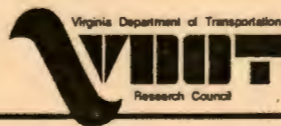


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PETROGRAPHIC MANUAL: PETROGRAPHIC METHODS OF EXAMINING HARDENED CONCRETE



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Research Petrographer



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<p>16. Abstract</p> <p>This manual was undertaken to record for all persons wishing to do concrete petrography the petrographic procedures that have been found useful here at the Virginia Transportation Research Council.</p> <p>This interim report is made up of the introduction, reading list, sections on sample preparation, and suggestions for further research. The final manual will also include sections on selection and collection of specimens, specimen preparation, use of various microscopes, aggregate, determination of the air void parameters, shrinkage cracking, water/cement ratio, retempering, and alkali-aggregate reactions.</p>			
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Including
Introduction, Sample Preparation,
Questions and Suggestions,
and Reading List

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(The opinions, findings, and conclusions expressed in this report are those of the author and not necessarily those of the sponsoring agencies.)

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INTRODUCTION

The methods of examining hydraulic cement concrete (HCC) and related concretes are akin to the methods used by the petrologist and mineralogist for examining naturally occurring earth materials. The word *petrography* was derived from the Greek: *petros*, which means "rock" or "stone," and *graphikos*, which means "written." *Petrography* has come to mean the description and classification of rock by any means, from a simple description of color or form to the use of highly technical chemical methods and instrumental methods, such as scanning electron microscopy, fluorescent X-ray analysis, and determination of the molecular structure of minerals by X-ray diffraction. Petrography is a branch of the science of petrology, which includes, in addition to description and classification, the deciphering of the origin of rocks, the study of the relationships between various rock and mineral deposits, the effect of various geologic processes, and the unraveling of the complex history of rocks.

Most of the persons presently doing concrete petrography were originally geologists with formal training in optical mineralogy and petrography. There is an abundance of literature available to specialists in the geologic fields. Many universities have large departments devoted to the education of petrologists and mineralogists. Many of the techniques used are also related to the sciences generally called "materials engineering." There is a multitude of universities with an excellent department of materials engineering, and entire libraries are devoted to these subjects. However, the techniques and skills required for the examination of HCC do not seem to be the subject of serious study at any schools of higher education.

The primary written works on the petrography of concrete are the applicable publications of the Transportation Research Board (TRB) (formerly the Highway

Research Board [HRB]), the American Society for Testing and Materials (ASTM),¹⁻³ the American Concrete Institute (ACI),⁴ the Construction Technology Laboratories (CTL) (formerly the Portland Cement Association [PCA]), and the National Research Council, Canada.⁵ Some of these works have detailed instructions for petrographic examination of concrete or aggregates for concrete.¹⁻⁴ Many mention that the work should be done by persons who are qualified by training and experience to operate the microscopes and other equipment used; record all the important information; recognize which data will have a bearing on any problems associated with the specimens or on the intended use of the material in question; and interpret the observations and record them in a form understandable by the person or persons who will be using the petrographic information.³

The petrography of HCC and of the aggregates of HCC is at once simpler and more complex than classical petrography. A few of the works on classical petrography are listed in the Reading List. It is simpler because mineral species that are not abundant are of little importance. It is more complex because the history of the concrete mixture (both the proportioning and workmanship) and the structural and chemical features of the concrete paste, itself, are subject to study and the physical and chemical reactions of the rocks and minerals in both fresh and hardened concrete must be determined.

The petrography of concrete must often be accomplished rapidly, on a limited budget, and with a minimum of the sorts of equipment required for instrumental analysis of the chemical components and the various physical forces existing between the molecules and granules of the substance. Because the experienced concrete petrographer has been able, in a limited amount of time, to make a useful diagnosis of the quality and possible problems associated with HCC, the associated basic science studies that would use instrumental analysis have almost never been done in the course of solving construction problems.

The petrographic examination of HCC is in many respects more qualitative than quantitative. The quantitative determination of the parameters of the air void system, the macroscopic appearance of specimens of HCC, the appearance of the concrete in the placement, and the appearance of variously prepared surfaces of the concrete as viewed with the stereoscopic microscope are the most used tools of the petrographer. Even the examination of carefully prepared thin sections of HCC with the petrographic microscope is usually a qualitative procedure, with the examination taking the form of ascertaining the presence or absence of particular features and the relative abundance of a few others.

There is no large collection of reference works on the examination of HCC and no collections of pictures of the features indicating durable concrete or of the ominous signs that indicate probable early failure and/or deterioration. The references on the petrography of concrete are few and lack good photographs that can be compared with the concrete an inexperienced microscopist might be viewing.

After many years of experience in examining concrete, the concrete petrographer has seen hundreds of pieces of concrete and his or her memory associates par-

ticular appearances of the concrete with the histories of durability or failure that have accompanied these specimens. Thus, the memory of the concrete petrographer is the data bank with which all new specimens of concrete are compared.

It is very difficult to transmit this sort of data bank to a new person who, although trained in the techniques of optical mineralogy or materials science, has had little or no experience in examining HCC and determining the various features that may indicate the quality of the material.

Access to laboratory-produced specimens of HCC and the mixture design by which they were fabricated can prove very useful to the petrographer. It will allow an excellent opportunity for the study of specimens of HCC produced with various experimental materials, a large variety of types of aggregate, and numerous different admixtures so that the variations between specimens can be correlated with known differences in the design of the mixture. Various curing methods under various conditions and at various degrees of maturity can also be studied. The results of the petrographic observations can be compared with the data obtained in the laboratory. The data available should include the exact nature and source of the ingredients, the proportions of the mixture, and the following:

- slump
- unit weight
- air content when fresh
- curing method
- maturity
- compressive strength
- permeability
- results of freeze-thaw testing
- results of any other testing.

Such studies are really research projects but must often be undertaken before a truly informed opinion can be made of the quality, cause for particular features, or reason for failure of HCC from construction sites.

Construction problems are rush problems and cannot wait for the results of long laboratory procedures. Waiting until a laboratory mixture is prepared, cured, and tested so that the resulting concrete can be compared with the concrete at a particular problem site is not often possible. In any case, it is a known fact that it is difficult to duplicate bad concrete in the laboratory. Features that are due to poor workmanship or incomplete mixing are especially difficult. This may be partially due to the difference in size between a ready-mix truck and a laboratory mixer and partially due to the natural reluctance of trained concrete technicians to violate normal procedures. Particular difficulty may be found when trying to duplicate problems that have been caused by field alteration of the mixture. All too commonly,

water has been added to the mixture after the concrete has begun to set and after the air content determinations have been made, and this retempering with water has not been documented and must be inferred from the parameters of the concrete.^{1, 2}

SAMPLE PREPARATION

Surfaces for Stereomicroscopic Examination

Because it is impossible to focus clearly and view a rough surface at the magnifications required for the examination of HCC with a stereomicroscope for general petrographic examination or analysis of an air void system, it is necessary that smooth (finely lapped) surfaces be prepared on sawed slices of the specimens. The smoother the saw cut, the easier will be the required lapping procedures.

A slice of the HCC should be cut from the specimen submitted. All fragments of the original specimen should be labeled to identify their origin and, whenever possible, their orientation. A slice approximately 7/8 inch thick is generally convenient. This thickness will generally be strong enough to withstand normal laboratory wear and handling but be thin enough to permit a comfortably seated microscopist to focus the microscope easily without stretching his or her neck or being cramped into the table or bench. If the HCC is badly deteriorated, has an extremely high air content, or is otherwise fragile, it may be necessary to use a somewhat thicker slice.

The slice must usually be trimmed to fit the slice holder of the lapping device. (Most holders are circular to allow free rotation.) Care must be taken not to trim away any portion of the area of greatest interest to the client requesting the petrographic examination. Often, the portion of greatest interest will be the top, or wearing, surface. In such cases, any trimming will have to occur on the bottom. If there are no obvious differences to be seen between the top and the bottom of the cores and the section does not exceed 6 inches in any dimension, a lapped section can be produced that will be representative of the full depth of the specimen. Very often, the lowest inch or so does not require examination unless it is very different from the upper portions.

On occasion, it may be necessary to determine the individual air void systems at various depths. When this is the case, the cutting plan will include taking horizontal slices at various depths and preparing each slice individually. Often, facing surfaces separated by only the thickness of the saw cut are considered as one specimen.

Apparatus and Materials

Apparatus and materials for the preparation of surfaces of HCC samples for microscopical observation are described in ASTM C856³ under "Apparatus." Other apparatus may be equally suitable. The polishing wheel (mentioned in ASTM C856) is not required for the production of finely lapped concrete surfaces because this preparation method does not require polished surfaces. Polished surfaces are often less flat than finely lapped surfaces. Polishing of concrete surfaces frequently causes "undercutting." Undercutting is the process in which softer constituents are removed more rapidly than harder constituents so that hard aggregate stands up higher than does paste. Undercutting can occur on hard laps, such as are used for fine lapping, but may be prevented with the use of sufficient weight on the specimens. It is very difficult to do quantitative analysis of badly undercut specimens.

