoxy. One impregnated specimen was sectioned perpendicular to the wafer and examined using SEM. Pores and voids were filled with epoxy only in the outer ~100  $\mu\text{m}$  of paste, and the interior appeared to be free of epoxy, suggesting that the specimen was only partially impregnated.

Studies were carried out to determine the causes for these problems, and to modify the procedure so specimens could be reliably impregnated without microcracks for microscopical examination. Incomplete replacement of pore solution by ethanol or of ethanol by epoxy was studied as the most likely cause both of inadequate impregnation and of inadequate curing if epoxy were contaminated with water or ethanol. Therefore, methods were explored to directly monitor each replacement step, and the diffusion rate of ethanol was studied. Based on these studies, modifications in the impregnation procedure were developed, and the performance and reliability of the modified impregnation procedure were assessed.

## EXPERIMENTAL APPROACH

The procedure described by Struble and Byrd [1] allowed impregnation of 1-mm thick specimens of hydrated cement paste by ultra-low viscosity (ULV) epoxy (described below). The procedure, similar to methods used in biological studies [2], consists of the following sequence:

- 1) immerse the specimen in ethanol for 4 days;
- 2) immerse in a 50:50 mixture of ethanol and ULV epoxy for 24 hours;
- 3) immerse in the ULV epoxy for 24 hours; and
- 4) cure by heating at 60°C for 2 days.

The sensitivity of epoxy hardening to contamination by water or ethanol and to varying proportions of the individual components was assessed. Because hardening was found to be sensitive to both water and ethanol, a procedure was devised to monitor directly the replacement of water by ethanol. Unfortunately, though no such procedure could be devised for replacement of ethanol by epoxy. Based on these studies, a modified impregnation procedure was developed to provide full impregnation of epoxy and proper, reliable curing. The modified procedure includes a direct measure of ethanol impregnation. The modified procedure was utilized for samples of either normal or reduced permeability (water-cement ratio) to assess the performance and reliability of the procedure.

### Materials

Samples for impregnation were prepared using cement and water, with a few samples including a superplasticizer to provide reduced water-cement ratio. The cement was a Type I portland cement, Cement No. 86 in the Cement and Concrete Reference Laboratory program at National Bureau of Standards. Its chemical composition is shown in Table 1. Sulfonated melamine formaldehyde superplasticizer<sup>1,2</sup> was used in preparing a few samples.

Reagent-grade, 200 proof, ethanol was used to replace pore solution in hardened cement samples. Because water was expected to affect hardening of the epoxy, the ethanol was dried using a molecular sieve<sup>3</sup>, a technique used previously [3] to provide water-free methanol.

The ultra-low viscosity (ULV) epoxy, described by Mascorro et al. [2], is available from various suppliers of electron microscopy accessories<sup>4</sup>. It was selected for its very low viscosity and its miscibility with

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<sup>1</sup>This and other trade names and company products are identified to specify adequately the experimental procedure. In no case does such identification imply recommendation or endorsement by the National Bureau of Standards, nor does it imply that the products are necessarily the best available for the purpose.

<sup>2</sup>Melment, obtained from SKW, Atlanta, GA.

<sup>3</sup>Linde 3A, Union Carbide Corp., South Plainfield, NJ.

<sup>4</sup>For example, ultra-low viscosity embedding medium obtained from Polysciences Inc., Warrington, PA.



Table 1. Chemical composition of cement [4].

Constituent	(%)
SiO <sub>2</sub>	0.44
Al <sub>2</sub> O <sub>3</sub>	5.11
Fe <sub>2</sub> O <sub>3</sub>	1.88
CaO	62.19
MgO	3.68
SO <sub>3</sub>	3.31
Na <sub>2</sub> O	0.25
K <sub>2</sub> O	0.99
Loss on Ignition	1.8

alcohol. The dye is Oil Red EGN<sup>5</sup>, a proprietary dye miscible in alcohol, ULV epoxy, and other epoxies. Details on the components and mix ratios are provided in Table 2.

Potential hazards exist in handling the epoxy components, and safety data sheets should be obtained from the manufacturer concerning the ultra-low viscosity components. Materials should be stored and handled only in a well-ventilated fume hood, and gloves should be worn during mixing.

