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16. Abstract

This manual was undertaken to record for all persons wishing to do concrete petrography the petrographic procedures that have been found useful at the Virginia Transportation Research Council.

The manual is made up of an introduction and chapters on equipment, general initial procedures, cracks, preparation of specimens, voids (including determination of the air-void system), percentage analysis of solid components, examination with the stereomicroscope, water-cement ratio, alkali-aggregate reactions, particulate materials, and examination with the petrographic and the polarizing/epifluorescence microscopes. An extensive reading list, glossary, and other appendixes are also supplied.

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FINAL REPORT

PETROGRAPHIC METHODS OF EXAMINING HARDENED CONCRETE:

A PETROGRAPHIC MANUAL

Hollis N. Walker
Research Petrographer

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

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(A Cooperative Organization Sponsored Jointly by the Virginia Department of Transportation and the University of Virginia)

In Cooperation with the U.S. Department of Transportation
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I came to VTRC as a geologist with extensive training and experience in petrography, but I knew almost nothing about concrete. I am grateful to the VTRC for the opportunity to learn about this complex material and work with it for more than 25 years. My knowledge of concrete was gleaned from the literature and patiently imparted to me by Howard H. Newlon, Jr., during the years he was my immediate supervisor. My associations with Samuel S. Tyson, Michael M. Sprinkel, H. Celik Ozyildirim, and other concrete researchers were very informative and helpful.

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LIST OF ACRONYMS

ACI. American Concrete Institute.
ASTM. American Society for Testing and Materials.
BF. Barrier filter.
DM. Dichroic mirror.
GGBFS. Ground-granulated blast furnace slag.
HCC. Hydraulic cement concrete.
P/EF. Polarizing/epifluorescence.
RH. Relative humidity.
SI. International System of Measurements, i.e., metric.
VDOT. Virginia Department of Transportation.
VTRC. Virginia Transportation Research Council.

Chapter 1
INTRODUCTION

1.1 HISTORICAL BACKGROUND

The methods used for examining HCC and related concretes are akin to the methods used by the petrologist and mineralogist for examining naturally occurring ores, minerals, and rocks. The word *petrography* was derived from the Greek words *petros*, meaning "rock" or "stone," and *graphikos*, meaning "written." *Petrography* has come to mean the description and classification of rock by any means—from simply describing the color or form to using highly technical chemical and instrumental methods, such as scanning electron microscopy, x-ray fluorescence analysis, and x-ray diffraction. Petrography is a branch of the science of petrology, which, in addition to description and classification, includes the deciphering of the origin of rocks, study of the relationships between various rock and mineral deposits, study of the effects of various geologic processes, and unraveling of the complex history of rocks.

The petrography of HCC and its aggregates is at once simpler and more complicated 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 usually 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 are very important in determining the causes of the current condition of the concrete being studied.

Most of the people presently doing concrete petrography were originally geologists with formal training in optical mineralogy and petrography. Many of the techniques used in these two fields are also related to the branch of engineering generally called materials science. Many universities have an excellent department of materials science, and entire libraries are devoted to the subject. However, the techniques and skills required for the examination of concrete do not seem to be the subject of intensive study or training at any known institution of higher education—hence, this manual.

1.2 CURRENT PRACTICES

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 various physical forces existing between the ions, atoms, and granules of the material. Because the experienced concrete petrographer can, within a limited amount of time, make a useful assessment of the quality and possible problems associated with HCC, the associated basic science studies that would use instrumental analysis are almost never performed in the course of solving construction problems.

The petrographic examination of HCC is in many respects more qualitative than quantitative. Other than the parameters of the air-void system, the information

used most by the HCC petrographer is the macroscopic appearance of the specimen, appearance of the concrete in the placement, and appearance of variously prepared surfaces of the concrete as viewed with the stereomicroscope. Even the examination of 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.

Generally, the concrete petrographer is consulted only when the material in question has failed to perform properly, has failed a particular physical test, or is suspected to have a serious flaw. For example, concrete from a construction project may be submitted to the petrographer because the compressive strength is lower than required or the air content values determined in the field are questioned. Very commonly, the specimens of HCC are submitted because it is known that the construction procedure was not standard in some way and the engineers wish to discover whether the nonstandard practice adversely affected the durability or appearance of the material produced. For example, if the HCC was placed during a driving rainstorm, the engineers may want to know if the HCC was overwatered or if the surface was weakened by the extra water. If a placement shows cracking soon after construction, the petrographer may be called on to determine the nature of the cracking and speculate as to its cause. If an HCC placement shows distress before its expected life span has elapsed, the engineers may desire a petrographic analysis of the material to determine the cause of the distress. The cause might be any one or more of the various chemical reactions or a stress that is not related to the nature of the material. Only once in my more than 20 years of experience has any HCC been submitted to the VTRC petrography laboratory because the question was "Why is this concrete so good?" This is not a question that can be answered by a petrographer who is not familiar with the many different types of HCC.

After years of experience in examining concrete, the concrete petrographer has usually seen hundreds of pieces of concrete, and his or her memory associates particular appearances of the concrete with the histories of durability or failure that accompanied the specimens. Thus, the concrete petrographer's memory is the data bank against which all new specimens of concrete are compared. It is difficult to transmit this sort of data bank to a petrographer who, although trained in the techniques of optical mineralogy or materials science, has little or no experience in examining HCC and identifying the various features that may indicate the quality of the material.

There is no large collection of reference works on the examination of HCC and no collection of photographs of the features that indicate durable concrete or the ominous signs that indicate probable early failure or premature deterioration. The references on the petrography of concrete are few and lack good photographs that can be compared with the concrete an inexperienced petrographer might be viewing. The main written works on the petrography of concrete are in the applicable publications of the Transportation Research Board (formerly the Highway Research Board), ASTM, ACI, Construction Technology Laboratories (a division of the Portland Cement Association), and the National Research Council of Canada. Some of these works provide detailed instructions for the petrographic examination of concrete or aggregates for concrete. Many mention that the work should be done by people who are qualified by training and experience to operate the microscopes and other equipment used, record 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 people who will be using the petrographic information.

1.3 HOW TO USE THIS MANUAL

This manual was created to provide a set of instructions for the petrographic examination of HCC used in highways, usually those associated with VDOT. It does not discuss the concrete used in floor slabs, walls of buildings, prestressed beams, or other such materials. Included in this list are shotcrete, preplaced aggregate concrete, cinder concrete, and cellular concrete. Concrete with lightweight (either naturally lightweight or artificially expanded) aggregate is discussed briefly in Appendix E. There are many good reference works on the petrography of the constituents of natural mineral aggregates and their use in concrete; this manual discusses the petrography of HCC and the reactions of rocks in HCC, not the petrography of rocks and minerals.

The composition of the instructions was heavily influenced by problems occurring during the construction of highway pavements and bridge decks under conditions where delays could be very costly. No attempt was made to include all the instructions that are available in the literature. Rather, an effort was made to report and suggest ways of performing examinations of HCC that have been refined and developed at VTRC and report any additional instructions and ways of considering a problem that have been found useful at VTRC.

Emphasis is on the procedures possible with simple stereomicroscopes and the necessary sample preparation methods. This manual includes photographs for study by a microscopist who wishes to become highly familiar with the features of HCC. Included also are descriptions of particular features and theoretical discussions of a few features that seem to have been incompletely discussed in the literature. When considered appropriate, certain lines of reasoning that have been developed at VTRC and are not clearly described elsewhere in the literature are discussed. It is hoped that such procedures, instructions, and photographs will be of use to persons who have no specific petrographic training but who have a great familiarity with and a great interest in HCC. Certain features of HCC and aggregate materials are discernible only in thin section using the various procedures possible with the petrographic and P/EF microscopes. Instructions for the use of the petrographic microscope are included when the specialized techniques for the observation of the particular features of HCC differ from the classic, geological petrographic methods. These instructions should lead the reader to an understanding of the value of this microscope and a study of some of the various texts on the subject. Instructions for fabricating thin sections of a specimen for viewing with the petrographic and P/EF microscopes are included when the procedure was developed at VTRC or was not generally described in the literature. Procedures requiring more complex equipment (e.g., x-ray diffraction, differential thermal analysis, atomic absorption spectroscopy) and complex chemical tests are mentioned only if they have been used at VTRC.

Numerous references are cited throughout, and further information is provided in the Reading List. The bibliographies in the works cited and the works in the Reading List provide direction to information and instructions that are not included in this manual.

A glossary is included to provide information concerning the terms used by geologists and concrete technologists. Certain terms are used rather than others because they are compatible with the other publications of VDOT and VTRC.

The client, as referred to in this manual, is generally considered to be someone other than the petrographer. In general, the client is the person, group of people, or organization that has decided on the necessity for petrographic examination. In the case of a petrographer working for a transportation department, the client may be a division of the department, highway engineer, fellow concrete technologist, or fellow researcher. Throughout this manual, the word *client* is used to signify the person or organization making the request for the petrographic examination.

Chapter 2
EQUIPMENT, MATERIALS & ENVIRONS

2.1 OVERVIEW

In this chapter, brands and makes of equipment are not specified; rather, the functions of the equipment and the reasons the equipment is required are described and explained. The successful operation of a petrographic laboratory is dependent on the skill, knowledge, and judgment of the petrographers and petrographic technicians and the quality, sufficiency, and operating convenience of the equipment available to these people. The better the quality of the equipment is, the more efficient and accurate the results of the petrographic examinations will be. Table 2-1 is a list of the equipment needed in a petrographic laboratory. Much of this equipment is demonstrated in Walker (1988).

Table 2-1
EQUIPMENT FOR PETROGRAPHIC LABORATORY

For Sample Preparation Room
- Exhaust system
- Table
- Hood
- Running water
- Sink
- Floor drains
- Water-cooled drill press and diamond-edged drill bits
- Rock trimmer

For Preparation of Slices
- Water-cooled, diamond-edged, rotary saw with an overhand arm
- Large, oil-cooled, diamond-edged, rotary saw
- Diamond-edged trim saw
- Bench lap
- Rotating laps
- Weights
- Safety-approved container
- Ultrasonic cleaner

For Production of Thin Sections
- Thin-sectioning equipment
- Glass plate
- Drying oven
- Vacuum oven
- Set of clamps
- Vibrating lap

continues

Table 2-1 (cont.)

For Examination of Specimens
- Stereomicroscope
- Microtools
- Equipment for determination of air-void parameters
- Petrographic microscope
- P/EF microscope
- Sieves

Expendable Materials
- Lapping oil
- Grinding compounds
- Fluorescent dye
- Impregnating epoxy
- Mounting epoxy
- Flat glass petrographic slides
- Welled glass petrographic slides
- Carpet tape
- Carnauba wax or colorless nylon fingernail hardener
- Pens
- 10% HCl acid
- Notebooks
- Glass trays
- Dropper bottles or rods
- Glass dishes
- Absorbent cotton, paper towels, etc.
- Index of refraction oils
- Disposable 12-in. liners for vibrating lap
- Disposable plastic Petri dishes (60-mm diameter)
- Equipment for specific tests
- Reference specimens

2.2 FOR SAMPLE PREPARATION ROOM

2.2.1 General Comments

The sample preparation room is a dirty room because it is usually the room to which new specimens, complete with any adhering dust or soil, including concrete in all stages of disintegration, are brought and in which loose grinding compounds and lapping oils are stored and used. Water, oil, specimen fragments, mud, and grinding compounds accumulate on the floor and may be tracked to other portions of the building. This room requires periodic cleaning and sorting to make it possible to work in.

2.2.2 Equipment

The following equipment is needed in the sample preparation room:

- **Exhaust system.** For exchanging the air frequently and ridding the room of the chemical fumes and oil vapors produced from cutting, lapping, and cleaning specimens.