It is most convenient to have more than one horizontal lap wheel. (The lapping wheel is illustrated in Figure 1.) Fine grits should be used on one, and coarse on the other. The lap wheels should be sturdy and have durable bearings. Heavy weights are used on the specimens to prevent the lapping away of the softer material before the harder (undercutting.) These weights should cover nearly all the back of the specimen and should be 14 to 16 pounds for a specimen 4 inches in diameter and 16 to 18 pounds for one 6 inches in diameter. Coffee cans of appropriate diameter weighted with lead shot have been found satisfactory.



Figure 1. Lap for producing finely lapped surfaces for microscopic examination and air void analysis. A. Slotted specimen holder, rotating. B. Cover plate to set on top of specimen. C. Guide yoke that retains specimen holder. D. Grit-slurry cup mounted on its motor and containing a spiral agitator/pump. E. Drain. F. Grooved lap.

Lapping Oil

It is strongly recommended that all preparation of HCC specimens exclude the use of water on any surface that is to be examined and that the lubricants required be low viscosity, water-free lapping oil or denatured kerosene mixed with 1/10th part motor oil.* Such lubricants will not dissolve reaction products and secondary minerals as readily as will water-based lubricants. (Although water may be used as a lubricant to cut a surface on the specimen that will not be further examined microscopically, it is recommended that water be used as little as possible.) With this in mind, equipment that uses an oil bath for the diamond-edged saws rather than running water should be selected. The diamond-edged saw should have a smooth (not toothed) edge. The smooth-edged blades produce a smoother cut with fewer ridges and minimize the difficulty of producing finely lapped surfaces. The laps on which the final surfaces are produced should be lubricated with oil. The oil may be reused after filtering and decanting.

The following grinding compounds are used:

- No. 100 (nominal 150 μm), silicon carbide abrasive
- No. 220 (nominal 75 μm), silicon carbide abrasive
- No. 320 (nominal 35 μm), silicon carbide abrasive
- No. 600 (nominal 17.5 μm), silicon carbide abrasive
- No. 800 (nominal 12.5 μm), silicon carbide abrasive
- 5 μm aluminum oxide.

The grit numbers are given only as a guide because the grinding compounds produced by one manufacturer may vary from those produced by another.

Preparation of Finely Lapped Slices

If the specimen surface is quite smooth as it comes from the diamond saw, the coarsest abrasive need not be used. If the grooves from the saw are very distinct, if any fine aggregate has loosened up, or if other gross irregularities are present, the lapping of the surface should begin with the coarsest abrasive. Usually, this is done by hand holding and hand feeding the grit on a small lap, as shown in Figure 2. Once the person is satisfied that he or she has made the surface as flat as possible with the abrasive, then two mechanical laps should be used to lap the surface with successively finer abrasives until it is suitable for microscopical observation.

From time to time, during lapping, when changing to a finer abrasive or changing laps, and when lapping is complete, all surfaces of the specimen should be cleaned gently and thoroughly to remove the grinding compound. Air voids and

*Warning. Take care not to inhale the vapors arising from the lapping oils and solvents used in these procedures. It is best to work in an efficient hood. If that is not possible, a powerful exhaust system should continually clear the air.

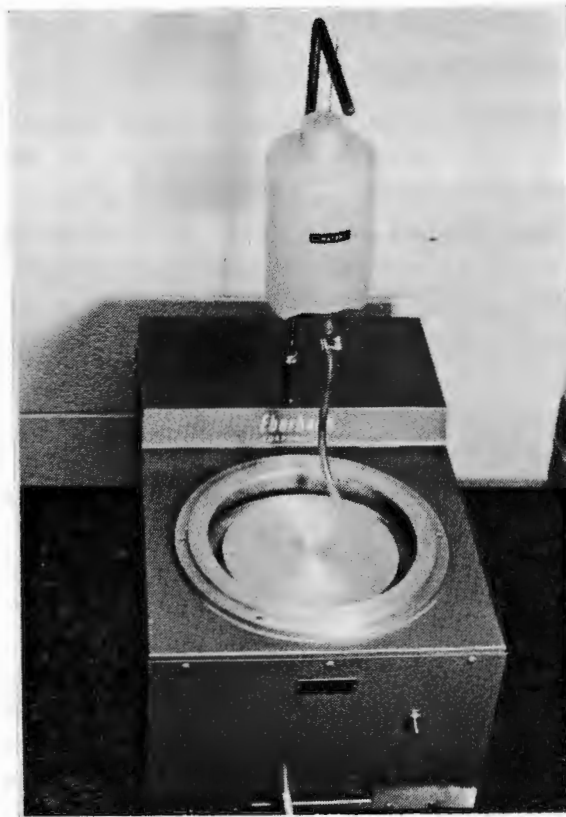


Figure 2. Lap for rough grinding rock and concrete specimens. Suspended bottle is for water or other lubricant. Grits may be shaken like salt from a plastic bottle with a pierced cover.

cracks should be free of such compounds. If the cleaning is not done promptly, the volatiles will evaporate and the grinding compound deposits will cake and become hard to remove. In this laboratory, a few minutes of ultrasonic cleaning have routinely been used for this purpose.* The reaction products seem to be still in place after an ultrasonic cleaning, and the aggregate does not seem unduly loosened.** Alternatively, the specimen can be carefully cleaned of grinding compound and loose particles with a soft brush (baby's hair brush) while immersed in a container (safety approved) of trichloroethylene or with a fine high-velocity spray of solvent. As much grinding compound should be removed as possible without damaging the surface.

Refer to ASTM C856, Standard Practice for Petrographic Examination of Hardened Concrete, and ASTM C457, Standard Practice for Determination of Air

*Warning. It has been reported that use of ultrasonic cleaning equipment may be harmful to the surface of concrete specimens; therefore, such treatment should not be used without care and experimentation.⁶

**Warning. Do not immerse any portion of your hand in an ultrasonic bath while the ultrasound is on. The cavitation can erode fingernails and damage skin.

Void Content and Parameters of the Air-Void System in Hardened Concrete, for further details on specimen preparation.^{3, 6}

A surface that is satisfactory for microscopical examination (either a general petrographic examination with the stereomicroscope or an analysis of the air void system) will show an excellent reflection of a distant light source when viewed at a low incident angle, and there should be no noticeable relief between the paste and the aggregate surfaces.⁶ Such a surface is illustrated in Figure 3. If the day is reasonably bright, the light from a window should be scanned as it is reflected from a finely lapped slab. It should be possible to discern the reflections of outdoor objects, such as buildings and trees. Alternatively, the slice should be held so that it reflects light from an overhead light fixture. It should be possible to see the small details of the diffusion cover of the light or even discern the presence of writing on the light bulb. The edges of the sections of the air voids will be sharp and not eroded or crumbled; air void sections including those as small as 10 μm in diameter will be clearly distinguishable at magnifications of less than 150x; and all cracks will be easily visible and free of debris.

If difficulty is encountered in preparing a lapped surface of the proper quality, the usual cause is a weak cement paste matrix. The problem is manifested by the plucking of sand grains from the surface during the lapping, with consequent scratching of the surface, and by undercutting of the paste around the harder aggregate particles. A second cause of difficulty may be friable particles of aggregate. For any quantitative analyses, areas that are scratched or imperfect indicate the need for additional preparation of the specimen. Generally, additional time on the free abrasive laps is all that is required.

If undercutting is the major problem, additional weight on the back of the specimen while it is being lapped can be helpful.

If the concrete is immature, further carbonation of the paste may help produce a satisfactory surface. Carbonation may often be achieved by exposure of the cut surface to laboratory air of moderate humidity (20 to 80 RH) for 48 hours. Alternatively, the cut specimen can be supported over water that is kept agitated for 10 hours by pieces of dry ice. If the container in which this accelerated carbonation is taking place is loosely covered, the carbonation will continue until all the carbon dioxide is dispersed, even after all the dry ice has sublimed away.

Very fragile specimens may require impregnation techniques. A number of materials and techniques of impregnation are available. Various modifications of the techniques used to impregnate thin sections (for observation at higher magnifications) may be used. Caranuba wax is the impregnating material recommended in ASTM C457,⁶ but it must be used with certain precautions, as mentioned. Further details on the preparation of surfaces for microscopical analysis may be found in ASTM C457.⁶

If horizontal slabs were taken from cores in order to study changes within the air void system (or other feature) with depth, it may be desired later to finely lap and examine a vertical cross section of one or more of the lapped sections already