Materials for grinding and polishing impregnated specimens included reagent-grade propylene glycol, SiC grinding papers, and synthetic diamond paste.

### Methods

The normal cement paste was prepared using deionized water and portland cement at a water-cement ratio of 0.45. These were mixed as follows in a blender<sup>6</sup> operated at low speed and cooled using tap water: mixed for 1 min, allowed to rest for 2 mins while scraping the sides of the blender, and mixed for 1 min. Pastes of lower water-cement ratio containing superplasticizer were mixed using a somewhat different method based on the method used by M. Brown in our laboratory for preparing high-strength concrete. Cement and water, at a water-cement ratio 0.22 to 0.35, were first mixed using a mortar and pestle, scraping the sides of the mortar often with a spatula; half the superplasticizer was added after 4 min and the remainder after 6 min, continuing to mix using the pestle for a total of 13 min; the paste was transferred to the blender and mixed for an additional 10 min at low speed, stopping every 2 min to scrape the sides and allow the mix to cool.

Paste samples were cast in 10-mm cube molds, which were vibrated for a few seconds to consolidate the paste; the molds were covered with a wet

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<sup>5</sup>Obtained from Polysciences Inc., Warrington, PA.

<sup>6</sup>Commercial blender, model CB-6, Waring, New Hartford, CT.

Table 2. Ultra-low viscosity epoxy components.

Component	Standard Mix [2] (by weight)
Vinylcyclohexene dioxide (VCD, Epoxy resin)	0.5 part
n-Hexenyl succinic anhydride (HXSA, Hardener)	1.0 part
1,4-butanediol diglycidyl ether (Araldite RD-2, Modifier)	0.075 part
Dimethylamioethanol (DMAE, Catalyst)	0.028 part (1.0 percent by volume)
Dye	-0.1 part (until color intensity is satisfactory)

cloth and sealed in polyethylene, and stored at room temperature (~24°C) for 24 hours. Samples were then demolded and immersed in a saturated Ca(OH)<sub>2</sub> solution for curing at room temperature. After curing in the Ca(OH)<sub>2</sub> solution, cubes were removed from solution and specimens prepared for impregnation. A slice was cut for impregnation using a low speed, diamond-blade wafering saw<sup>7</sup>, with the blade and specimen lubricated by saturated Ca(OH)<sub>2</sub> solution; approximately 1 mm of material was removed to expose a fresh face, then a 1-mm thick wafer was cut for epoxy impregnation.

The pore solution in the hydrated cement was replaced by epoxy in a sequential two-stage replacement. Specimens were immersed in ethanol then in epoxy until replacement was considered to be complete (discussed below). The intermediate immersion in a 50:50 mixture of ethanol and epoxy used in the previous procedure [1] was eliminated with no effect on the impregnation process.

The replacement of pore solution by ethanol was monitored using a dye soluble in ethanol and insoluble in water (described below). Penetration of this dye was monitored in a companion specimen immersed in ethanol containing the dye. Every 48 hours the monitor cube was cut to expose the interior, which was examined visually to assess the extent to which the dye and alcohol had penetrated. Because the dye is soluble in ethanol and insoluble in water, the extent of penetration by the dye can easily be visually assessed. Using this method, it was found that full replacement of pore solution by ethanol in a 1-mm specimen required approximately 8 days, considerably more than the 4 days found in the previous study [1].

Elevating the temperature to 60°C during replacement of pore solution by ethanol was explored as a method to reduce the time required for the

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<sup>7</sup>Isomet saw, Buehler, Ltd., Lake Bluff, IL.

diffusion process, whose rate is expected to depend on temperature. It is recognized that heating the specimens may cause dehydration of hydrated cement phases and thereby affect microstructure. The epoxy-impregnated specimens must be heated to cure the epoxy, and in the previous study the 60°C curing temperature was selected as a temperature low enough to minimize any microstructural effects while high enough to allow the epoxy to cure within 48 hours. Therefore, it was not considered harmful to heat the specimens at the same temperature while immersed in ethanol.

Epoxy components were proportioned by weight, dispensed using a disposable plastic pipette in the proportions listed in Table 1. The epoxy was stirred by hand until it appeared that the dye had dissolved and was homogeneously blended.