• **Table.** For sorting and labeling specimens and preparing them for preparation procedures. Equipment for marking specimens, paper, pens, and pencils should be available at this table.

• **Hood.** For enclosing any area where strong solvents (e.g., acetone, 1,1,1, trichloroethane) and epoxy components are stored and used.

• **Running water.** For cooling and lubricating some of the saws and the drill press. Usually, it is most convenient to have adjustable, mixable, hot and cold water permanently supplied to each piece of equipment. Hoses that drape across the floor and must be reattached to each piece of equipment as it is put into use are a safety hazard and are inefficient. In many cases, the operator's hands must stay in the running water for a long period of time. Thus, although the main purpose of the water is to remove heat from the specimen, it must be possible to adjust the water temperature so that the operator is comfortable and can maintain a firm grip on the specimen being shaped.

• **Sink.** For washing hands and pieces of equipment.

• **Floor drains.** For draining spilled and splashed water. A large sump should be placed under the areas where oil may be spilled.

• **Water-cooled drill press and diamond-edged drill bits** (Fig. 2-1). For fabricating small cores of rock or concrete. Bits should be of internal-diameter sizes required for any tests using small cores of rock. This item will not be required unless the preparation of rock specimens for tests such as those specified in ASTM C 277 is a function of the petrography laboratory. Quantity: 1.

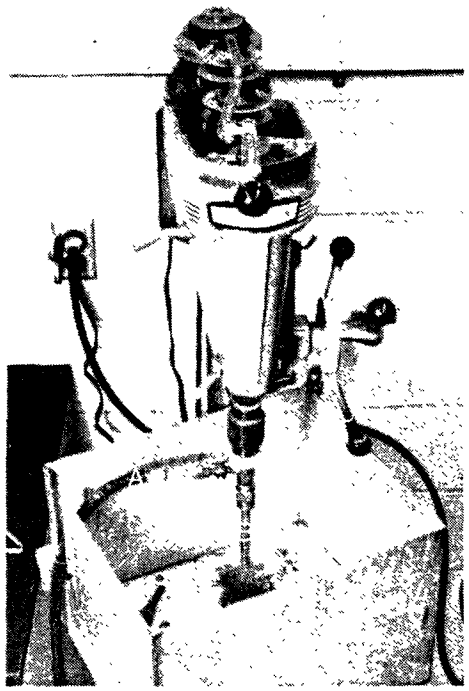


Figure 2-1 WATER-COOLED DRILL PRESS. *A* = water supply hose; *B* = drill bit; *C* = splash guard.

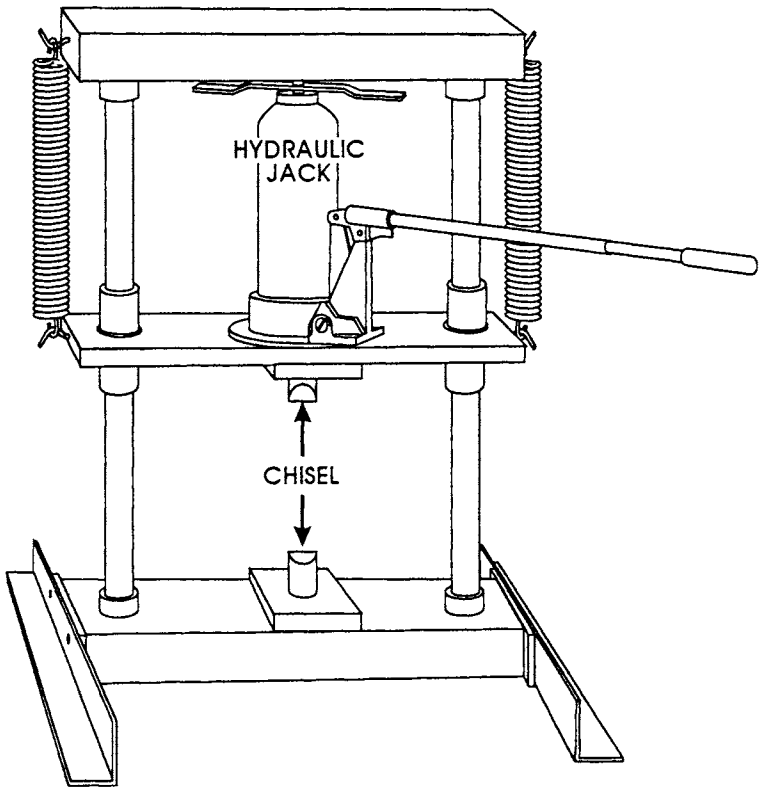


Figure 2-2 ROCK TRIMMER. With chisel points and hydraulic jack to apply up to 5 tons of force.

• **Rock trimmer** (Fig. 2-2). A hydraulic press that by means of opposing chisel points produces controlled cracks in rock, concrete, and similar materials.

2.3 FOR PREPARATION OF SLICES

2.3.1 General Comments

Apparatus and materials for the preparation of surfaces of HCC specimens for microscopical observation are described in ASTM C 856, "Apparatus." Other apparatus may be equally suitable. Water should be used as little as possible on portions of HCC that are to be examined microscopically. Water dissolves certain components of HCC (see 6.3.3). With this in mind, whenever a choice is possible, equipment for sawing concrete that uses an oil bath should be selected rather than equipment that uses running water or a water bath containing a rust inhibitor. The diameters listed for the rotary saw blades reflect the fact that the depth of cuts made with a rotary saw cannot exceed the distance from the supporting central plate to the edge, a distance that is always less than half the diameter of the saw blade. The flatness and accuracy of the saw blade are greatest at the rim, where it moves with the greatest velocity relative to the specimen. Some blades thicken or bow in the middle and cause binding. The polishing wheel mentioned in ASTM C 856 is not required for the production of finely lapped concrete surfaces since lapped surfaces are not polished. Polished surfaces are not desired because they act as mirrors and reflect images at all angles of incidence.

2.3.2 Equipment

The following equipment is needed for shaping and lapping slices:

• **Water-cooled, diamond-edged, rotary saw with an overhand arm** (Fig. 2-3). For reducing large fragments and long cores and cylinders to sizes that may be held in the vise of the next saw. The 14-in. diamond-edged blade is usually slotted into wide teeth. The saw is cooled with running water to allow specimens to be hand held for quick setup and obviate the need for a cover. Both hands are often needed to hold the specimen and roll the table. The foot-pedal control for adjusting the height is a necessity. Quantity: 1.

• **Large, oil-cooled, diamond-edged, rotary saw** (Fig. 2-4). For cutting specimens. The 24-in. (minimum) diamond-edged, smooth-edged blade produces a smooth cut with few ridges and minimizes the difficulty of producing finely lapped surfaces. A vise that holds the specimen firmly and automatically moves it into the saw is an integral part of this equipment. The saw is covered to contain the splashing oil and oil vapors. Quantity: 1 (minimum).

• **Diamond-edged trim saw.** For shaping small specimens of rock or concrete, such as thin-section blanks. The saw in use at VTRC (Fig. 2-5) is water cooled, but an oil-cooled saw is preferable so that specimens are not exposed to water that might dissolve certain phases in concrete. The diamond-edged blade should be 8 in. or less in diameter. Quantity: 1.

• **Bench lap** (Fig. 2-6). For smoothing surfaces of hand-held specimens of rock or concrete. The usual lubricant is water added by hand or dripped from an overhead



Figure 2-3 WATER-COOLED, DIAMOND-EDGED, ROTARY SAW WITH OVERHAND ARM. The 14-in. blade is diamond edged and slotted into wide teeth. The foot pedal controls the height of the saw.

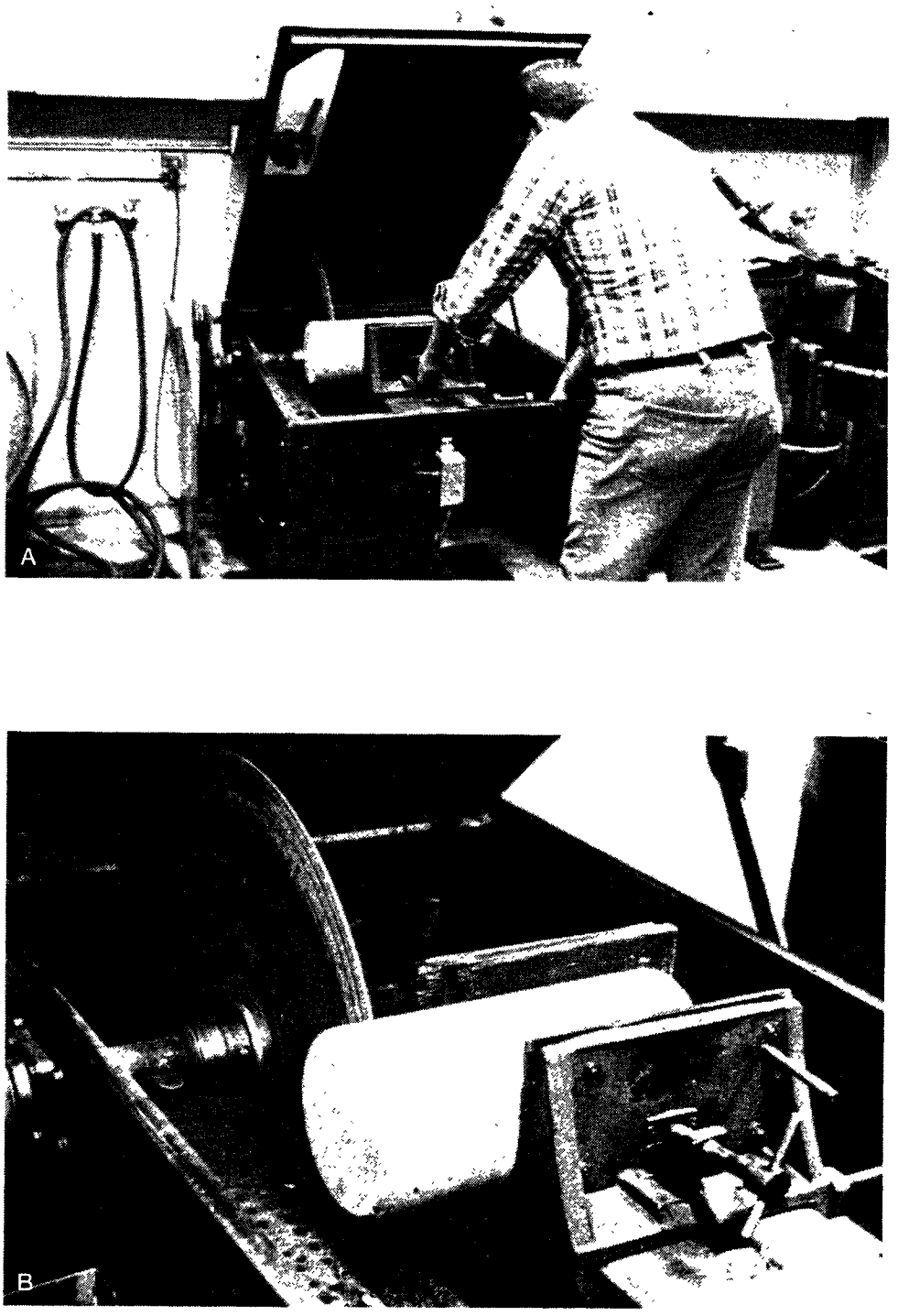


Figure 2-4 LARGE, OIL-COOLED, DIAMOND-EDGED, ROTARY SAW. A. The smooth-edged blade, at least 24 in. in diameter, runs in an oil bath. **B.** Interior vise that holds specimen firmly and automatically moves it into the saw.