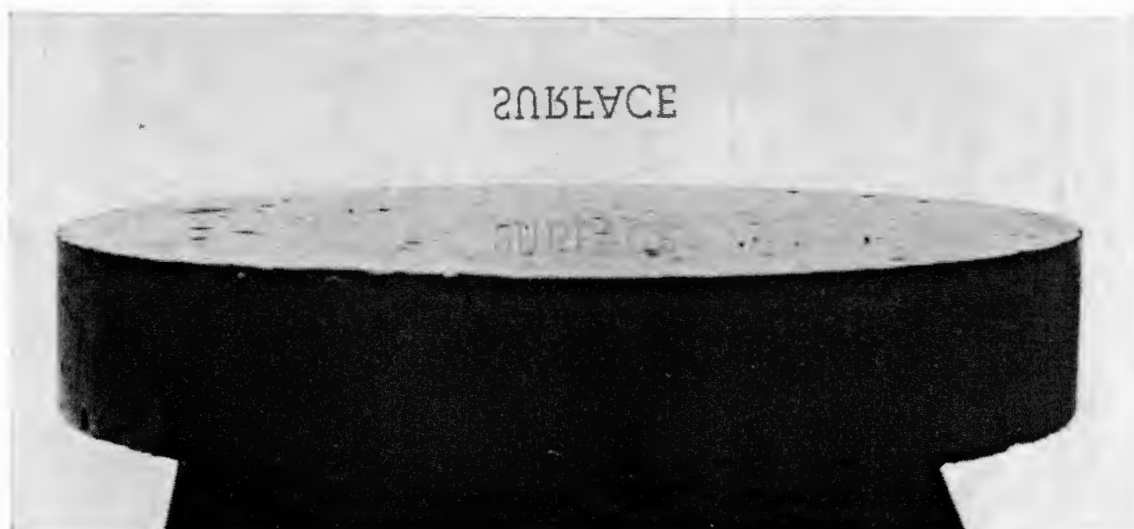
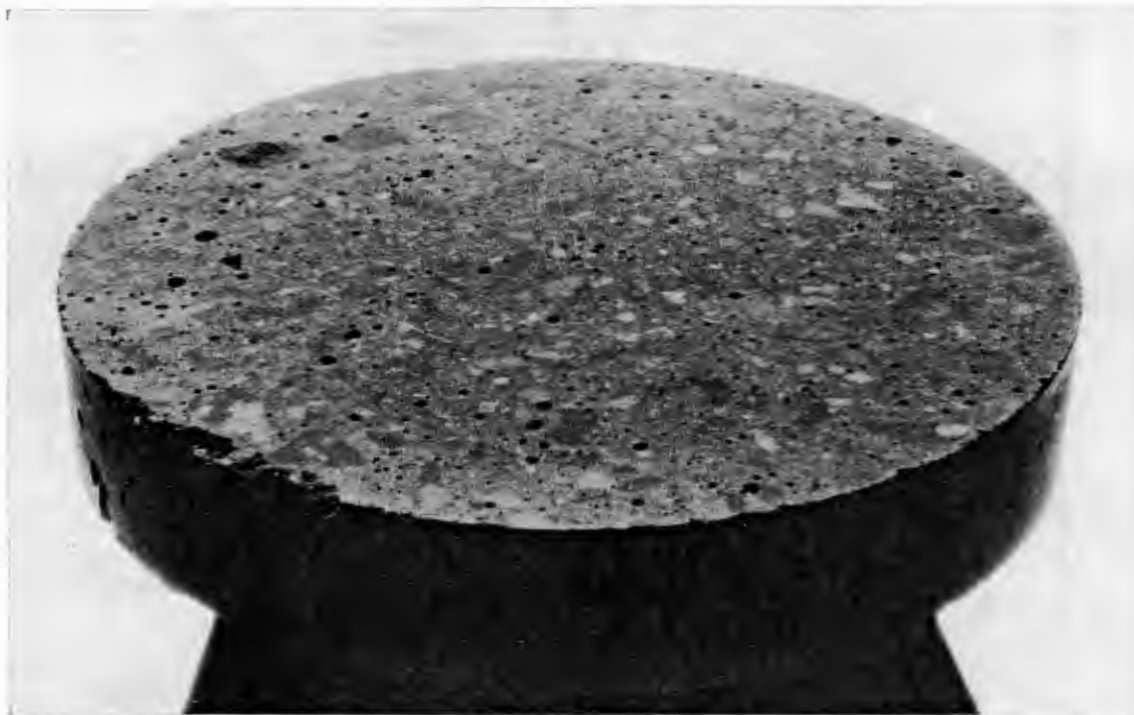


Figure 3. These photographs of the same well-prepared surface illustrate the quality of the surfaces required for microscopical examination and analyses. *A.* The surface viewed in ordinary illumination. *B.* The surface viewed at a very low angle toward a source of illumination. The quality of the reflection of a distant object (here, the quality of the reflection of the reversed word "SURFACE" on the screen) indicates the flatness of the lapped surface.

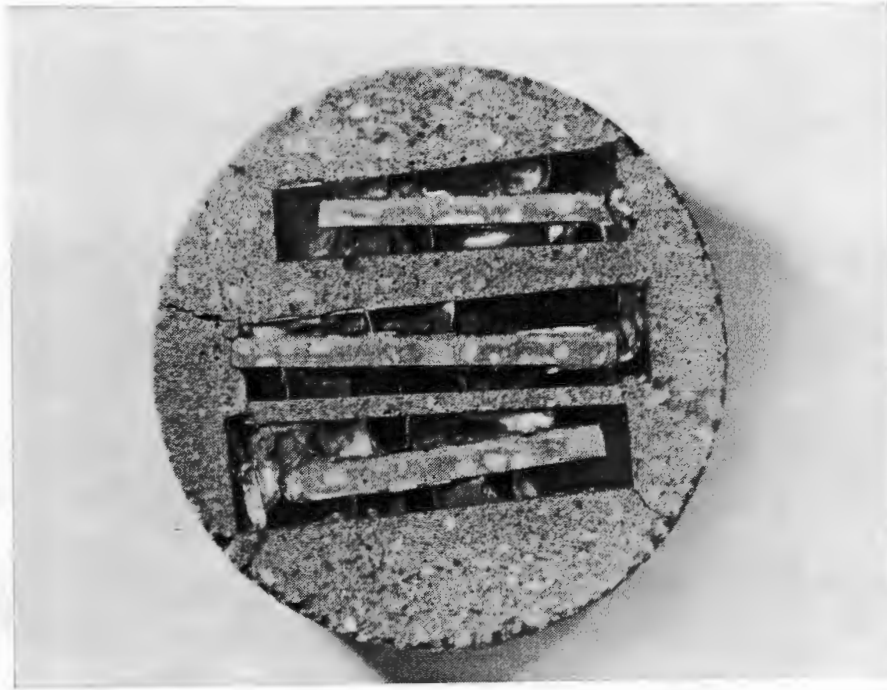


Figure 4. *A.* Edges of the thin top surfaces of a concrete core embedded in epoxy and repotted in mortar in a mold 6 inches in diameter and then resliced and finely lapped to allow examination of the vertical surface. In this case, the concrete has been slightly etched with a weak HCl solution to delineate the depth of carbonation. *B.* An enlarged portion of the middle slice in *A.* The carbonated area is the area above the lower edge of the whiter area near the wearing surface.

available. In such case, it will be necessary to secure the section in question in a upright position in some sort of mounting medium so that a vertical surface can be cut and lapped (see Figure 4).

Thin Sections

For the Standard Petrographic Microscope

The petrographic microscope used to examine nonopaque materials uses light that travels through the thin section parallel to the optic axis of the microscope. The light is collimated into a bundle of parallel beams that are polarized in one direction. This direction varies from microscope to microscope (most often N-S) and may be adjustable. The usual thickness of thin sections fabricated for the examination of rocks and minerals is 25 to 30 μm . For the determination of the component minerals in coarse-grained igneous and metamorphic rocks, this thickness general works fairly well. Some rock-oriented petrographers depend on thin sections being a standard thickness so that they can judge the birefringence of a mineral grain under observation by the dispersion caused by the standard thickness. On the other hand, many concrete petrographers prefer to work with much thinner thin sections. They have become accustomed to judging birefringence by comparison between mineral species.

When the size of the phases of interest is less than twice the thickness of the thin section, the phase boundaries tend to become obscured by the overlap of the phase individuals. The finer the size of the phase individuals, the less well defined the boundaries, and the less the difference in the index of refraction, color, and birefringence, the more difficult it becomes to determine the identity of the phases or even identify the boundaries.

Some of the aggregates whose structure must be determined accurately in concrete petrography are very fine grained. The microstructure of the deleteriously reactive carbonate aggregates cannot usually be deciphered in thin sections of standard thicknesses. These rocks are composed of rhombic crystals of dolomite (usually less than 20 μm across) "floating" in a murky calcite micrite. (The murky background is caused by submicroscopic particles of organics, iron oxides, and sulfides and various other insolubles that have been included in the micrite. When these insolubles include relatively high percentages of opaques, it can be impossible to discern details in a thin section as thin as 20 μm). The micrite itself may have a grain size of less than 5 μm .) The undulating extinction of alkali-reactive coarse-grained quartz can usually be seen in sections 25 to 30 μm thick, but much of the reactive quartz present in fine-grained metabasalts and mylonites cannot be detected in thin sections of standard thickness.

Most cement is very fine. The greater portion will pass a 200 mesh sieve (less than 74 μm). The cement hydrates into even finer phases of low to negligible birefringence with nearly identical color and indices of refraction (low). Observa-

tion of a thin section of HCC 25 μm in thickness could lead a rock-oriented petrographer to state that the material between the aggregate grains is "ground-mass." The term "ground-mass" has long been used by petrographers to indicate that the material is too fine for determinative microscopy, as the "ground-mass" of a basalt.

The microscopic study of HCC at the magnifications available with the petrographic microscope must include data concerning the degree of hydration and the pore space between the hydrated phases.