No direct measure of epoxy diffusion could be devised for these studies. Epoxy diffusion could not be monitored using the dye because the dye is soluble in both the epoxy and ethanol. Changes in weight and density as the epoxy replaces the alcohol were found to be inconclusive.

The epoxy replacement step consisted of immersing the ethanol-saturated wafers in the ULV epoxy. The immersed specimen was stored at -2°C to facilitate diffusion of the epoxy, because the epoxy exhibits a marked increase in viscosity in 24 hours at room temperature [2] and storage at 2°C was considered to prevent this increase. In addition, the epoxy was replaced every 48 hours with a fresh mixture to assure that the epoxy in contact with the sample is low in viscosity.

Finally, specimens were immersed in fresh epoxy, prepared as described above, and cured at 60°C for 24 to 48 hours. After curing the specimens were stored under a vacuum or over a desiccant, because exposure to moisture causes the epoxy to soften.

Impregnated specimens were ground and polished for SEM examination as follows: ground using 240 grit and 400 grit SiC with no lubricant, followed by 600 grit SiC using propylene glycol as a grinding lubricant, then polished on a glass plate using a sequence of diamond pastes (6 μm, 3 μm, 1 μm and 0.25 μm) lubricated using propylene glycol. Microstructural examination was carried out using SEM/BEI.

Tests were conducted to examine the tolerance of the epoxy to added ethanol or water. In addition, tests were performed to assess the curing with variation in the amount of each of the four epoxy components; low levels of ethanol were also added to these mixtures. To test the effect of ethanol, an epoxy mixture (Table 2) was divided into four parts, to which were added 0, 10, 20 or 50 percent (by weight) ethanol. To test the effect of water, a mixture was divided into five parts, to which were added 0, 2, 4, 6 or 8 percent (by weight) water. To test the effect of slight variation in the proportions of each component, mixtures were prepared in which each of the four components was added at 90 percent or 110 percent of its correct proportion. To each of these latter mixtures was added 0, 2, or 4 percent (by weight) ethanol. Each mixture was cured at 60°C and examined visually to assess its hardness.



A modified impregnation procedure, itself the result of studies described here, was used to prepare specimens of normal and low permeability to assess the performance and reliability of the procedure. The procedure, summarized in Table 3, was followed to prepare specimens 1 mm in thickness for examination using SEM/BEI. The specimens were immersed in ethanol at 60°C until examination of the separate monitor cube indicated that ethanol (containing dye) had penetrated >0.5 mm on each face, a period of approximately 6 to 14 days. (When the monitor cube exhibits a 0.5-mm band of color, it is assumed that the wafers, 1 mm thick, are fully replaced with ethanol.) Specimens were then removed from ethanol, immersed in epoxy, and stored at -2°C for the same length of time required for the ethanol replacement, replacing epoxy with a fresh mixture every 48 hours. Finally, specimens were cured, ground, and polished as described above.

## RESULTS AND DISCUSSION

Results concerning the epoxy sensitivity to contaminants and to mix proportions are summarized in Table 4. Curing was suppressed by ethanol addition in excess of 4 percent, and at 2 or 4 percent addition when the proportions of some epoxy constituents were reduced slightly. Curing was not suppressed by water at a 2 percent addition, but was suppressed at 4 percent or greater. After twelve days, the samples containing 4 percent or more water had cured everywhere but the exposed surface, and when demolded they shattered.

Epoxy prepared by varying each of the 4 components cured normally in 24 hours. The most pronounced change on varying proportions was in the color of the cured epoxy, from a control color of slight brown tint to altered blend colors of amber and dark brown.