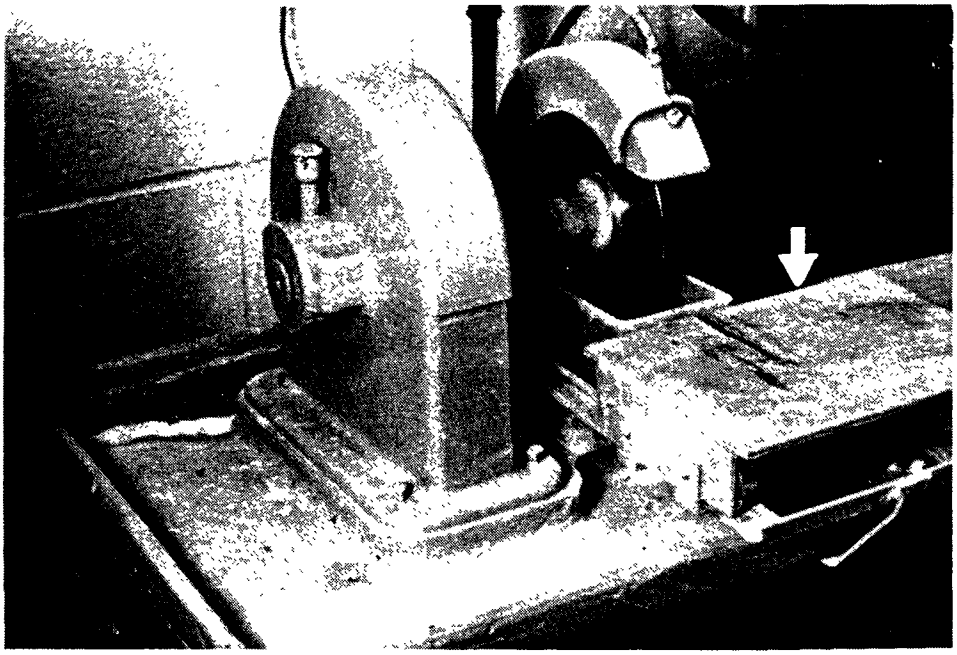


Figure 2-5 ROTARY SAW WITH THIN, DIAMOND-EDGED, SMOOTH-EDGED BLADE. Diameter of 6 in. The *arrow* points to the sliding table on which the specimen can be braced.

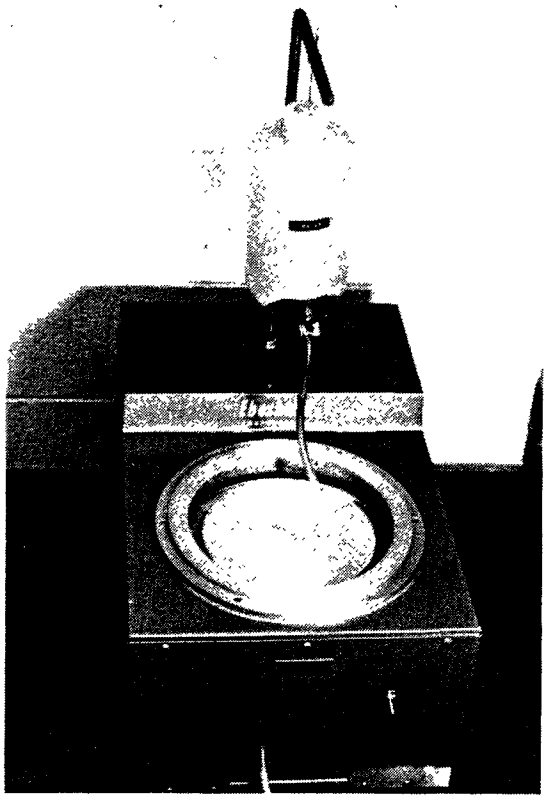


Figure 2-6 BENCH LAP. Diameter of 8 in. For rough grinding rock and concrete specimens. The suspended bottle is for water or another lubricant. Grinding compounds may be shaken like salt from plastic bottles with a pierced cover.

container. The grinding compounds are usually dispensed from small shakers. Quantity: 1.

• **Rotating laps.** For producing finely lapped surfaces for microscopic examination and air-void analysis. The laps should be fabricated of cast iron and radially grooved to provide drainage. The equipment includes an adjustable timer and automatic feed for the slurry of lapping oil and grinding compound. A lap 16 in. in diameter will handle three 4-in. specimens or two 6-in. specimens. The lapping equipment includes slotted, rotating, sample-holding rings (sized to fit common slice sizes), yokes to hold the rings, and cover plates to rest between the slice and its weight. The lapping of concrete or rock slices is time-consuming. Two laps speed up operations. If there is only one horizontal lap wheel, great care must be taken to clean the iron wheel and associated equipment carefully (such as the feeder for grinding compound) whenever a change is made to a finer compound. A very small amount of a coarse grit left in the equipment can significantly damage a lapped surface produced with fine grits. The finest grinding compound should be used on one lap, and all coarser grits on the other. The lap wheels (Fig. 2-7) should be sturdy and have durable bearings to withstand the stresses of unbalanced samples and heavy weights. Quantity: 2.

• **Weights** (Fig. 2-8). For lapping slices smoothly. Weights fit on the back of the slices, loosely inside the rings. The weights press the specimen firmly against the lap to prevent the lapping away of the softer material and leaving the hard material as high spots. The weight increases the wear on the high (hard) portions of the specimen and prevents a buildup of grinding compound under the softer portions. Without the weights, the softer portions will be worn away more deeply. The

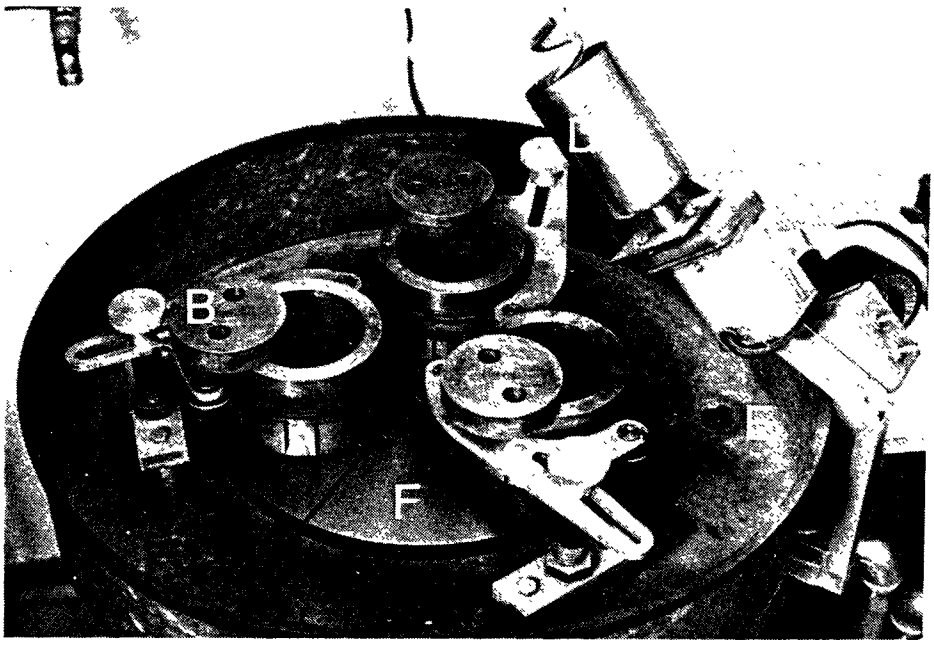


Figure 2-7 LAP. Diameter of 16 in. *A* = rotating slotted specimen holder; *B* = cover plate to set on top of specimen; *C* = guide yoke that retains specimen holder; *D* = grit-slurry cup mounted on its motor and containing spiral agitator-pump; *E* = drain; *F* = grooved lap.



Figure 2-8 WEIGHTS. For lapping slices smoothly. Weights fit on the back of the slices, loosely inside the rings. Here, coffee cans of appropriate diameter weighted with lead shot are used.

weights should cover nearly all the back of the specimen and should weigh 14 to 16 lb for a specimen 4 in. in diameter and 16 to 18 lb for a specimen 6 in. in diameter. Coffee cans of appropriate diameter weighted with lead shot have been found satisfactory. Quantity: The number of specimens it is possible to lap at one time.

• **Safety-approved container** (Fig. 2-9). For cleaning specimens. Specimens are cleaned in solvents, such as acetone or 1,1,1, trichloroethane, to remove oil and grinding compound accumulated during lapping.

• **Ultrasonic cleaner** (Fig. 2-10). For cleaning specimens. The cleaner should be of sufficient size to submerge easily the largest lapped specimen that will require cleaning. The cleaner bath must be of a material that can hold acetone or 1,1,1, trichloroethane. Quantity: 1.

CAUTION: 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 with the specific frequencies of the equipment (ASTM C 457).

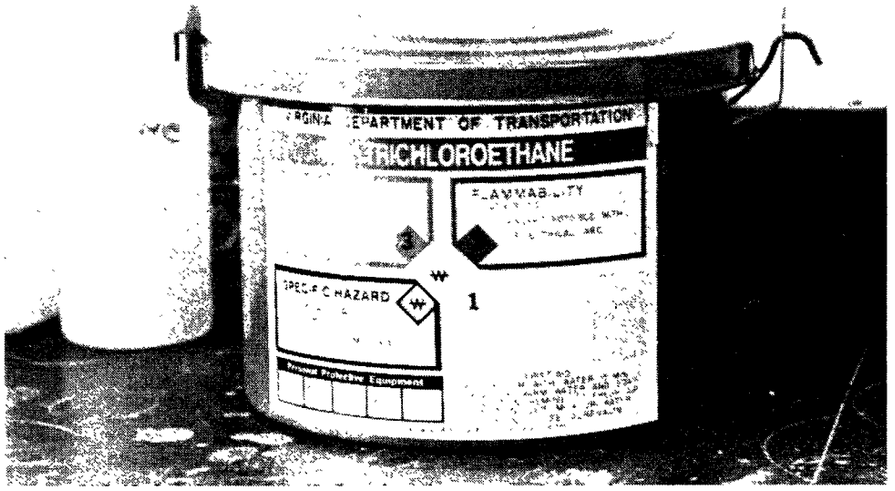


Figure 2-9 SAFETY-APPROVED CONTAINER FOR CLEANING OF SPECIMEN

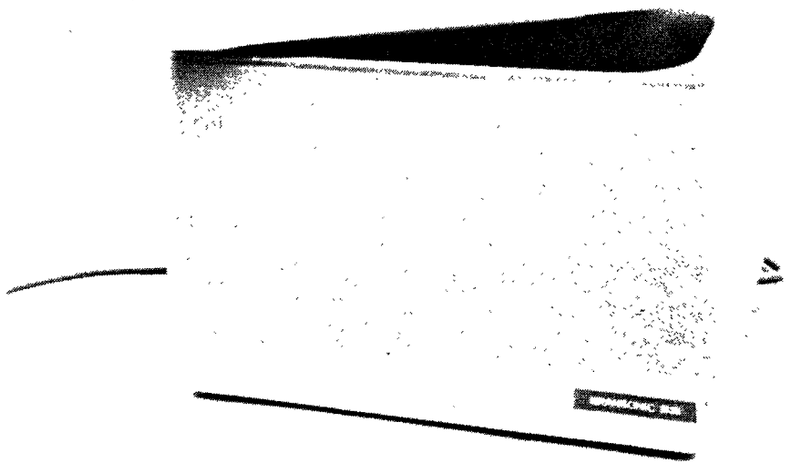


Figure 2-10 ULTRASONIC CLEANER. The tank is of a material that will resist most solvents.

2.4 FOR PRODUCTION OF THIN SECTIONS

2.4.1 General Comments

To fabricate a specimen chip of concrete or rock that will fit the glass that will be used to support the thin section will usually require the use of at least one of the two large saws and the small trim saw. The room that is used for the further fabrication of thin sections should be isolated from the operations of the preparation of lapped slices and requires an efficient exhaust system or two hoods because the cutting and grinding of thin sections to the required thickness produces an oil vapor and the production of thin sections requires the use of epoxy (toxic in large quantities).