Thus it can be seen that thin sections of portland cement concrete and certain aggregates must of necessity be very thin. Ideally, the thin section should also be flat and cover a large portion of the glass on which it is mounted.

Unless special equipment and techniques are used, lack of flatness and small areal extent will sometimes have to be overlooked and data will have to be obtained from small thin sections that are thicker in the middle than at the edges. This, of course, means that the loss of entire thin sections during fabrication is great and that it is good to begin fabrication with a number of thin section blanks so that the likelihood of two or more surviving the procedures is pretty good. These blanks are shaped to the size of a petrographic glass slide with a thin-bladed 6-inch diamond saw (see Figure 5).

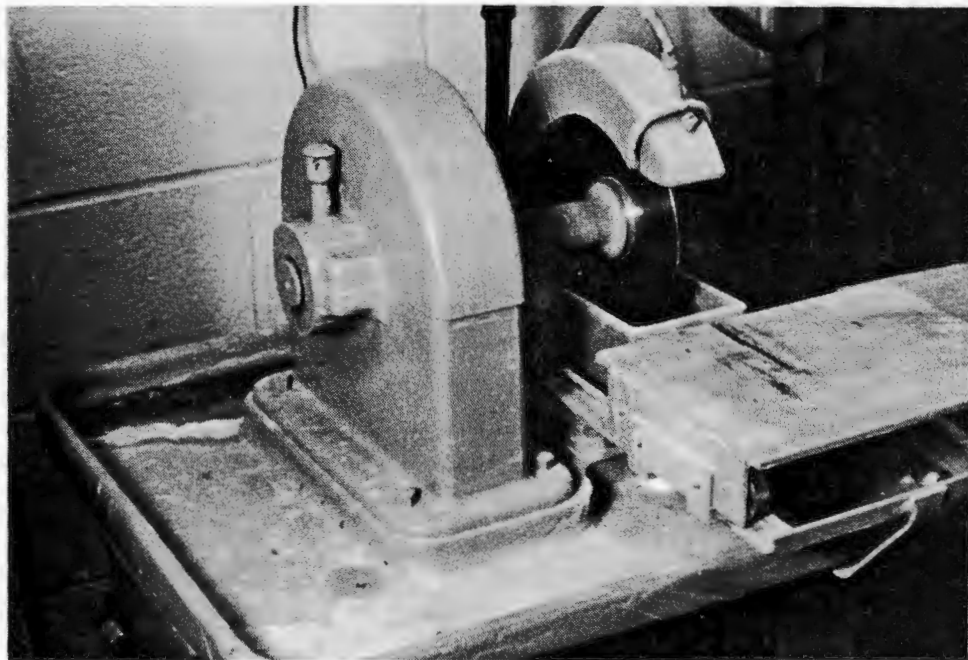


Figure 5. Thin, diamond-edged, 6-inch rotary saw for shaping thin section blanks. This saw is cooled with tap water. It would be better if an oil lubricant/coolant was used.

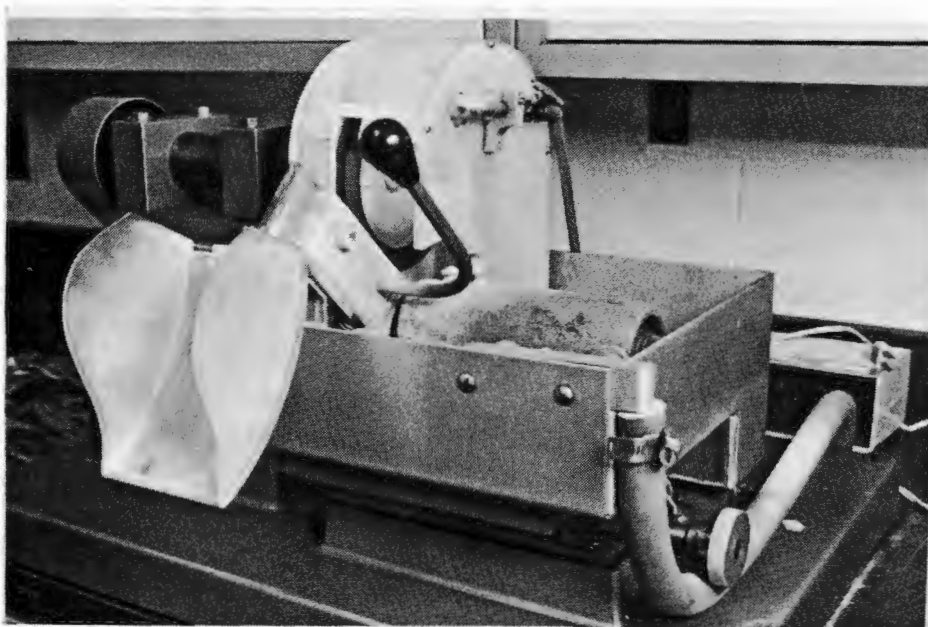
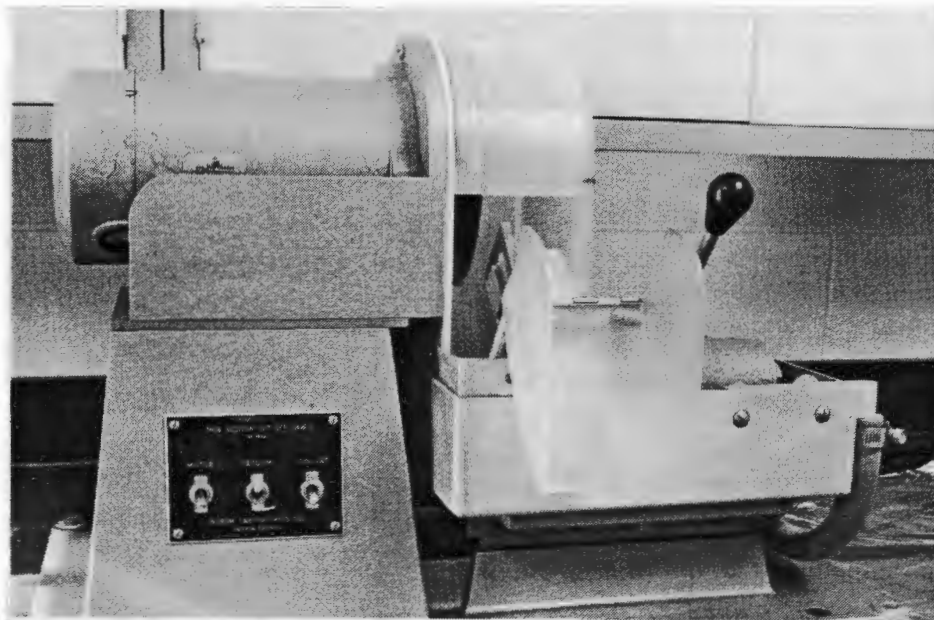


Figure 6. Ingram-Ward thin sectioning equipment. A. Diamond-edged cut-off saw. B. Diamond-bearing, cupped ceramic grinder. The glass of the thin section is held on the vacuum chuck, and the handle is used to move the work into position up against the saw or up against the face of the grinder. The relative position of the chuck to the saw or grinder can be adjusted with a small dial at the right-hand end of each piece of equipment.



Figure 7. Glass (from a hardware store) covered with a grinding compound slurry, in a pan to catch the overflow. Used for final grinding of ordinary thin sections. The two plastic bottles have holes in the screw-on covers and waxed paper cups for dust covers. These bottles are used for grit and lubricant slurries.

In this laboratory, both portions of the Ingram-Ward thin sectioning equipment (Figure 6) is used. The procedures recommended by the manufacturer of the equipment should be followed. No water should be used in the preparation of thin sections of HCC. After the grinder has reduced the section to about 30 μm in thickness, the section should be hand finished with 600-mesh SiC and 5- μm Al_2O_3 in a water or oil slurry on a glass plate (in a firmly supported flat pan to catch overflow) (Figure 7) until the section is approximately 25 μm thick or less, depending on the material being sectioned and the purposes of the examination. The grinding motion used should be that of making a numeral 8 and should cover as much of the entire surface of the glass as is possible without letting the edge of the glass touch the section being produced; the 8s should move over the glass surface and rotate, crossing as many different places on the glass as possible.* For most purposes, the pressure on the back of the thin section should be evenly distributed and just sufficient to slide the section around on the abrasive when sufficient lubricant is used.

*Warning. Examine the glass plate regularly for flatness. When it becomes dished due to the grinding action, discard and replace.