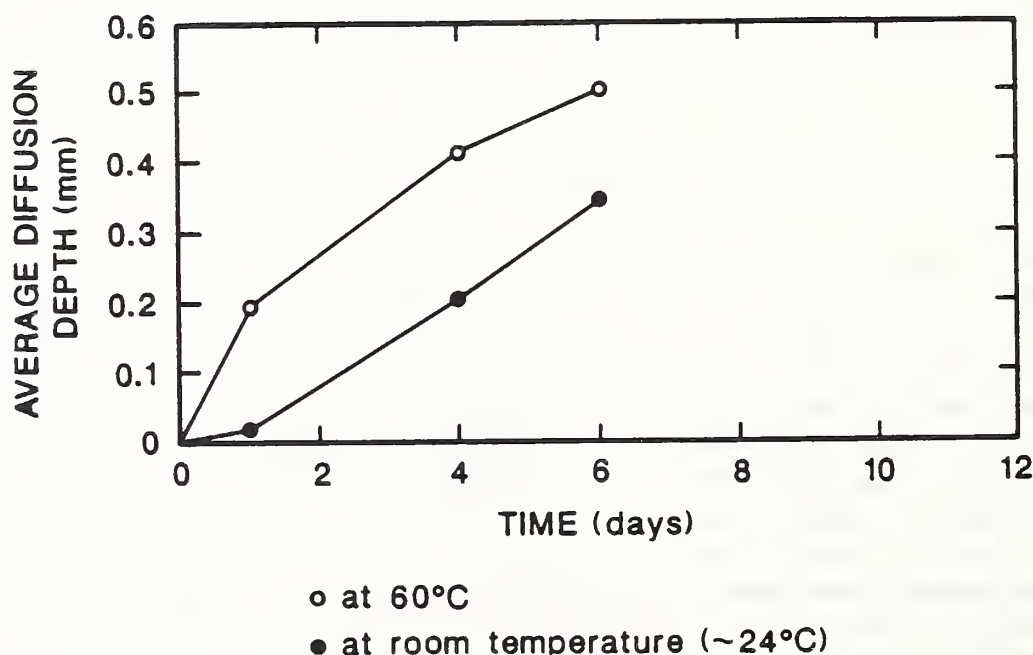


Figure 1. Depth of penetration of dye in hardened cement paste immersed in ethanol.

Table 3. Modified impregnation procedure.

Step	Description
1	Immerse specimen in water-free ethanol at 60°C
2	Immerse monitor cube in water-free ethanol containing dye at 60°C
3	Examine monitor cube until dye indicates that ethanol has penetrated at least 0.5 mm on each face
4	Immerse specimen in epoxy and stored at -2°C for the same length of time required for the ethanol replacement, replacing epoxy with a fresh mixture every 48 hours
5	Immerse specimen in fresh epoxy and cure epoxy by heating at least 24 hours at 60°C
6	Grind and polish for microscopical examination



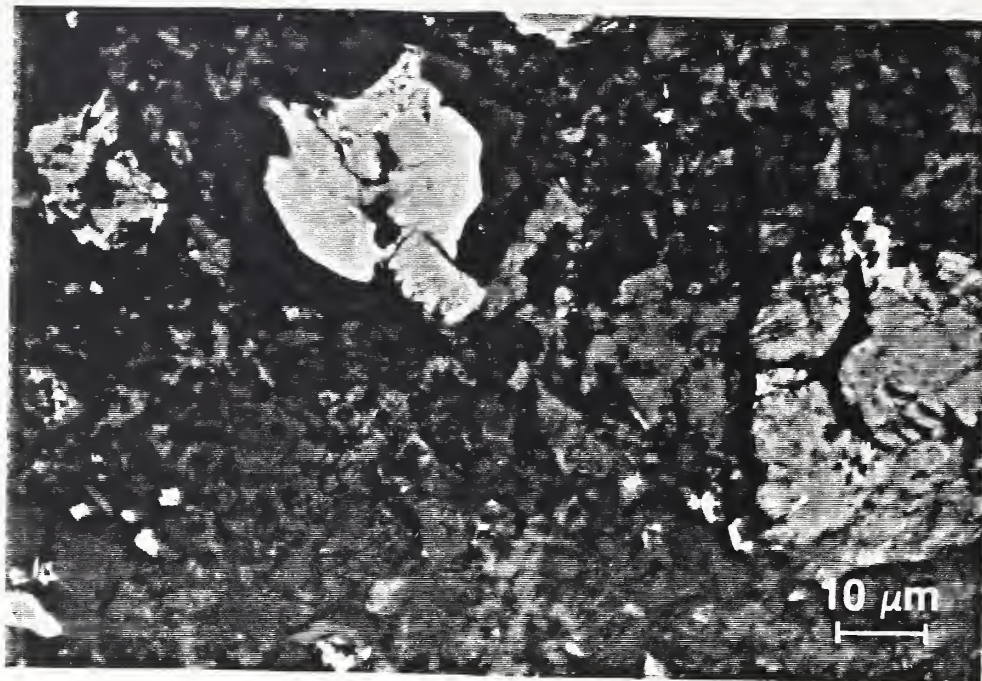
Table 4. Curing of epoxy contaminated by ethanol or water.