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 of Battelle, Richland, Washington, recommended the use of a belt sander and a bench lapping machine with diamond-impregnated laps (Beauchamp, Williford, & Gafford, 1972). Labor fur Praparation and Methodik, Beinwil am See, Switzerland, recommended the use of a diamond-tooled milling machine (Wilk, Dobrolubov, & Romer, 1974). VTRC has Ingram-Ward thin-section machines.

2.4.2 For Examination with Petrographic Microscope

• **Thin-sectioning equipment.** For producing thin sections of rock and similar materials for examination with the petrographic microscope. This manual describes the use of Ingram-Ward equipment (Fig. 2-11). The instructions can probably be adapted for use with any type of thin-sectioning equipment that permits variability in section thickness and thus will make possible ultrathin and extra-thick sections. Part A of the set is a rotating cut-off saw with a very thin blade used to slice off excess specimen material, thus reducing the specimen chip (mounted on a glass slide and held in a vacuum chuck) to 50 to 30 μm in thickness. Generally, the thinner the slice of specimen chip remaining on the glass slide, the better. Part B of the set holds the specimen in another similar vacuum chuck and permits it to be moved carefully and evenly over a rotating, diamond-bearing, cupped, ceramic grinder to grind it to nearly the desired thinness. 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. The lubricant (denatured kerosene with 1/10th part motor oil) is collected in the surrounding tank and pumped to the top of the saw or wheel to cool and lubricate the work. Quantity: 1 set.

• **Glass plate firmly supported in a flat pan** (Fig. 2-12). For catching spilling slurry when thin sections are hand lapped after they have been cut and ground on the Ingram-Ward equipment. Ordinary window glass is usually sufficient. Used for final grinding of ordinary and intermediate grinding of fluorescent ultrathin sections for use with the petrographic and P/EF microscopes. Quantity: 1.

• **Drying oven** (Fig. 2-13). For evaporating water or lapping oil from slices, thin sections, or rock fragments and curing the epoxy of specimens impregnated for thin sections. Vented to allow the escape of vapors. A thermometer is mounted near the center of the oven. Steam must not form in the HCC; therefore, the temperature in the oven must be thermostatically controllable (adjustable to less than 100°C). Quantity: 1.

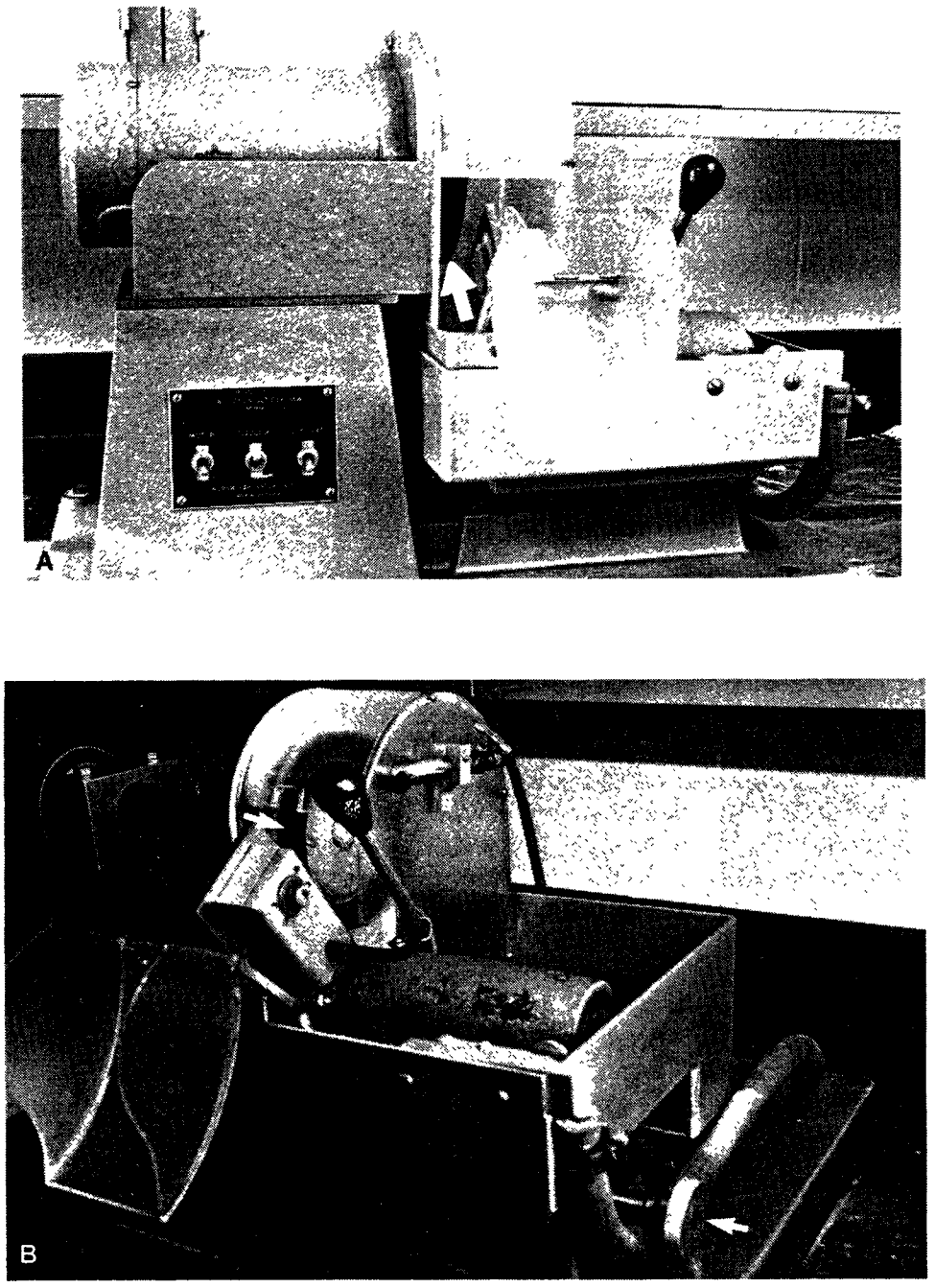


Figure 2-11 INGRAM-WARD THIN-SECTIONING EQUIPMENT. A. Diamond-edged cut-off saw with very thin blade (see *arrow*) used to slice off excess specimen material. **B.** Diamond-bearing, cupped ceramic grinder (see *top arrow*). The handle and chuck permit the specimen to be moved carefully and evenly over the rotating, diamond-bearing, cupped, ceramic grinder. The relative position of the chuck to the saw or grinder can be adjusted with a small dial (see *bottom arrow*) at the right-hand end of each piece of equipment.

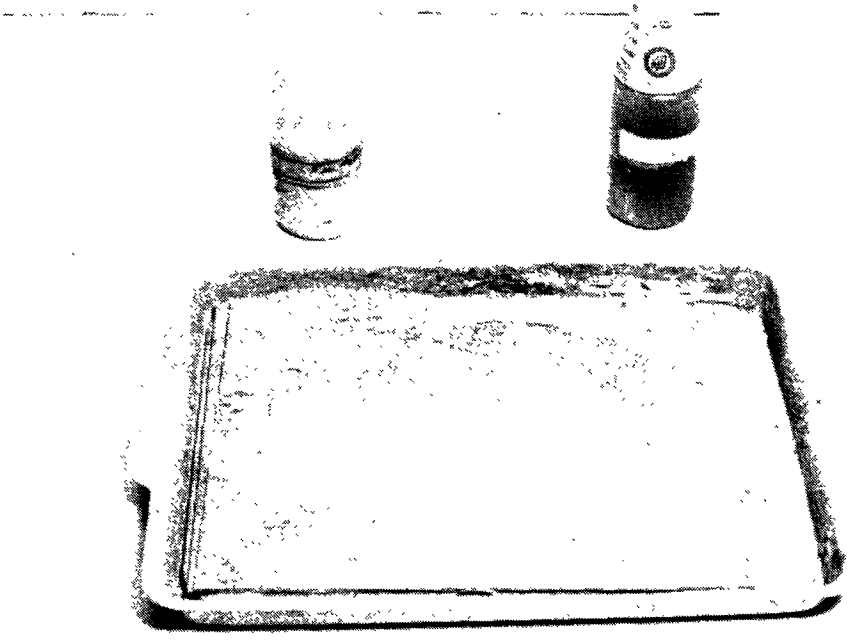


Figure 2-12 GLASS COATED WITH GRINDING COMPOUND SLURRY. The pan used to catch the grinding slurry, which overflows the edge of the glass. The two plastic bottles have holes in the screw-on covers and waxed paper cups for dust covers. The bottles are used for grit and lubricant slurries.

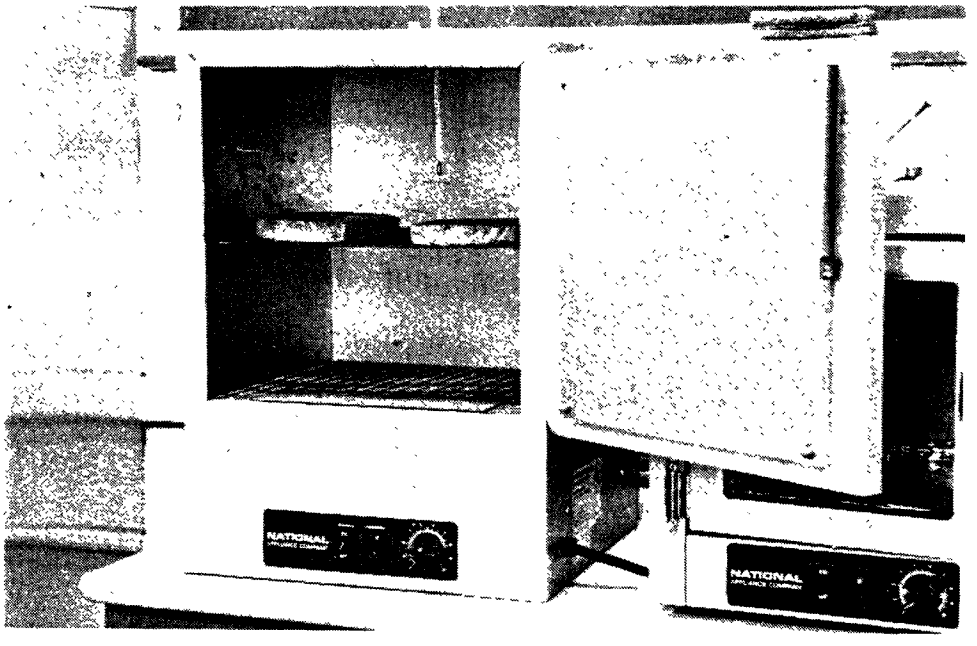


Figure 2-13 DRYING OVEN. Thermostatically controlled.

2.4.3 For Examination with Polarizing/Epifluorescence Microscope

• **Vacuum oven** (Fig. 2-14). For vacuum impregnating thin-section stock with epoxy and slow, even drying of specimens. For fluorescence, fluorescent dye is added to the epoxy. Quantity: 1.

• **Set of clamps mounted on a sturdy metal plate** (Fig. 2-15). For cementing an impregnated thin-section chip to the final, welled, glass slide. Quantity: 1.

• **Vibrating lap with weights** (Fig. 2-16). For producing the final surface of fluorescent ultrathin sections. The thin sections are attached to the bottom of the weights. The circular pan is lined with Pellon PaN-W, a specially formulated lapping pad, which is coated with diamond paste thinned with a thin oil. Quantity: 1.



Figure 2-14 VACUUM OVEN. A. With thin-section stock in liquid dyed epoxy in a small disposable dish. Door will not stay shut without the vacuum. **B.** Close-up of bubbling of liquid epoxy as vacuum removes air and water from specimen.