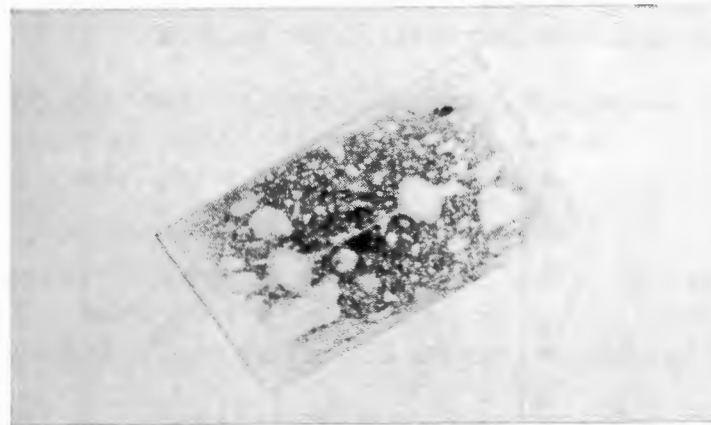
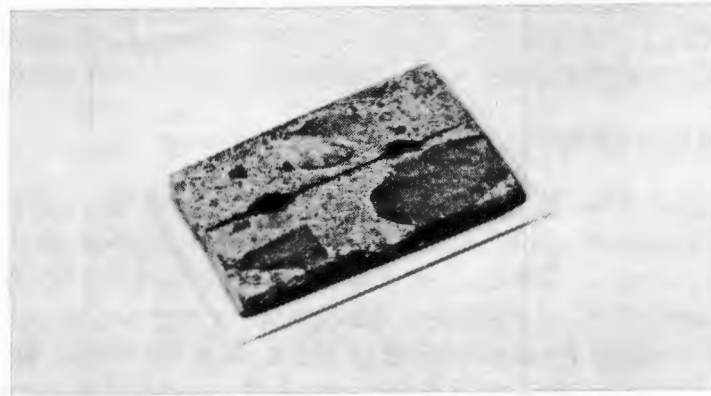
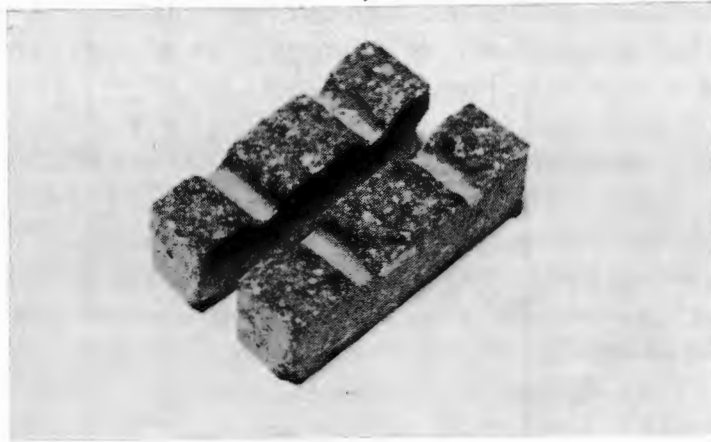


Figure 8. Steps in preparing a thin section showing wearing surface details. Actual size. *A.* Small blocks of wearing surface. *B.* Blocks cemented to microscope slide. *C.* Thin section of surface blocks.

Alkali-Carbonate Thin Sections

In the case of making thin sections to detect the presence of alkali-reactive carbonate rocks, the thin section may be lapped down to zero thickness at one end and wedge toward a more standard thickness at the other. This may be accomplished by pressing more heavily on one end of the back of the section during the hand finishing of the thin section. By this method, there will exist at least a small area of the section that is of the correct thickness to allow detail visibility and the likelihood of losing the entire section will be greatly lessened. It is recommended that the pressure be kept nearly evenly distributed until the section is down to 25 μm thick. The best thickness for viewing the texture of carbonate materials is usually achieved when observation with crossed polarizers indicates that much of the carbonate shows a pink of the fourth order (or less) and the overall, glaring, high-order white, generally typical of the carbonates, has begun to change to colors.

The author estimates that the best compromise thickness for thin sections of concrete and some aggregates is about 10 μm . Each material studied will probably have its own optimum thickness. Unfortunately, the optimum is often quite thin and difficult to achieve without loss of the entire specimen cemented to the glass.

Wearing Surface Thin Sections

On occasion, the exact details of the texture and the aggregates of the wearing surface require examination and study with the higher powers of the compound petrographic microscope.⁷ When this is desired, two small portions of the surface under study are cemented to a slide with the wearing surfaces together in the center (to protect the area of interest from the greatest thinning action) and a thin section of the thickness appropriate for the material and for purposes of the study is produced. Figure 8 illustrates some of the steps in this process.

For Use with Fluorescent Incident Illumination

Sections for use with fluorescent illumination are much like ordinary thin sections. The major difference is that they have been impregnated with a fluorescent dyed epoxy.⁸ The thinness of the section is of paramount importance in fluorescent sections.

Because the section is of concrete (usually with considerable porosity) that has been impregnated with a dyed epoxy and properly cured, it is not as difficult as might be thought to produce sections 15 μm and less in thickness.

The methods detailed⁸ were those devised in this laboratory that would involve the least cost for equipment outlay. The Ingram-Ward equipment (Figure 6) was already available, and the major purchases were the vacuum oven (Figure 9) and the Syntron vibratory polishing lap (see Figure 10). It was found necessary to make sure that the most impregnated side of the chip was very flat and was cemented down to the final slide to be used because the impregnation was usually only about 1.5 mm deep. With the Ingram-Ward equipment, the use of the series of work



Figure 9. Vacuum oven with thin section blank in small disposable dish that will hold the dyed epoxy.



Figure 10. Syntron vibratory polisher and weights complete with sponge rubber bumpers. The thin sections are attached to the bottom of the weights.

glasses described in the numbered steps below was necessary. Other laboratories could easily devise other methods of producing these sections, depending on the equipment available to them.

The method is as follows:

1. Select and mark the specimen area. Generally, the area selected should be centered on mortar rather than on coarse aggregate. The area should be about 16 x 32 mm to fit the well area of the final mounting glass.
2. With the small diamond blade saw (Figure 5) and, preferably, an oil lubricant, cut a small slab 4 mm thick from the specimen area. Extremely fragile specimens may require thicker slabs.
3. Rinse the slab well in acetone or 1,1,1-trichloroethylene.* Thoroughly air dry or oven dry the slab, and mount with quick-set glue to the work glass. Use a thin, even layer of glue so that the specimen will be parallel with and tight on the glass.
4. Mount the slab in a vacuum chuck of the Ingram-Ward grinder (Figure 6b), and grind with the cupped ceramic diamond grinder until the entire surface of the specimen has been cleaned of saw-damaged material.
5. Wash by soaking in four changes of acetone over a period of 2 or 3 hours. Air dry.
6. Dehydrate in a vacuum oven at 80°C overnight (Figure 9). Maintain a vacuum of 10 mm Hg for about 1 hour before leaving for the night. In the morning, turn the heat off and allow the closed oven and specimen to come to room temperature before releasing the vacuum.
7. Pot the specimen with the ground surface up in the fluorescent epoxy to which has been added some 0.3-mm Linde (a grinding compound) to protect the edges during later polishing. At this laboratory, disposable plastic Petri dishes (5 cm in diameter) are used as potting containers. The corners of the work glass must be snapped off to make the specimen fit. The use of larger containers would result in a waste of epoxy. The containers holding the specimens covered with epoxy are placed in the vacuum oven at room temperature, and the vacuum is intermittently and slowly brought down as low as possible and kept there until gassing has stopped. This step takes about 3 or 4 hours and is best done in stages to prevent overflow of the bubbling epoxy and to allow it time to penetrate the specimen. Release the vacuum, and allow the air pressure to force epoxy into all portions of the void system. Check for further gassing by lowering the pressure again. Repeat until gas evolving from the specimen is minimal.

*Persons unfamiliar with the hazards of these compounds are referred to the Chemical Safety Data Sheets published by Manufacturing Chemists Association, Inc., 1825 Connecticut Avenue, N.W., Washington, D.C. 20009.

8. Cure at room temperature for 36 hours, or until the epoxy is hard and brittle and does not show any plasticity when tested with a pick. (It is necessary that the grinding procedures to follow remove the epoxy by the same infinitesimal chipping method as used for removing the aggregate rock and cement paste.)
9. Trim excess epoxy and glass from the specimen with a saw. Clean the bottom of the glass of plastic and epoxy by peeling, and mount on additional work glass with quick-set glue. (This work glass is required to cover the vacuum chuck in the next step.)
10. Clean the final mounting surface by placing the new work glass on the vacuum chuck of the Ingram-Ward grinder (Figure 6b), and carefully grind off the epoxy coating on the specimen surface. A change in the sound of grinding will be noticed when the epoxy is gone. Remove as little of the specimen as possible. This highly impregnated area is the best portion of the specimen for use as the thin section.
11. Lap the mounting surface by hand with a 5- μm Al_2O_3 water slurry on a glass. Clean in an ultrasonic cleaner with acetone solvent. Place under a 10-mm vacuum at 50°C for 3 or more hours to remove all oil and water. (Do not soften epoxy at high heat.)
12. Mount the lapped, impregnated surface in a well petrographic slide with undyed epoxy. It is very important that the mounting step be performed carefully to eliminate air pockets and ensure a good bond. Place the mounted sample in a clamp on a clamp stand to cure overnight, as shown in Figure 11.
13. Cut off the excess thickness of the specimen by mounting the final well slide against the vacuum chuck of the Ingram trimmer saw (Figure 6a), as shown in the diagram in Figure 12. Thus the specimen is made reasonably thin and the two work glasses are removed.
14. Reduce the specimen to about 30 mm in thickness in the Ingram-Ward grinder. Rinse well with acetone or 1,1,1-trichloroethylene.
15. Lap by hand with 5- μm Al_2O_3 water slurry on the glass. Clean in an ultrasonic cleaner with acetone.
16. Polish on a Syntron vibratory polisher (Figure 10) by attaching the slide to the flat side of the weights with double-sided adhesive tape. Place rubber bumper rings on the weights and, using 3 mm of diamond compound and lapping oil, vibrate for about 16 hours, or until the thickness is reduced to 10 μm and the surface is polished and free of scratches. Certain types of aggregate will necessitate further polishing (after thorough cleaning) with 1- μm diamond compound in a second Syntron bowl. Clean sections carefully.
17. When not in use, store sections in a dark, airtight cabinet with ascarite and drierite to prevent carbonation.