Mixture	Curing time	Condition
Control (Table 2)	3 days	hard (normal)
Control + 10% ethanol	3 to 11 days	semi-solid, very viscous
Control + 20% ethanol	3 to 11 days	liquid, viscous
Control + 50% ethanol	3 to 11 days	liquid, very fluid
Control (Table 2)	1 day	hard (normal)
Control + 2% water	1 day	hard (normal)
Control + 4% water	1 day	semi-solid, very viscous
	12 days	hard, soft on exposed surface
Control + 6% water	1 day	semi-solid, very viscous
	12 days	solid, soft on exposed surface
Control + 8% water	1 day	semi-solid, very viscous
	12 days	hard, soft on exposed surface
Control + 2% ethanol	1 day	hard (normal)
Control + 4% ethanol	1 day	hard (normal)
90% VCD	1 day	hard (normal)
110% VCD	1 day	hard (normal)
90% HXSA	1 day	hard (normal)
110% HXSA	1 day	hard (normal)
90% ARD-2	1 day	hard (normal)
110% ARD-2	1 day	hard (normal)
90% DMAE	1 day	hard (normal)
110% DMAE	1 day	hard (normal)
90% VCD + 2% ethanol	1 day	solid, not hard
90% VCD + 4% ethanol	1 day	solid, not hard
110% VCD + 2% ethanol	1 day	hard (normal)
110% VCD + 4% ethanol	1 day	hard (normal)
90% HXSA + 2% ethanol	1 day	hard (normal)
90% HXSA + 4% ethanol	1 day	solid, not hard
110% HXSA + 2% ethanol	1 day	hard (normal)
110% HXSA + 4% ethanol	1 day	hard (normal)
90% ARD-2 + 2% ethanol	1 day	hard (normal)
90% ARD-2 + 4% ethanol	1 day	solid, not hard
110% ARD-2 + 2% ethanol	1 day	hard (normal)
110% ARD-2 + 4% ethanol	1 day	hard (normal)
90% DMAE + 2% ethanol	1 day	hard (normal)
90% DMAE + 4% ethanol	1 day	solid, not hard
110% DMAE + 2% ethanol	1 day	hard (normal)
110% DMAE + 4% ethanol	1 day	hard (normal)

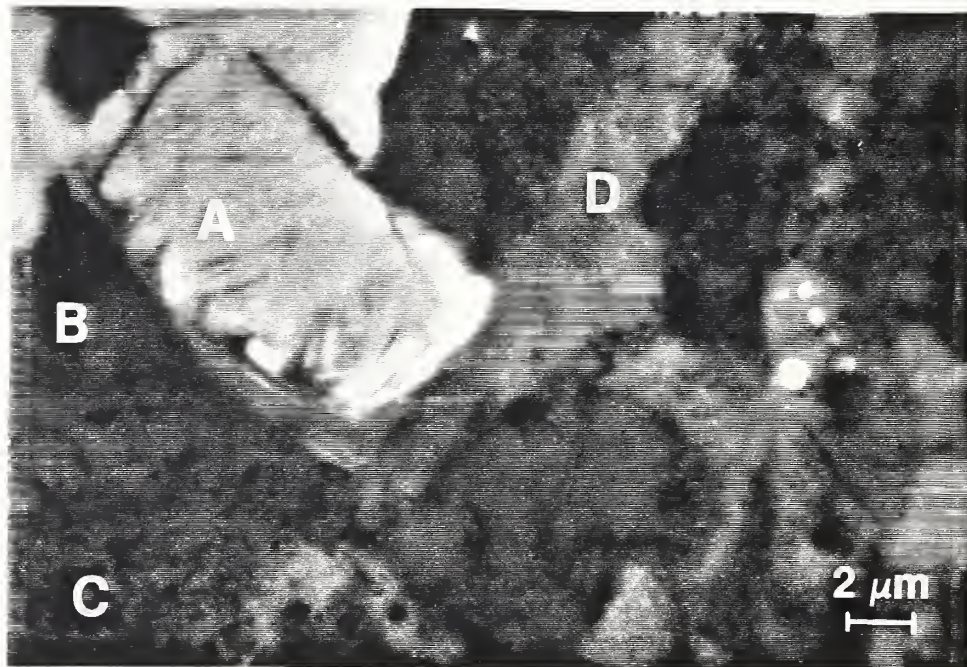
The extent of ethanol diffusion into hardened cement paste was measured both at room temperature and at 60°C (Fig. 1). After the first day the diffusion rates at the two temperatures were similar, but the extent of diffusion was greater at the higher temperature. Based on these results, specimens were subsequently stored in a 60°C oven while immersed in ethanol to facilitate the diffusion process.