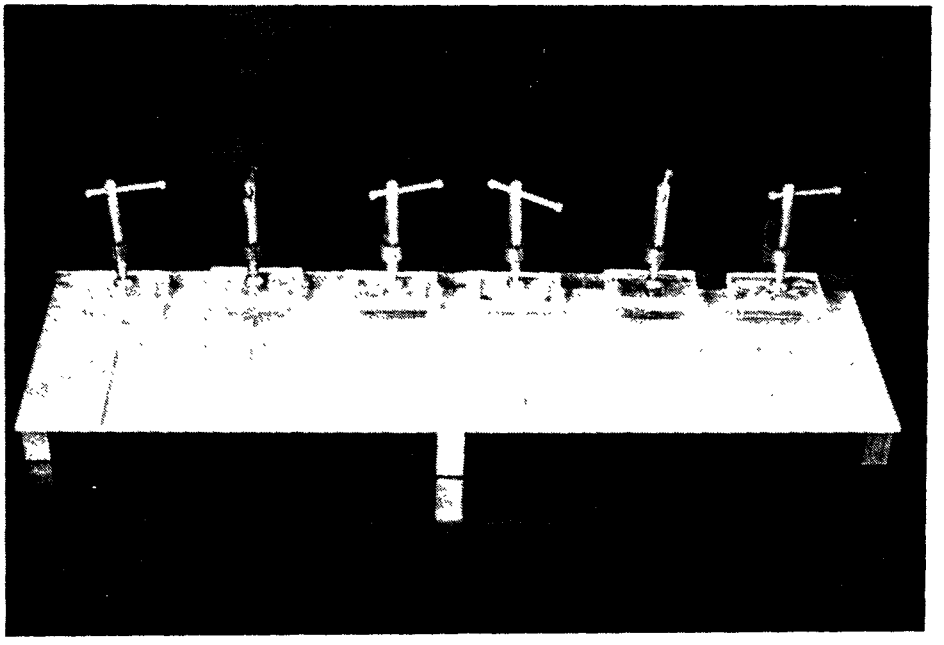


Figure 2-15 MOUNTED SET OF CLAMPS. The little rectangles of lucite are used to distribute stress (Walker & Marshall, 1979).

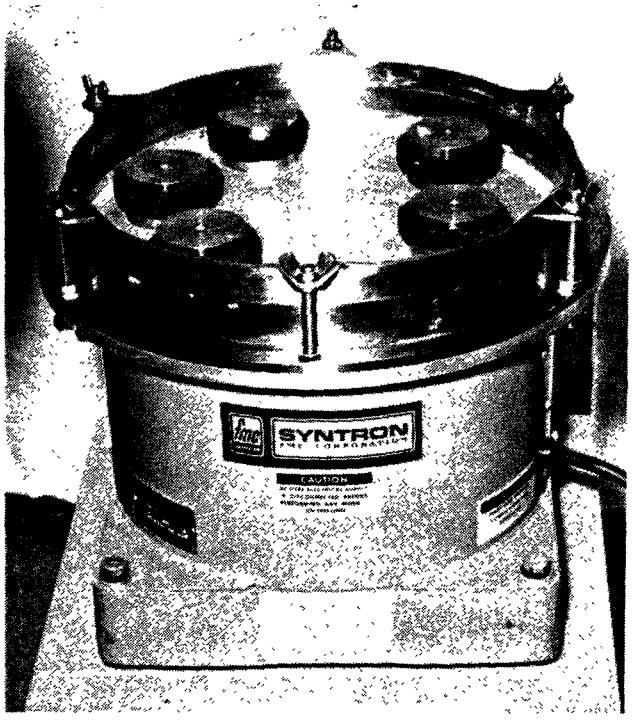


Figure 2-16 SYNTRON VIBRATORY POLISHER AND WEIGHTS. Complete with sponge-rubber bumpers (Walker & Marshall, 1979).

2.5 FOR EXAMINATION OF SPECIMENS

2.5.1 General Comments

The room in which specimens are examined is also where the microscopes are stored and used and should be as close to a "clean" room as can be managed without the use of special clothing. A worker whose hands or clothing is contaminated with grinding compounds, excessive dust, lapping oil, grease, soil, or materials used to mix concrete should remove the contaminants from his or her person before approaching a microscope. It is best if the air in the room is under positive pressure and an anteroom is equipped with an exhaust system to create the concomitant negative pressure. The room should be furnished with tables and microscope benches and a number of adjustable-height stools and chairs.

2.5.2 Equipment

• **Stereomicroscope** (Fig. 2-17). For conducting a general examination of specimens. May also be used in the quantitative determination of air-void parameters. This is the instrument most used by concrete petrographers. The stereomicroscope should have a magnification variable between 25 and 120 diameters. The higher magnifications are attainable with a 2X accessory lens below the objective. It is most convenient to have a zoom objective, but excellent observations can be made with turret-mounted objectives. It is very tedious and annoying to have to change objectives by dismounting them and remounting them. The eyepieces should be the wide-field type. A measuring reticle in one eyepiece can be convenient (must be cal-

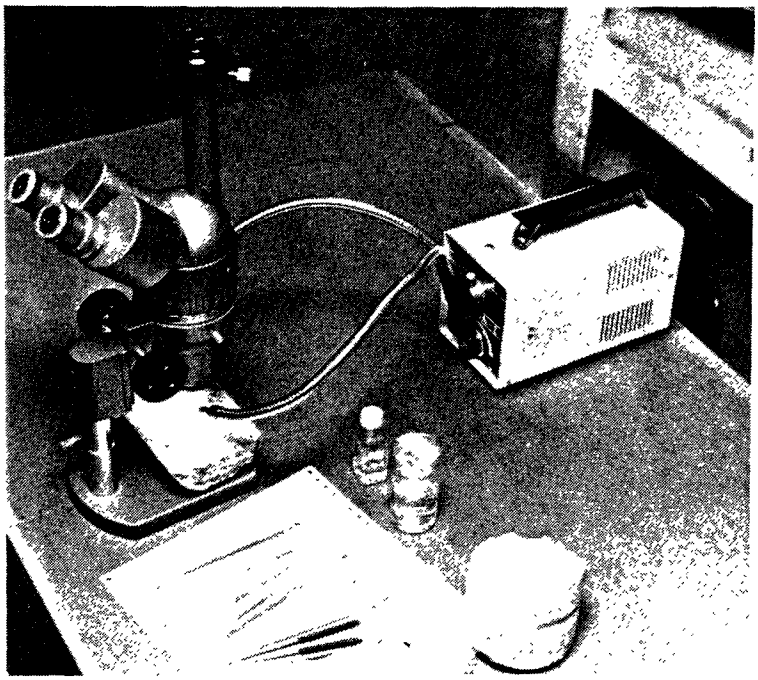


Figure 2-17 STEREOMICROSCOPE WITH LIGHT SOURCE AND ACCESSORIES

ibrated for each magnification). By the use of this microscope, decisions are made concerning concrete quality and the planning of further testing. If there is a difference in the quality of the microscopes available, the one with the least distortion and the widest field of view should be the one used in the quantitative determination of air-void parameters. Quantity: at least 2.

• **Microtools.** For measuring, manipulating, and removing portions of slices of concrete and manipulating the grains of grain mounts under any kind of magnification. The most useful microtools will probably be ones that have been fabricated in the petrographic laboratory (Fig. 2-18). Sets of microtools are commercially available, but they often do not meet the needs of the concrete petrographer. The best sets have a variety of needles, shovels, and scrapers. The tiny tools must be small enough to use in the cracks, crevices, and voids of concrete but must be strong and resilient to resist breaking. Some of these sets are constructed with the microtools as detachable pieces that screw or clamp into a handle. Such sets should have more than one handle. The needles used for biological dissection are usually strong enough, but the point thickens so rapidly that the tool may not be of use in tight cracks and voids. Sewing needles with a handle of a section of a small dowel rod can be very useful. They are strong and sharp and come in a variety of sizes. Often, what is required is an assortment of needles with bends of different radii, well within the working distance of the microscope, to allow the needle to attack the concrete from a variety of angles. Such needles are commercially available for biological microscopy but may not be strong enough to pick out reaction products and small pieces of aggregate from concrete. Duplicate tools should be obtained so that breakage will not cause a delay in the progress of the work.



Figure 2-18 MICROTOOLS. These include needles, shovels, and scrapers small enough to be useful in the cracks and crevices of concrete and fit in the working distance under the stereomicroscope. The *arrow* indicates a small scale engraved on very thin, flexible metal attached to a thin rod that is attached to a handle. The scale has 5 mm marked into tenths of a millimeter on one side and 1 tenth of an inch marked into 5 thousandths of an inch on the other.

The most unreplaceable commercially available microtool is a small scale engraved on very thin flexible metal attached to a thin rod, which is attached to a handle. The scale may have 5 to 10 mm marked into tenths of a millimeter and 1 to 2 tenths of an inch on the other side marked into 5 thousandths of an inch. The flexibility allows better positioning of the scale. The thinness allows the scale to be more nearly in the same focal plane as the item examined. Unfortunately, the solder joint between the flexible metal and the stiff rod connection to the handle may be very fragile. It is good to have a replacement scale available. For less precise measurements, a transparent scale, usually SI, used by the microscopist viewing through the scale with the markings down on the concrete (to allow best focus) is useful. Such scales may be found in stationery and variety stores.

Excellent picks, miniature knives, shovels, and scrapers can usually be obtained from most dentists if a request for used tools is made. These tools are strong and designed for approaching the work area in close quarters. They may require sharpening and reshaping to suit the needs of the concrete petrographer. For this purpose, a stick or two of abrasive dressing compound are useful. These tools may corrode in the presence of chemicals and must be kept clean and sharp. Quantity: minimum of 1 each, many items.

- **Equipment for air-void determinations.** This equipment is described in Chapter 6.
- **Petrographic microscope.** This microscope is described in Chapter 12.
- **P/EF microscope.** This microscope is described in Chapter 13.
- **Sieves** (Fig. 2-19). For sorting aggregate particles (pebbles, sands, and crushed fragments) by size and preparing specimens for microscopic examination. Sieves should comply with the requirements of ASTM E 11. Two sizes of frames are required: 8 in. and 3 in. Both sets of sieves should include standard covers and pans. The 8-in. size is used for sizing and sorting aggregate material to determine compliance with the client's specifications and prepare aggregate specimens for various testing procedures. The 8-in. set should include the mesh sizes necessary to determine the compliance of an aggregate with the client's specifications. If other laboratories that are concerned with aggregates are on the premises, this size of sieve will probably be available. The 3-in. size is used to prepare sands and rock and concrete fragments for grain mounts or other microscopic studies. The 3-in. set should include the mesh sizes of the larger that are smaller than 3/8 in. (9.5 mm) and an assortment similar to the following:

VDOT Designation	ASTM (SI) Designation
No. 30	600 μm
No. 40	425 μm
No. 60	250 μm
No. 100	150 μm
No. 200	75 μm
No. 400	38 μm

- **Other equipment.** Various instrumental methods of analysis such as x-ray diffraction, elemental analysis by x-ray dispersion, scanning electron microscopy, and certain wet chemical tests may be employed in the identification of the raw materials and reaction products and deleterious substances that may be associated with

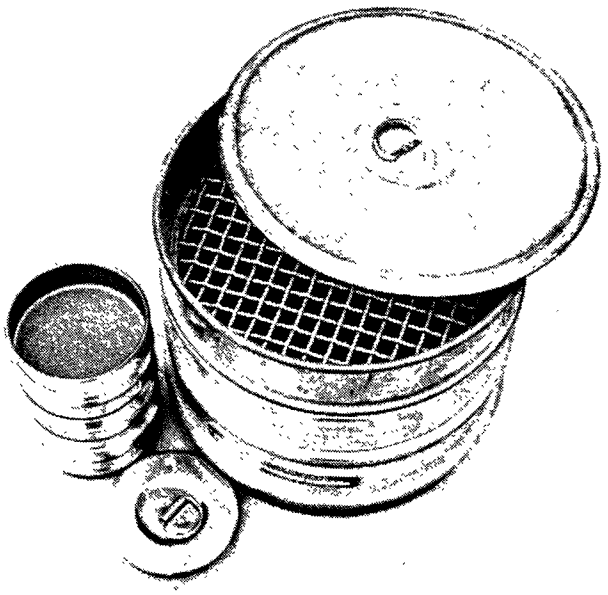


Figure 2-19 SIEVES. Types of sieves used in examination of concrete and concrete materials, 8-in. and 3-in. sizes.