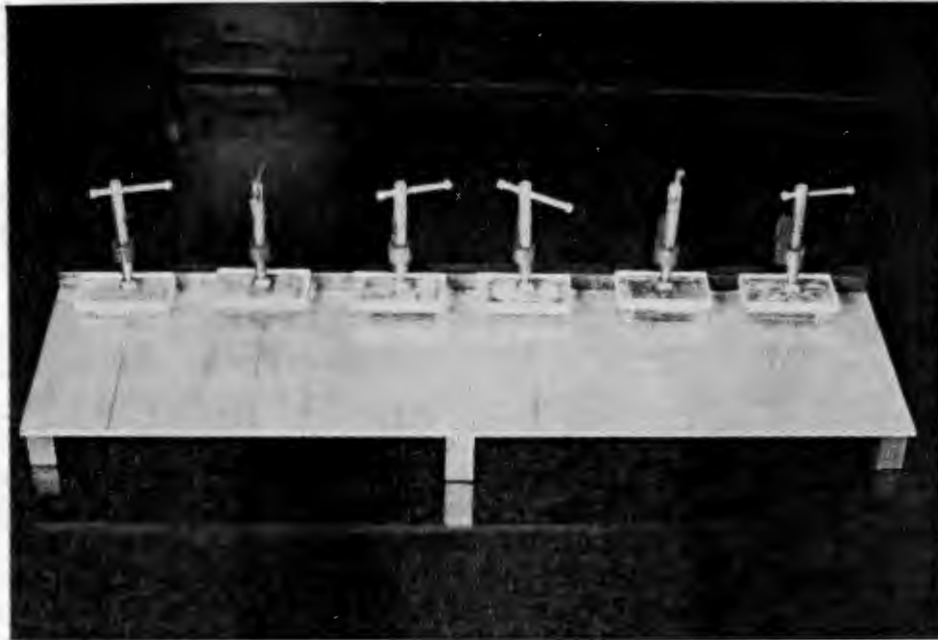


Figure 11. Clamp stand for mounting thin sections to the final well slides. The little slabs of lucite are used to distribute stress.

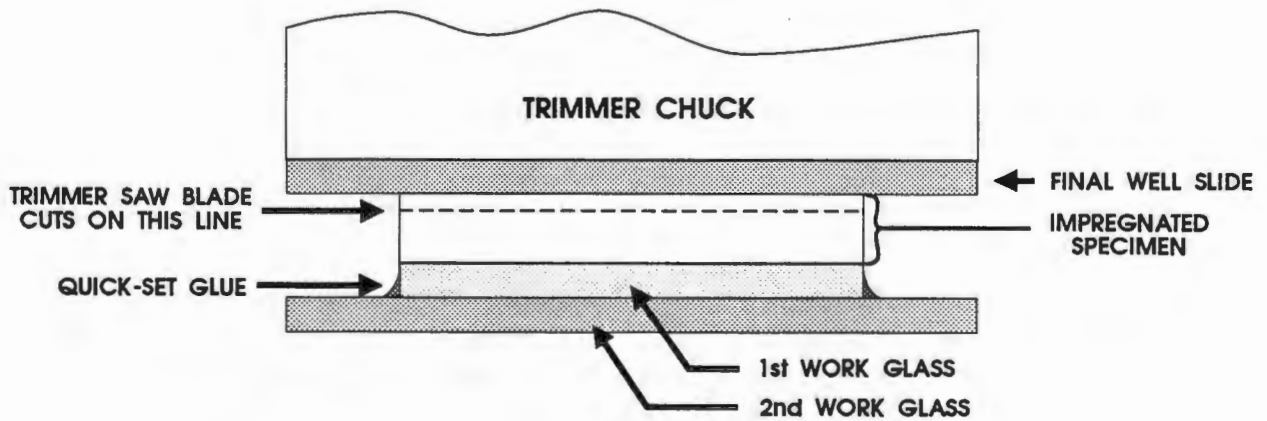


Figure 12. Impregnated thin section slab, with work glasses mounted on final well slide; ready for trimmer saw.

The method any particular laboratory uses to produce thin sections will depend on the equipment available and the preference of the technical personnel. Pacific Northwest Laboratories, Richland, Washington, recommends the use of a belt sander and a bench lapping machine with diamond-impregnated laps.⁹ Romer, of Labor für Präparation und Methodik, Beinwil am See, Switzerland, recommends the use of a diamond-tooled milling machine.¹⁰ Experience in this laboratory has been with the Ingram-Ward thin sectioning machines. The ultrathin sections are being examined by eye rather than with an electronic image analyzing device. It has been found that sufficient flatness for our present studies has been obtained with the equipment listed by Walker and Marshall.⁸ The expendables they listed in Table 2 were those used in 1978. Since that time, the dye and both of the epoxies have become unavailable.

The dye presently used is Fluorol Yellow 088, available as samples from BASF Wyandotte Corp., Holland, Michigan. This dye dissolves completely in epoxy; that is, no particles can be seen at 600x magnification and the epoxy is evenly colored by the dissolved dye.

Epo-Tek 301-2 from Epoxy Technology, Billerica, Massachusetts, is a good impregnating epoxy. It dissolves the dye completely. It has sufficient shelf life to be useful after a year or so although it may have to be warmed a little. It has a sufficient pot life to remain fluid through several hours of vacuum impregnation and can be formulated to cure with a little heat treatment and over several days to a hard brittle substance that shows no plasticity. When cured, the epoxy is not soluble or softened in oil, alcohol, acetone, or trichloroethylene.

The mounting epoxy presently used can be either Epoxide from AB Buehler, Lake Bluff, Illinois, or Section-Lok epoxy mounting medium from Microtech Engineering Laboratory Inc., Clifton, Colorado. Of the two, the Section-Lok has the longer shelf life.

QUESTIONS AND SUGGESTIONS FOR FURTHER RESEARCH

Changes in Materials and Methods

As with so many sciences, there are many changes and advances being made in the study and diagnosis of HCC by means of all the petrographic methods. New materials are being used to fabricate and maintain HCC pavements and structures. Each of these new materials brings a new look to the microstructure of HCC, and the petrographer must continually devise new methods to keep pace.

As our civilization changes and as governmental regulations change, certain shortages in materials will develop. Because a large share of the cost of aggregate is the transportation from the quarry to the site of use, shortages in the supplies of

low-cost aggregate can be due to urban expansion over older high-quality source sites. Certain innovative companies are using expensive complex beneficiation technology to mine sand and gravel deposits with a short haul distance and find that they can compete economically with companies that have a longer haul but material that requires little beneficiation. Petrographers need to assess such cleaned up material and the reactions of the aggregates in HCC.

Due to EPA air quality requirements and the economics and environmental value of preheaters, portland cement with a low alkali content is becoming scarce. In some cases, the cement from a single plant may vary in alkali content from season to season.

Cement technologists need to assess this situation and make provision for overcoming the problems that the higher alkali cements will create when used with the alkali reactive aggregates.

Many of the practices that generate waste will have to be changed. We have the means to change some of them now. Concrete that is returned to the plant in the ready-mix truck is presently discarded, and the truck washed out to prevent setting up of the mixture in the truck. (All trucks must be washed out daily.) This means that not only does the waste concrete have to be disposed of, but the wash water must go into settling ponds before it is returned to the environment. It has been found that if a retarder is added to the excess returned concrete, the concrete can be kept in stasis over a night or a weekend. When the concrete is required for use, an accelerator can be added and the concrete will be ready for use. The problem is that it is easy to make a mistake on the dose of these chemicals. It will require the efforts of many types of concrete technologists to discover the proper methods of using these chemicals. As it becomes common to conserve concrete and protect the environment by such methods, the dose errors possible will become more common and concrete petrographers will have to be on the lookout for the results of such errors.

Equipment

New equipment is continually being devised to monitor the parameters of hardened concrete. It is the job of the petrographer to assess the value of new methods and equipment and to decide which equipment is of value in which situation.

Air Void Parameters

Air void system analyses can be efficiently performed with several kinds of equipment. The linear traverse type tabulates the chord lengths and records them for later analysis. The point count equipment records the type of substance (air void, paste, or aggregate) at a large number of randomly distributed points and uses the data to calculate the air void parameters. These two types of equipment depend

on the operator's eye for decision making and the operator's hand on the buttons to record the data. Image analysis equipment requires that the specimen be specially prepared so that the three phases of interest are three distinct tones (for example, white, black, and medium gray). The presence and shape of areas of the three selected tones are determined by electronic eye, and the data automatically recorded and sorted. In all cases, the data must be manipulated so that the parameters of the air void system detailed in ASTM C457⁶ are calculated. It is not known which method produces the most accurate results or how accurate their results need to be. Point count is favored by those who need speed. Linear traverse is favored by those who wish a record of the chord length distribution. Image analysis is favored by those whose equipment budget permits it and who do not care that the specimen is not examined by the human eye and that the specimen has been dyed and filled and is no longer a natural surface.