Approximately 20 specimens, both normal and low water-cement ratio, have been impregnated using the modified procedure summarized in Table 3, and all specimens have cured normally. An example of normal water-cement ratio paste is shown in Fig. 2, and an example of low water-cement paste with superplasticizer is shown in Fig. 3. Both specimens appeared to be fully impregnated and free of microcracks. Various features of the paste microstructure may be identified by their contrast in the backscattered electron image. The unhydrated clinker grains appear as the lightest constituent. Calcium hydroxide is light gray and calcium silicate hydrate gel is slightly darker. The darkest constituent is epoxy. Of particular interest in both examples is the distinction between the relatively dense inner hydration product and the more porous outer hydration product, in which the porous gel appears to be impregnated with epoxy.





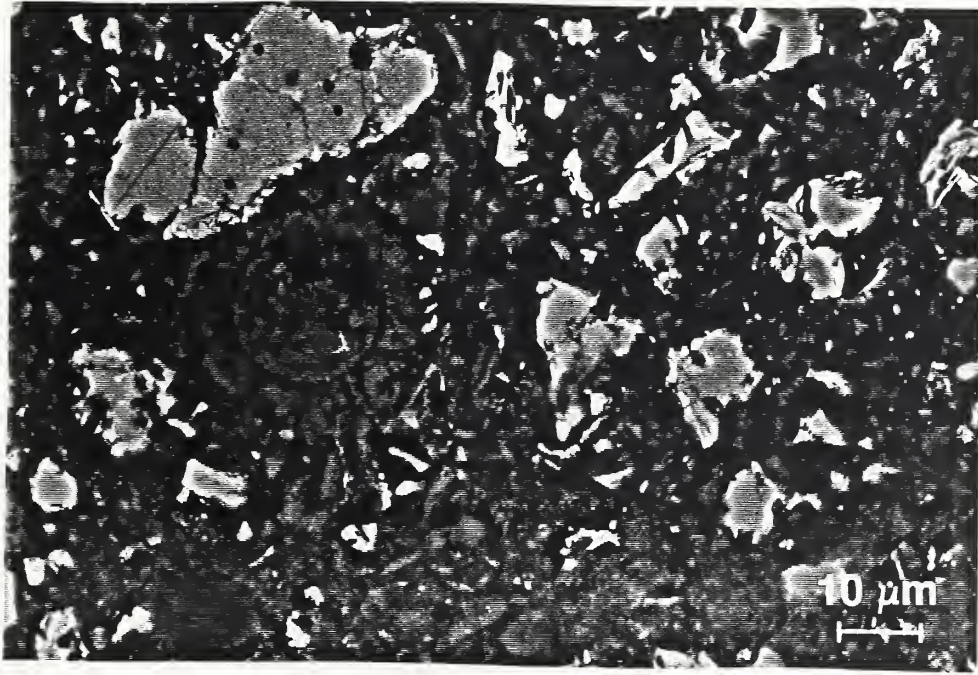
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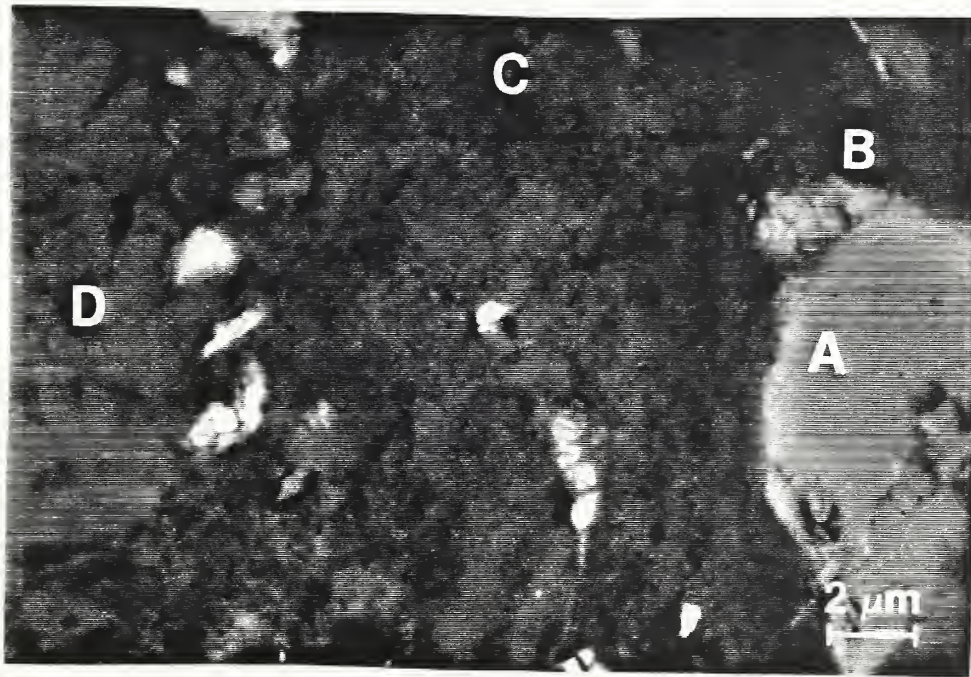
(b)