HCC. Each of these methods requires careful work; detailed study; long practice; usually a wide assortment of reference books, charts, and reference samples; and often very expensive equipment.

2.6 EXPENDABLE MATERIALS

2.6.1 General Comments

WARNING: 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, NW, Washington, DC 20009, or to the Materials Safety Data Sheet that should be obtained from the supplier of the particular compound.

2.6.2 Lapping Oil

Water should not be used on any surface of an HCC specimen that is to be examined under magnification. The lubricant used for cutting and lapping specimens should be low-viscosity, water-free lapping oil, such as denatured kerosene mixed with 1/10th part motor oil. The motor oil lessens the production of vapors from the kerosene. A number of light oils are sold specifically as lubricants for cutting with diamond saws. One example is the odorless, stainproof, diamond-saw lubricant sold by Lapidabrade, Inc., Havertown, Pennsylvania. Specialized lubricants are required

for grinding and polishing thin sections with diamond grinding compounds. One of these might be Formula C from the Glennel Corporation, Chester Springs, Pennsylvania.

Oil-based lubricants will not dissolve reaction products and secondary minerals as readily as will water-based lubricants. It is convenient to use a lapping lubricant that will evaporate and leave the surface of the specimen oil free. At magnifications such as are used in the analysis of concrete, an oil film can obscure the exact edges of voids and aggregate particles. The evaporation of the oil may be hastened by treatment in a warm (<70°C), not hot, drying oven. High heat can crack the concrete specimen or heat the oil to its flash point. The oil drained from the laps may be reused after filtering and decanting.

2.6.3 Grinding Compounds

For preparing finely lapped surfaces on concrete specimens. The grit numbers and sizes listed are given only as a guide. The grinding compounds produced by one manufacturer may vary from those produced by another.

- **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 abrasive**
- **0.3-μm Linde,** an aluminum-oxide grinding compound. For using in the epoxy of impregnated thin sections to prevent excess wearing of the epoxy-rich areas of ultrathin sections. The distinctive particle shape will not be confused with any ingredient of concrete or aggregate.
- **oil-soluble diamond paste compounds,** heavy concentration (those from Penn Scientific Products have been found satisfactory)

  —4–8 μm, U.S. Bureau of Standards Grade No. 6, indigo colored

  —2–4 μm, U.S. Bureau of Standards Grade No. 3, violet colored

  —0–2 μm, U.S. Bureau of Standards Grade No. 1, white colored

2.6.4 Dyes and Epoxies

• **Fluorescent dye.** For inclusion in the impregnating epoxy of ultrathin sections to create fluorescence in the voids and cracks. This is available only as samples (believed to be a paint ingredient). The dye presently used is Fluorol Yellow 088, available as samples from BASF Wyandotte Corp., Holland, Michigan. This dye dissolves completely in epoxy; no dye particles can be seen at 600X magnification, and the epoxy is evenly colored by the dissolved dye.

• **Impregnating epoxy.** For strengthening thin-section stock and carrying fluorescent 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 properly cured, the hardened epoxy is not soluble or softened in oil, alcohol, acetone, or 1,1,1, trichloroethane.

- **Mounting epoxy.** For attaching the smoothed thin-section stock to the final supporting glass slide. The mounting epoxy presently used can be either Epoxide from A.B. Buehler, Lake Bluff, Illinois, or Section-Lok epoxy mounting medium from Microtech Engineering Laboratory Inc., Clifton, Colorado. Of the two, the Section-Lok product has the longer shelf life.

2.6.5 Miscellaneous Supplies

- **Flat glass petrographic slides** (27 by 46 mm). For mounting thin sections and grain mounts for examination with the petrographic microscope. A standard item in most scientific catalogs. The slides used for temporary grain mounts can be cleaned and reused.

- **Welled glass petrographic slides** (27 by 46 mm). For mounting ultrathin section chips. The edge of the well provides some protection from the tearing stresses of the procedures (A.B. Buehler Co., Lake Bluff, Illinois).

- **Carpet tape** or other strong, double-sided tape. For attaching thin sections to the bottom of weights.

- **Carnauba wax**, or colorless nylon fingernail hardener. For impregnating fragile concrete.

- **Pens.** For fine marking concrete slices. The pen used should be such that when the inky point is positioned on a crack a very small portion of the ink will flow into the crack and be drawn along the crack and will thus indicate the next direction of the crack.

- **10% hydrochloric acid.** For etching entire slices or portions of slices.

- **Notebooks.** For recording observations.

- **Glass trays.** For etching or other chemical treatment of finely lapped specimens.

- **Dropper bottles** or rods. For dropping acid and water.

- **Glass dishes.** For using in chemical tests.

- **Absorbent cotton,** tissues, paper towels, etc.

- **Index of refraction oils.** For determining the optical properties of minerals and other translucent substances with polarized light microscopy. The oils should range in index from 1.40 to 1.50 in steps of 0.04, from 1.500 to 1.600 in steps of 0.002, and from 1.600 to 1.700 in steps of 0.01. Index oils change with age and must be checked periodically with a refractometer or standard solids.

- **Disposable 12-inch liners for the vibrating lap.** Pellon PaN-W, polishing pads.

• **Disposable plastic Petri dishes,** 60 mm in diameter, 20 mm in depth. For potting thin-section specimens in fluorescent-dyed epoxy.

• **Equipment for specific tests.** Examples are the concrete molds and the length comparators required by certain ASTM procedures. The equipment required for the uranyl-acetate fluorescence test for the presence of alkali-silica gels is listed in 10.2.3.

• **Reference specimens.** For permitting comparisons between materials under study and carefully labeled and documented specimens in a reference collection. Two general kinds of materials should be included in such a collection: (1) raw materials from which concrete is fabricated, and (2) various kinds of HCC in both rough fragments and finely lapped slices. If storage space is limited, photographs may have to be substituted for the larger specimens. Ideally, enough of each specimen should be available for various destructive testing procedures. Some of the items to be included are listed in Tables 2-2 and 2-3.

Table 2-2
REFERENCE SPECIMENS OF MATERIALS USED IN FABRICATING HCC

Aggregates
- Coarse
- Fine
- Those are known to have caused problems (e.g., alkali-reactive aggregates, aggregates that create a high water demand)

Particulate Admixtures (in moisture-resistant containers)
- Fly ash
- GGBFS
- Silica fume
- Other

Types of Cement Approved for Use (in moisture-resistant containers)
- Portland cement
- Portland cement blended with slag, fly ash, etc.
- Other cements, such as GGBFS

Note: Specimens should include as many types of each as are available locally. They should be labeled as to source and type and stored in transparent containers.

Table 2-3
REFERENCE SPECIMENS OF VARIOUS CONDITIONS OF HCC

1. Each of the materials listed in Table 2-2 used in the proper manner in hardened HCC mixtures
2. Materials used in varying amounts:
   - Several lapped slices of HCC with a high water-cement ratio
   - Several lapped slices of HCC with a normal water-cement ratio
   - Several lapped slices of HCC with a low water-cement ratio
   - Lapped slice of HCC with a normal amount of visible cement ferrites
   - Lapped slice of HCC with a less than normal amount of visible cement ferrites
3. Various forms of distress (in large fragments) according to occurrence in the area and accompanied by photographs of the surface condition:
   - Examples of deterioration due to lack of resistance to freezing and thawing (include layered scaling and D-cracking due to the lack of a sufficient air-void system)
   - Examples of deterioration due to an alkali-aggregate expansive reaction
   - Examples of concrete deteriorated by D-cracking due to aggregate susceptibility
   - Examples of plastic shrinkage cracking (including specimens with lapped surface cutting across the cracks)

Chapter 3
GENERAL PROCEDURES

3.1 OVERVIEW

Clients submit a specimen to the petrographic laboratory for a variety of reasons. Some of these reasons are listed in Table 3-1. The different types of specimens that may be submitted are listed in Table 3-2. For HCC, the word *specimen* is used and the word *sample* is usually avoided because the specimen is seldom a truly representative sample of the HCC placement. Clients may also submit a suite of related specimens. For the sake of brevity, I use the word *specimen* even when a suite of specimens is meant.

Table 3-1
REASONS PETROGRAPHIC SERVICES ARE REQUESTED AND CORRESPONDING PLANS FOR ANALYSIS

- **Situation:** Concrete submitted failed test for required compressive strength. **Client's question:** What makes this concrete so much weaker than it was designed to be? **Preliminary plan of analysis:** (1) Perform analysis of air-void system to determine if excess air is major cause of low strength. (2) Perform general examination with stereomicroscope with emphasis on paste quality and aggregate-paste bond.

- **Situation:** Mixture was harsh, and workability was very poor. **Client's question:** Why? Is the coarse aggregate too large, of a difficult shape, or both? Does the aggregate have abundant reentrant angles and angular particle shapes? Was the void content of the fine aggregate within specifications? **Preliminary plan of analysis:** (1) Perform general examination with stereomicroscope with emphasis on size, shape, and lithology of aggregates and the quantity of paste. (2) Consult with client and any involved testing laboratories to acquire test results on aggregates or obtain samples of aggregates for testing.

- **Situation:** Inspector reported that contractor added water because workability was poor. **Client's question:** Did the contractor add too much water? Did he or she add it too late? Was it mixed in properly? **Preliminary plan of analysis:** (1) Perform analysis of air-void system. (2) Perform general examination with stereomicroscope with emphasis on variations in paste color and quality throughout mass, condition of aggregate-paste bond, and distribution and size of air voids.

- **Situation:** Inspector reported that tines used for surface texturing seemed to tear concrete surface rather than produce usual continuous lands and grooves. **Client's question:** Why? What is the depth of damage? **Preliminary plan of analysis:** (1) Perform general examination with stereomicroscope with emphasis on size and shape of aggregates and condition of paste. (2) Prepare several lapped surfaces that are at right angles to both wearing surface and grooves for examination of depth of damage. Consider using special preparation methods (see 5.3.3).

- **Situation:** Inspector reported that vibrating screed failed to operate and the concrete was hand screeded. **Client's question:** Was the quality of the concrete adversely affected? Was the consolidation sufficient? **Preliminary plan of analysis:** (1) Perform analysis of air-void system, including sorting voids encountered into entrained, entrapped, and irregular. (2) Perform general examination with stereomicroscope with emphasis on paste quality.

continues

Table 3-1 (cont.)

- **Situation:** Air-meter test showed that mixture had low air content, but contractor placed mixture anyway. **Client's question:** Is the air-void system sufficient to provide durable concrete able to resist freezing and thawing? **Preliminary plan of analysis:** (1) Perform analysis of air-void system. (2) Perform general examination with stereomicroscope.

- **Situation:** There was a driving rainstorm during placement of concrete. **Client's question**: Is the concrete overwatered? Depth of damage? Durability of the surface texture? Ability of the surface to resist abrasion? **Preliminary plan of analysis:** Perform general examination with stereomicroscope of several lapped surfaces that are at right angles to both wearing surface and grooves to examine depth of damage and quality of paste and check air-void distribution, size, and stability. Consider using special preparation methods (see 5.3.3).