Chloride Permeability

The quick chloride ion permeability test devised by Whiting¹¹ is a good method for obtaining the relative permeability of chloride. Appendix I in his report gives detailed instructions on the method of test. Trained technicians can easily perform this test without the close supervision of a petrographer or chemist.

Other

The basic sciences have contributed a number of methods that can be used in petrographic and related types of examination of HCC and other materials. If funds are available, every concrete petrographic laboratory should be equipped with image analysis equipment, a scanning electron microscope, and X-ray diffraction and X-ray fluorescence equipment. If these advanced types of equipment are used in the study of HCC, some of the basic questions that have remained unanswered concerning these materials could receive answers.

Further Research

At the present time, we have several different theories to explain effects known to occur in HCC, but in many cases we are working from strictly empirical evidence, with no proof of the exact mechanisms that produce these effects or any real knowledge concerning the limits over which these effects operate.

Questions on Freezing and Thawing

The specification for the percentage of air to be entrained in freezing and thawing resistant concrete has been devised to protect saturated concrete from the freezing of the water contained therein. We have little or no knowledge concerning what the saturated state is. We have no method for measuring the degree of saturation. We do not know if the entrained air voids become partially water filled or if

they are completely empty when the concrete is susceptible to freezing and thawing distress. Although we know that water in a very small space cannot freeze, we do not know what portion of the water in a concrete system becomes frozen or at what temperature the water in the various sizes of capillaries freezes.

Deicers increase the severity of freeze-thaw distress. The reason for this has never been fully investigated. Does the salt increase the number of cycles of freezing and thawing or does the salt, scattered on the surface, by dissolving in water and taking up heat (as with freezing ice cream) actually lower the temperature in the top layer of the concrete and thus increase the depth of freezing?

We theorize that the air void system provides relief from the pressures caused by freezing. We are not sure that we know the carrier of those pressures.

- Is water or air forced ahead of the freezing front?
- Is it forced into the air voids?
- Would nonpermeable but collapsible plastic-coated spheres provide protection?
- Is it possible to have a concrete so completely saturated and subjected to freezing temperature (whatever that is) for such a long time and so repeatedly that the air void system (regardless of how good) can fail to protect the concrete?

Questions on Alkali-Aggregate Reactions

We know that the alkali-aggregate reactions require reactive aggregate, alkalis in the cement or in contaminants, and the presence of water. We are fairly certain that the alkali-carbonate reactions are active at lower alkali contents than are the alkali-silicate reactions with the more reactive silica-bearing materials. We do not have a good idea of the exact minerals or the state of crystallization of the minerals involved in the slower alkali-silica reactions or of the alkali limits required to prevent these reactions. The present thinking is that the addition of a sufficient amount of good quality pozzolanic material will prevent these reactions indefinitely. So far, this theory has not had enough time to be proven. It may be that we should not be thinking about preventing these reactions completely but rather should be concerned with preventing them over certain periods of time.

Numerous theories about the exact mechanisms of these reactions have been reported in the literature. Many of these theories seem to be contradictory or conflicting. Some questions remain unanswered:

- Is there an osmotic cell mechanism in operation when a silica gel imbibes water and expands and causes distress?
- What is the nature of the expansive material in an alkali-carbonate reaction?

It has been reported that alkali-silica distress is more severe where the structure has received deicing salts.

- Is this due solely to the sodium and potassium salts present in a particular kind of rock salt used or does pure salt also increase the distress?
- Is there any similar effect on the alkali-carbonate reaction?

Questions on Cement Content

Because the cement is the most expensive ingredient in concrete, the purchasers of this material are concerned about whether or not the concrete contains the specified amount of cement. In our experience, in Virginia, under government contract, it is only rarely that a concrete supplier by either accident or design fails to supply concrete of the correct proportioning to the job site. The problems usually arise when water is added at the construction site to facilitate placement and finishing. Under these circumstances, the concrete placed may have a high water/cement ratio, may fail the strength testing, and may not be as durable as would be expected from the design of the mixture. There have been many tests devised to determine either the amount of cement or the water/cement ratio of fresh concrete. However, as in so many other cases, no test is worth the trouble it takes if it is not performed on the material representative of the final product purchased. It does not appear likely that any of these tests on fresh concrete would be very helpful.

Research should be devised to enable the specifications to advise the contractor what to do when any of the problems arise that have in the past led to the addition of water and retempering. In many cases, the prohibition against using aggregates with a high water demand unless a sufficient amount of a water-reducer additive of an approved type is employed should be enforced. The petrographer can be instrumental in the devising and enforcement of proper specifications.

READING LIST

Applicable ASTM Standards

- C33: Standard specification for concrete aggregates
- C39: Standard test method for compressive strength of cylindrical concrete specimens
- C42: Standard test method for obtaining and testing drilled cores and sawed beams of concrete
- C125: Standard terminology relating to concrete and concrete aggregates
- C227: Standard test method for potential alkali reactivity of cement aggregate combinations (Mortar-bar method)
- C311: Standard test methods for sampling and testing fly ash or natural pozzolans for use as a mineral admixture in portland cement concrete

- C289: Standard test method for potential reactivity of aggregates (chemical method)
- C294: Standard descriptive nomenclature of constituents of natural mineral aggregates
- C295: Standard practice for petrographic examination of aggregates for concrete
- C457: Standard test method for microscopical determination of parameters of the air-void system in hardened concrete
- C618: Standard specification for fly ash and raw or calcined natural pozzolan for use as a mineral admixture in portland cement concrete
- C666: Standard test method for resistance of concrete to rapid freezing and thawing
- C823: Standard practice for examination and sampling of hardened concrete in constructions
- C856: Standard practice for petrographic examination of hardened concrete
- C873: Standard test method for compressive strength of concrete cylinders cast in place in cylindrical molds
- C1084: Standard test method for portland cement content or hardened hydraulic cement concrete

Manual of Aggregate and Concrete Testing (gray pages, Vol. 4.02).

The ASTM standard methods should be used with care, and all applicable cautions taken. They should be conducted by persons who are qualified by education or experience to conduct such tests and/or use such standards. The list gives only a few of the test methods under the jurisdiction of Committee C9 on Concrete and Concrete Aggregates as published in the *ASTM Annual Book of Standards*, Section 4, Construction, Vol. 4.02, Concrete and Aggregates. Many of the standards not listed will be needed for specialized testing of individual materials, such as epoxy rebar coating, fly ash, and ground granulated blast furnace slag, or as they may be referenced in other standards or for testing individual properties or kinds of concrete. It is recommended that the entire volume be available for reference.

Reference Collections of Significant Value

ASTM Committee on Concrete and Concrete Aggregates. 1978. *Significance of Tests and Properties of Concrete and Concrete Making Materials*, STP 169B. ASTM, Philadelphia.

This worthwhile work is a remake of STP 169A (1966) and thus of STP 169 (1956). All volumes of the STP 169 series have the same title. Each of these volumes can be considered to outline the state of the art for the time each was written. Thus, together they detail the recent history of concrete technology.

ASTM Committee on Performance of Concrete—Chemical Aspects. 1965. *A Symposium on Alkali-Carbonate Rock Reactions*. Highway Research Record No. 45. Highway Research Board, Washington, D.C.

Bredsdorff, Per; Idorn, G. M.; Kjaer, Alice; Plum, M. N.; and Poulsen, Ervin. 1960. Chemical reactions involving aggregate, in *Chemistry of Concrete*, Proceedings of the Fourth International Symposium, Session VI, Volume 2, NBS Monograph, pp 749–806, Washington, D.C.

This entire volume is a classic work and includes discussions of many of the papers, including this one. The volume is mainly concerned with alkali-silica reactions.

Alkali-Aggregate Reactions

Diamond, Sidney. 1978. Chemical reactions other than carbonate reactions (Chapter 40) in *Significance of Tests and Properties of Concrete and Concrete Making Materials*. ASTM, Philadelphia.

Particularly good on alkali-silica reactions.

Hilton, M. H. 1974. *Expansion of Reactive Carbonate Rocks Under Restraint: Cement Aggregate Reactions*. TRR-525 (7 reports). Transportation Research Board, Washington, D.C.

This is the only experimentation the author has heard of in which the reaction took place under restraint.

Mather, Bryant. 1974. *Developments in Specification and Control: Cement Aggregate Reactions*. TRR-525 (7 reports). Transportation Research Board, Washington, D.C.

An excellent summary of the knowledge available at the time.

Newlon, H. H.; Sherwood, W. C.; and Ozol, M. A. 1972. *Potentially Reactive Carbonate Rocks: A Strategy for Use and Control of Potentially Reactive Carbonate Rocks* (including an annotated bibliography of Virginia research). VHRC Report No. 71-R41. Virginia Highway Research Council, Charlottesville.

All of the reports in this series may be found useful. This particular report includes an extensive bibliography and is therefore listed to provide a reference and guide to the others of the series.