Figure 2. SEM/BEI micrograph of hardened cement paste, 0.45 water-cement ratio, cured for 6 weeks; images at low magnification (a) and at high magnification (b) showing unhydrated clinker (A), dense inner hydration product (B), porous outer hydration product (C), and calcium hydroxide (D).





(a)



(b)

Figure 3. SEM/BEI micrograph of hardened cement paste, 0.25 water-cement ratio (containing superplasticizer) cured for 4 weeks; images at low magnification (a) and at high magnification (b) showing unhydrated clinker (A), dense inner hydration product (B), porous outer hydration product (C), and calcium hydroxide (D).

## CONCLUSIONS

The ultra-low viscosity epoxy was found to be tolerant of low levels (2%) of ethanol and water, but did not cure properly when contaminated by higher levels. Varying the proportion of each component by 10 percent did not appear to affect curing.

The two step process of replacing pore solution with ethanol and ethanol with ULV epoxy, assuring full replacement of pore solution by ethanol and of ethanol by epoxy, was found to provide reliable impregnation of hardened cement paste samples, both normal and low water-cement ratio. Measuring the extent of penetration of ethanol using a monitor cube immersed in ethanol containing a dye provides assurance that ethanol has fully replaced the pore solution, and provides an indication of the time required for epoxy replacement of ethanol.

After curing, epoxy serves to fill existing cracks and pores and provide support during subsequent grinding and polishing. As described previously [1], the impregnation process allows preparation of polished samples of hardened cement paste with little or no microcracking, and the epoxy provides contrast when examining voids and pores using SEM/BEI.

## ACKNOWLEDGEMENTS

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<b>10. SUPPLEMENTARY NOTES</b>  <input type="checkbox"/> Document describes a computer program; SF-185, FIPS Software Summary, is attached.			
<b>11. ABSTRACT</b> <i>(A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here)</i> <p>A method was developed previously for epoxy impregnation of hydrated cementitious materials for microscopical examination without drying the samples, by sequentially replacing pore solution with ethanol, then the ethanol with epoxy. During subsequent application of the procedure, many specimens were cured. Studies were carried out to identify the cause of these problems and to modify the procedure for more reliable impregnation. Contamination with low levels (4 percent) of water or ethanol was found to prevent proper curing. Modifications in the procedure to prevent contamination, including monitoring the replacement of pore solution by ethanol, were shown to provide consistent and reliable impregnation.</p>			
<b>12. KEY WORDS</b> <i>(Six to twelve entries; alphabetical order; capitalize only proper names; and separate key words by semicolons)</i> cement, epoxy, impregnation, microcracks, microscopy, scanning electron microscopy, ultra-low viscosity epoxy			
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