- **Situation:** Specimens are from a test mixture employing a new material or construction practice. **Client's request:** General petrographic examination and a report on anything unusual. **Preliminary plan of analysis:** (1) Perform analysis of air-void system. (2) Perform general examination with stereomicroscope.

- **Situation:** A crack system is developing in a pavement (or bridge deck) soon after placement. **Client's question:** Is this plastic shrinkage cracking? Why did it happen? **Preliminary plan of analysis:** Perform careful examination of specimens according to recommendations and suggestions in Chapter 4. A visit to the site and additional specimens may be required. *Note:* The data necessary to answer the "why" part of this question cannot be found in the petrographic laboratory but must be sought in the inspector's notes, an analysis of the weather conditions, and the recorded observations of any people who passed by the site.

- **Situation:** Placement developed a severe crack system over many portions of the surface. Deterioration is becoming worse at an ever-increasing rate, and concrete is requiring constant work from maintenance crews. [Obviously, the younger the placement, the more mystifying and crucial the problem. But, even when a pavement's life has surpassed its planned life cycle, the need for extensive repairs can seem to be a disaster to a transportation department's budget.] **Client's question:** What is causing this problem? How bad is it going to get? What can be done to stop the deterioration? **Preliminary plan of analysis:** (1) Identify aggregate lithologies. Thin sections may be required. (2) Perform careful examination of specimens according to recommendations and suggestions in Chapter 10. (3) Inspect placement to try to determine extent of use of materials present in deteriorated areas and advise client on number and location of specimens required. (4) Obtain copy of chemical analysis of cement batch used if alkali-aggregate reaction is likely cause. *Note:* If the deterioration is due to an alkali-aggregate reaction, the client should be advised that the deterioration may proceed to complete destruction and that there is no known way to stop it. In the case of an alkali-silica reaction, the same materials can be used in the future if sufficient pozzolanic material or GGBFS is included in the mixture.

- **Situation:** Shortage of a material from one source necessitated use of an unapproved material. **Client's question:** Is there anything wrong with the concrete? [Make sure that the concrete fabricated with the approved material is available for comparison with the concrete fabricated with the substitute material.] **Preliminary plan of analysis:** (1) Perform analysis of air-void systems of the two concretes. (2) Perform general examination with stereomicroscope of the two specimens.

- **Situation:** Concrete telephone conduit (or drainage ditch lining) in contact with soil was distressed by some mechanism that seems to be dissolving it. **Client's question:** What is happening? **Preliminary plan of analysis:** (1) Perform general examination with stereomicroscope with emphasis on aggregate type and presence of minerals that may produce acid during weathering. (2) If no acid-producing agents found in concrete, perform petrographic examination of soil and nearby rock outcrops and chemical analysis for contaminants of soil and ground water.

continues

Table 3-1 (cont.)

- **Client's question:** What is this slick-looking coating (or this dark coating or this white coating) that we find near the cracks in the placement? Does it indicate serious problems? **Preliminary plan of analysis:** (1) Determine composition of exudation. Often, this may be accomplished by examination of grain-mount preparations using petrographic microscope. (2) Request specimens that will include a full specimen of concrete (a depth of 5 or 6 in. is usually sufficient). (3) Perform general examination of specimen of full concrete with stereomicroscope with emphasis on aggregate types and any possible reaction products.

- **Client's question:** Why is this concrete such a funny color? Does it mean the concrete was made from the wrong materials? How durable is this sort of concrete? **Preliminary plan of analysis:** (1) Obtain copy of design of mixture. (2) Explain to client how certain materials produce different colors.

- **Client's question:** Why is there a peculiar dark blue-green color so unevenly distributed in this concrete? Does it mean that the mixing was not sufficient? Are there weaker and stronger places in this concrete? [Suspected: GGBFS was used in the mixture.] **Preliminary plan of analysis:** (1) Inquire if GGBFS was included in mixture or examine thin sections to check paste for presence of GGBFS. (2) Explain to client that dark color exists in portions of concrete that retain original moisture and that it fades unevenly as concrete becomes drier. Reassure client that mottled color does not indicate variation in distribution of cementitious material. *Note:* The color cannot be restored by rewetting.

- **Client's question:** Why didn't this concrete that was placed 3 weeks ago ever set up? The concrete can be shoveled up like gravel and is almost dry. **Preliminary plan of analysis:** (1) Rewet finer material, and test for setting properties. (2) Conduct chemical analysis to determine amount of retarder. (3) Recommend an investigation of admixture dispensing system at concrete plant to determine likelihood of addition of too much retarder.

- **Client's question:** What material is in these stalactites found hanging under this concrete bridge? Or, what is this white (gray, beige, etc.) material occurring as a coating on this concrete? What do they mean concerning the durability and strength of the bridge? **Preliminary plan of analysis:** (1) Collect some of material and determine its composition. Often, this may be accomplished by examination of grain-mount preparations using the petrographic microscope. (2) Determine presence or absence of alkali-silica gel (see 10.2.3). (3) Visit placement, and evaluate condition of concrete.

- **Situation:** This concrete set up much faster than expected. **Client's question:** What is wrong with it? Did it get properly consolidated? **Preliminary plan of analysis:** (1) Perform analysis of air-void system, including sorting voids encountered into entrained, entrapped, and irregular. (2) Perform general examination with stereomicroscope with emphasis on paste quality. (3) Perform chemical analysis to determine if admixtures (accelerators, water reducers, etc.) are present in specified amounts.

- **Situation:** This concrete had a very high slump and a runny consistency. **Client's question:** What is the water-cement ratio of this concrete? **Preliminary plan of analysis:** (1) Perform examination of specimens for aggregate segregation. (2) Perform analysis of air-void system, including sorting voids encountered into entrained, entrapped, and irregular. (3) Perform general examination with stereomicroscope with emphasis on paste quality and estimate of water-cement ratio. (4) If necessary, perform chemical determination of cement content.

- **Situation:** Pavement is pushing out and causing humps in adjacent bituminous concrete. **Client's question:** Can concrete grow? How? Why? **Reply:** Yes. Certain concretes are made using expansive cement and are designed to expand and thus minimize cracking. This type of concrete may have been used as a repair material. If an original placement appears to be expanding, the concrete may be affected by one of the alkali-aggregate reactions that cause expansion of the concrete (see Chapter 10). **Recommendation:** Investigation of problem site by petrographer or engineer experienced in techniques of 10.2.2, probably including a complete sampling program.

continues

Table 3-1 (cont.)

- **Client's question:** How deep are these cracks? Will they cause corrosion of the reinforcing rods? **Preliminary plan of analysis:** Measure depth of cracks and distance from end of crack (as visible with stereomicroscope) to reinforcement. If distance to reinforcement is less than 2 in., report that cracks will likely cause corrosion.
- **Concrete researcher's question:** Can you (the petrographer) actually see a difference between concrete with a low chloride ion permeability and that with a high permeability (see ASTM C 1202)? **Reply:** In extreme cases, yes. We can sometimes see the differences with the stereomicroscope. In other cases, we can see the differences in fluorescent-impregnated thin sections by the use of the P/EF microscope.
- **Concrete researcher's request:** Measure these cracks (depth and width at 1-in. intervals), and give me the data so that I can calculate the volume of the crack. **Plan:** (1) Prepare lapped surfaces at right angles to surface. (2) Gently mark lapped surfaces with graphite as close to wearing surface as possible and then at 1-in. intervals down depth of cracks. (3) Using stereomicroscope and various stage micrometers, measure width of cracks at marks, and report results.
- **Concrete researcher's request:** Compare these two beams subjected to testing for resistance to freezing and thawing, and report any internal differences. **Plan:** (1) Prepare lapped internal surfaces. Mark microcracks as described in 8.4. (2) Either count cracks along traverse lines in specimens or visually compare markings.
- **Concrete researcher's request:** Compare the bond in these different types of overlay (or different methods of achieving overlay bond), and report the location of any cracking or debonding. **Preliminary plan of analysis:** (1) Prepare lapped surfaces that cut across bond at right angles. (2) Using stereomicroscope and a pen as described in 8.4, mark all microcracks. (3) Study relationship of microcracks and any obvious debonding to line of bond between overlay and substrate. *Hint:* In many cases, especially when removal of damaged material has been vigorous, the break between the overlay and substrate will not occur on the bond between the two materials but rather in the substrate 2 or 3 mm below the bond.
- **Client's question:** "Why is this concrete so good?" (Only once in the more than 25 years of my experience was any HCC submitted with this request.) **Plan:** Examine a number of lapped internal surfaces. **In this case, the answer:** The concrete in question had a low water-cement ratio and had been fabricated with a particular sort of expanded aggregate that provided a paste-aggregate bond that was much better than that usually found in concrete fabricated with natural crushed stone or gravel.

Table 3-2
TYPICAL TYPES OF SPECIMENS

- Cores
- Cylinders, beams, or bars fabricated for various concrete testing procedures
- Fragments that cracked off a placement or were purposefully broken off with a sledge hammer or jackhammer
- Bags or boxes of deteriorated concrete (usually looks like powdery gravel)
- Specimens of concrete that reacted strangely during placement or finishing
- Chips or popouts of concrete
- Pieces of concrete tile, conduit, or ditch lining showing deterioration
- Sand, gravel, or crushed aggregate

No matter what the reason or type of specimen, four general procedures are performed for each specimen received for petrographic examination:

1. Formally receive the specimen.
2. Perform the initial examination.
3. Make a plan for analysis of the specimen.
4. File the appropriate documents.

3.2 FORMAL RECEIPT OF SPECIMEN

Upon receipt of a specimen, six tasks are performed, as listed in Table 3-3.

Table 3-3
PROCEDURE—FORMAL RECEIPT OF SPECIMEN IN LABORATORY

1. Study the accompanying documentation, and carefully consider any oral instructions from the client.
2. Make written notes concerning the condition of the specimen and any obvious forms of deterioration.
3. Make sure the specimen is suitable for the analysis requested by the client.
4. Make sure the specimen agrees with its accompanying documentation.
5. Mark and log the specimen.
6. Fill out a "request for petrographic services form."

1. Study the accompanying documentation, and carefully consider any oral instructions from the client. Take careful note of any indication that the results of the analysis are needed within a short time or that they may be required as evidence in any litigation.

2. Make written notes concerning the condition of the specimen and any obvious forms of deterioration.

3. Make sure the specimen is suitable for the analysis requested by the client (see Appendix B). If, for example, the client requests an analysis of the air-void parameters or a complete petrographic examination and the specimen was reduced to a rubble or thoroughly cracked during sampling or compressive strength testing, it will be impossible to prepare the necessary representative lapped surface. If the client's concern is the lithology of the aggregate, such a specimen will be suitable. If the specimen is only slightly cracked or can be easily glued back together, it is often possible to prepare a lapped surface of at least a portion of the specimen. The cracking from testing will make it impossible to study some of the causes of deterioration because it will be impossible to distinguish testing cracking from the cracking indigenous to the placement. If there is reason to suspect that the specimen has not been properly cared for (improper storage and curing, treated roughly, broken, etc.) and its condition will not allow an accurate reply to the client's request, the use of the specimen will be impossible. If the specimen is smaller than

the petrographer thinks necessary for the examinations requested (core less than 3 in. in diameter, small irregular fragments) or if, for any reason, the specimen is such that adequate data cannot be obtained from it, explain the effects of these conditions on the data that will be obtained to the client and request a better specimen. In all cases where a patched, undersized, or otherwise imperfect specimen must be used, discuss the matter in the final report on the specimen.

4. Make sure the specimen agrees with its accompanying documentation. The documentation should indicate the source of the specimen, the date of the placement, the amount of traffic it has suffered (in the case of a pavement surface), the method of removal from the placement, any testing procedures that have been performed, and any results of such testing. If the documentation does not discuss or ask questions concerning any unusual features that are easily seen, sufficient data (data related to the specific questions asked by the client) concerning the specimen are not included, or sufficient information concerning the billing of the charges is not received, contact the client for further explanatory material.