Rogers, C. A., ed. 1990. *Canadian Developments in Testing Concrete Aggregates for Alkali-Aggregate Reactivity*. Report No. EM-92 (19 papers). Ministry of Transportation, Engineering Materials Office, 1201 Wilson Avenue, Downsview, Ontario, Canada.

Recommended for information concerning the most up-to-date methods of testing for alkali-carbonate, alkali-silica, and alkali-silicate reactivity.

Ryell, J. et al. 1974. *The Uhthoff Quarry Alkali-Carbonate Rock Reaction: A Laboratory and Field Performance Study: Cement Aggregate Reactions*. TRR-525. Transportation Research Board, Washington, D.C.

Swenson, E. G. 1957. *A Reactive Aggregate Undetected by ASTM Tests*. ASTM Bulletin No. 226. Philadelphia.

The first recorded instance of deterioration caused by alkali-carbonate-aggregate reactivity.

Swenson, E. G., and Gillot, J. E. 1960. *Characteristics of Kingston Carbonate Rock Reaction*. Highway Research Board Bulletin No. 275. Washington, D.C.

Symposium on Alkali-Carbonate Rock Reactions. 1964. HRR Report No. 45. Highway Research Board, Washington, D.C.

Walker, Hollis N. 1974. *Reaction Products in Expansion Test Specimens of Carbonate Aggregate: Cement Aggregate Reactions*. TRR-525. Transportation Research Board, Washington, D.C.

Walker, Hollis N. 1978. Chemical reactions of carbonate aggregates in cement paste (Chapter 14), in *Significance of Tests and Properties of Concrete and Concrete Making Materials*. ASTM, Philadelphia.

Includes a bibliography that can be copied and pictures for which the Virginia Transportation Research Council has negatives.

Clinker

Campbell, D. H. 1986. *Microscopical Examination and Interpretation of Portland Cement and Clinker*. Construction Technology Laboratories, Skokie, Ill.

The best the author has seen on cement clinker. Numerous color photomicrographs.

Paste

Powers, T. C., and Brownyard, T. L. 1948. *Studies of the Physical Properties of Hardened Portland Cement Paste*. Research Laboratories of the Portland Cement

Association Bulletin 22. Authorized reprint from *Journal of the American Concrete Institute*, Vol. 43, 1947.

Powers, T. C. 1960. *Physical Properties of Cement Paste*. Research Laboratories of the Portland Cement Association Bulletin 154. Reprinted from *Chemistry of Cement*, Proceedings of the Fourth International Symposium, National Bureau of Standards, Monograph 43, Vol. II, Session V, Paper V-1.

Verbeck, George. 1966. *Pore Structure*. Research Laboratories of the Portland Cement Association Bulletin 197. Reprinted from *Significance of Tests and Properties of Concrete and Concrete Making Materials*. ASTM, Philadelphia.

Air Voids

Backstrom, J. E.; Burrows, R. C.; Meilenz, R. C.; and Wolkodoff, V. E. 1958. Origin, evolution, and effects of the air void system in concrete: Part 2--Influence of type and amount of air entraining agent. *Journal of the American Concrete Institute* 30: 261-272.

Backstrom, J. E.; Burrows, R. C.; Meilenz, R. C.; and Wolkodoff, V. E. 1958. Origin, evolution, and effects of the air void system in concrete: Part 3—Influence of water-cement ratio and compaction. *Journal of the American Concrete Institute* 30: 359-375.

Meilenz, R. C.; Wolkodoff, V. E.; Backstrom, J. E.; and Flack, H. L. 1958. Origin, evolution, and effects of the air void system in concrete: Part 1—Entrained air in unhardened concrete. *Journal of the American Concrete Institute* 30: 95-121.

Meilenz, R. C.; Wolkodoff, V. E.; Backstrom, J. E.; and Burrows, R. W. 1958. Origin, evolution, and effects of the air void system in concrete: Part 4—The air void system in job concrete. *Journal of the American Concrete Institute* 30: 507-517.

These four reports were written during the time that air entrainment was beginning to come into widespread use. There was still much resistance to its use, a lack of belief in its efficacy, and fear that air would lower the compressive strength of the concrete below the specified strength.

Petrographic Methods

Beauchamp, R. H., and Williford, J. F. 1974. Metallographic methods applied to ultrathinning lunar rocks, meteorites, fossils, and other brittle materials for optical microscopy. In *Metallographic Specimen Preparation: Optical and Electron Microscopy*, ed. J. L. McCall and W. M. Muller. New York: Plenum Press.

Beauchamp, R. H.; Williford, J. F.; and Gafford, E. L. 1972. *Final Report, Parts I & II: Exploratory Development and Services for Preparing and Examining Ultrathin*

Polished Sections of Lunar Rocks and Particulates. NASA 9-11993, 211B00862. NASA Manned Spacecraft Center, Houston, Texas 77068. Richland, Washington: Pacific Northwest Laboratories, A Division of Battelle Memorial Institute.

Of these two reports by this team, the first is more readily available and has some very instructive photographs, but the second report has much more detailed information concerning the procedures adapted for use at this laboratory.

Bloss, F. D. 1961. *An Introduction to the Methods of Optical Crystallography.* New York: Holt, Rinehart and Winston.

DeHoff, R. T., and Rhines, F. N. 1968. *Quantitative Microscopy.* New York: McGraw-Hill.

Kerr, P. F. 1959. *Optical Mineralogy*, 3rd ed. New York: McGraw-Hill.

Wilk, W.; Dobrolubov, G.; and Romer, B. 1974. *Development in Quality Control of Concrete During Construction.* TRR 504. Transportation Research Board, Washington, D.C.

The main value of this paper is that it is the source of the idea for using fluorescent illumination to define the difference between empty space and hydration products of HCC (extremely low birefringence and index of refraction.) This paper has many other ideas in it, for example, quick freezing-thawing testing and a numerical scale on which to place each concrete.

Natural Aggregates

Deer, W. A.; Howie, R. A.; and Zussman, J. 1962. *Rock Forming Minerals.* Vols. 1-5. London: Longmans, Green and Co. Ltd.

Dolar-Mantuani, L. 1983. *Handbook of Concrete Aggregates.* Park Ridge, N.J.: Noyes Publications.

This is a definitive and very important work that is recommended for study by anyone who has any control over the selection and purchase of aggregates for use in highway construction.

Williams, H. et al. 1954. *Petrography: An Introduction to the Study of Rocks in Thin Section.* New York: Freeman and Co.

Cements and Artificial Aggregates

Insley, H., and Frechette, V. D. 1955. *Microscopy of Ceramics and Cements.* New York: Academic Press, Inc.

Lea, F. M., and Desch, C. M. 1956. *The Chemistry of Cement and Concrete*. London: Edward Arnold.

Orchard, D. F. 1962. *Concrete Technology. Volume 1: Properties of Materials*. New York: John Wiley & Sons, Inc.

REFERENCES

1. Mather, K. 1978. Petrographic examination (Chapter 11), in *Significance of Tests and Properties of Concrete and Concrete Making Materials*. ASTM Special Technical Publication 169B. (This chapter is about the petrography of concrete.)
2. Meilenz, R. C. 1978. Petrographic examination (Chapter 33), in *Significance of Tests and Properties of Concrete and Concrete Making Materials*. ASTM Special Technical Publication 169B. (This chapter is about the petrography of aggregates.)
3. ASTM Standard Practice for Petrographic Examination of Hardened Concrete, C856, in Vol. 04.02, *Concrete and Aggregates*, *ASTM Annual Book of Standards*. ASTM, Philadelphia.
4. American Concrete Institute. 1987 (updated yearly). *ACI Manual of Concrete Practice*. Detroit.
5. Dolar-Mantuani, L. 1983. *Handbook of Concrete Aggregates*. Noyes Publications, Park Ridge, N.J.
6. ASTM Standard Test Method for Microscopical Determination of Parameters of Air-Void System in Hardened Concrete, C457 in Vol. 04.02, *Concrete and Aggregates*, *ASTM Annual Book of Standards*. ASTM, Philadelphia.
7. Webb, John W. 1970. *The Wearing Characteristics of Mineral Aggregates in Highway Pavements*. Report 70-R7. Virginia Highway Research Council, Charlottesville.
8. Walker, H. N., and Marshall, B. F. 1979. Methods and equipment used in preparing and examining fluorescent ultrathin sections of portland cement concrete. *Cement, Concrete, and Aggregates*, Vol. 1, No. 1, pp. 3-9.
9. Beauchamp, R. H.; Williford, J. F.; and Gafford, E. L. 1972. *Final Report, Parts I & II: Exploratory Development and Services for Preparing and Examining Ultrathin Polished Sections of Lunar Rocks and Particulates*: NASA 9-11993, 211B00862. NASA Manned Spacecraft Center, Houston, Texas. Richland, Washington: Pacific Northwest Laboratories, A Division of Battelle Memorial Institute.
10. Wilk, W.; Dobrolubov, G., and Romer, B. 1974. *Development in Quality Control of Concrete During Construction*. TRR 504. Transportation Research Board, Washington, D.C.
11. Whiting, D. 1981. *Rapid Determination of the Chloride Permeability of Concrete*. Constructions Technology Laboratories, Skokie, Ill.