The documentation and oral exchanges between the petrographer and the client should make clear the proposed use of the data obtained by the petrographic examination. If the data obtained from the specimen will become part of a legal controversy, note this fact and consider it throughout the entire analysis. Certain short cuts may be considered permissible for work done within one organization, but data collected for presentation by an expert witness must be gathered according to the exact procedures detailed by the test method employed. For example, ASTM C 457 states that three randomly selected test specimens are required from a placement for a determination of air-void structure for compliance with specifications. For work done in one organization, air-void determinations are often made on only one specimen; for data to be presented in a court of law, unless the data are obtained from three specimens randomly selected from the entire volume of the placement in question, the data must be qualified as having been obtained from the specimen as submitted and not necessarily representative of the void system of the entire placement.

Keep the original container (if any), the specimen, any documentation, letters of request, field notes, photographs, maps of the sampling plan, and other identifying papers all together until all these items have been entered into the logging system and files of the petrographic laboratory and the specimen and papers have been marked or otherwise made identifiable by a numbering system unique to the petrographic laboratory.

5. Mark and log the specimen. Mistakes are made in all laboratories, but it is most important to avoid mistakes at the time the specimen is received. Many other errors can be corrected if one can be assured that the identifying marks on all the individual fragments of the specimen and the original entries in the logbook and on the request documents are correct. Therefore, it is extremely important that this work be performed correctly and checked carefully. Do not do part of the job one day and leave the remainder to do the next. The most important procedure is the marking so that the specimen will always be identifiable and never confused with any other specimen. Never assume that this or that specimen or this or that fact will be easy to remember. *No petrographic procedure is more important than proper identification of the specimen.* There can be no reason to postpone specimen identification until some other procedure is begun on the specimen.

Experimentation has shown that the most durable markings are those made with graphite (ordinary "lead" pencil, or carpenter's pencil). Unfortunately, graphite markings are often difficult to find and distinguish on concrete surfaces. If a felt marker is used, the marks may have to be refreshed after the specimen is subjected to oil, acetone, or alcohol. Even India ink cannot always withstand the rigors of the solvents used in the petrographic laboratory. Great marking security can be achieved with the heavy use of graphite, with additional identification clearly marked with a felt marker.

The in-house specimen numbering system of the petrographic laboratory must be individual to the laboratory and nearly impossible to confuse with any other numbering system that may be associated with the specimen. For example, at VTRC, specimens fabricated in the concrete mixing laboratory have a numbering system that uses what is called the master numbers. The petrography laboratory receives specimens from the concrete laboratory with master numbers on them, but it creates confusion if the petrography staff tries to use the master numbers as the sole means of identification. A petrography number is assigned to the specimen, and the master number is recorded in the logbook, as are all other identification marks that accompany the specimen.

Petrography specimen numbers are preceded with a P, e.g., P-1222. The appropriate petrography number (including the P) is clearly marked on each specimen with a felt marker and graphite (see Fig. 3-1). Such numbers are called P-numbers. The use of P-numbers is the major method of tracking specimens and facilitating the location of data when questions concerning a specimen are received. The number is

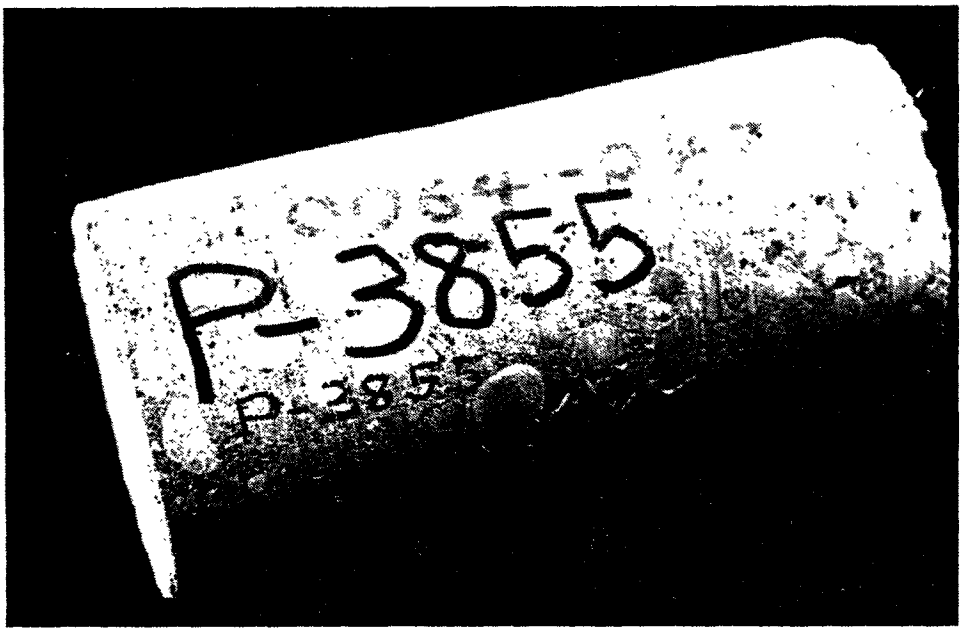


Figure 3-1 CORE WITH P-NUMBER. The original construction number is not obscured, and the P-number is marked with a felt marker and graphite.

included on all correspondence so that recipients of the correspondence may use the number for making inquiries concerning the specimen.

CAUTION: Many ordinary inks begin to fade after they have been in contact with HCC paste for a few days.

In the petrographic laboratories of VTRC, the most useful documentation of the receipt of a specimen was found to be a chronological log of all specimens entering the system (see Fig. 3-2). It is often used long after the original investigation has been concluded to discover when and how many specimens were submitted from a placement, what examinations were performed, where the specimens are currently, and how the data can be found. The logbook stays in one place and thus can be easily found. The log provides a guide to all the information available concerning a specimen from any source.

Entering the initial data must be easy and not very time-consuming. In its simplest form, the log might merely record the date, any specimen identification marking received on or with the specimen, the type of specimen received, the file number under which correspondence will be stored, and, most important, the petrography in-house specimen number. Indicate the general size of the specimen and whether the material (1) was cored with a diamond core drill from a hardened concrete placement; (2) was cast in a cylinder when the concrete was placed; (3) was produced in the laboratory or field as a cylinder, beam, or bar; (4) was found as a fragment; or (5) is a fragment that was sawed or hammered from a placement. Information that cannot be derived from the specimen should be available in the original documentation. In addition, the logbook can be used to record the progress of the investigation, tests performed, and disposal of the specimen or portions of the specimen.

5. Fill out a "request for petrographic services" form. The form used by VTRC (see Fig. 3-3) has been changed many times, but it now seems quite adequate.

If the client is a person who is nearby (in the building), he or she should fill out the request form; otherwise, a petrographer should fill it out after the specimen is logged. The form should provide places for the project name, dates, charge numbers, file numbers, the source of the specimen, a brief description of the specimen, requested examinations, instructions to technicians, P-numbers, and the client's original numbers. Place the original of this request form with any other documents associated with the specimen. Send a copy to the client to notify him or her of the P-numbers assigned and that the specimen is in the system. Include a copy of the request form with the specimen as it is moved from office to preparation room and then to examination rooms for the microscopical procedures.

Petrographic Examination & Sample Testing

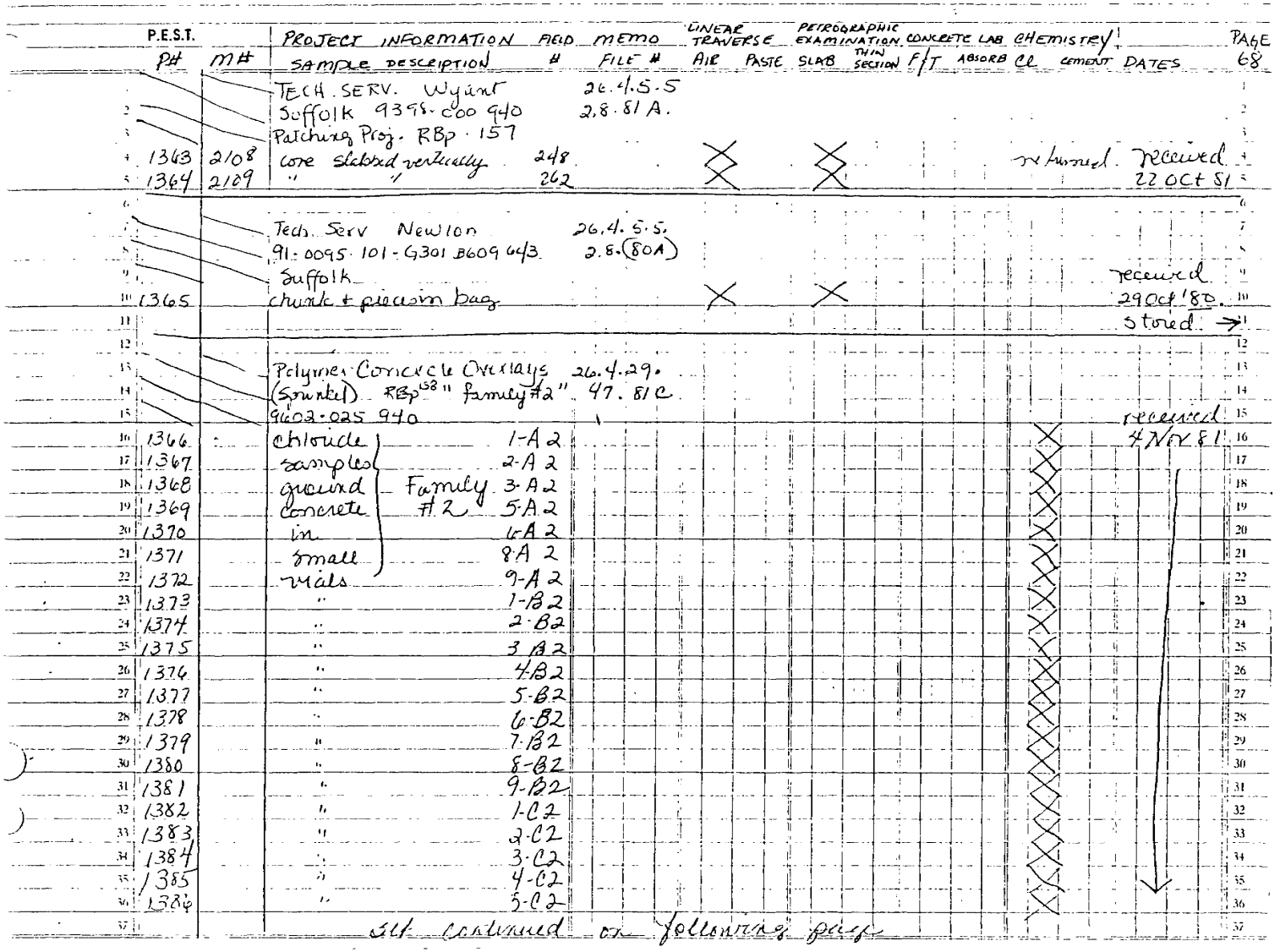


Figure 3-2 PAGE OF LOGBOOK

1174

TO BE COMPLETED BY PETROGRAPHY

Request Book Page 299

CLASS OF WORK A

Sheet 1 of 1

Date: 1 JAN 91

REQUEST FOR PETROGRAPHIC SERVICES

Project Title: DETERIORATION OF BRIDGE DECK

A CO CO	B ROUTE ROUTE	C CITY/CO. SECTION	D SECTION CREW	E JOB NO.	F LAND OWN	ACTIVITY
2,6	6,8, ,	0,7,1	, ,	, ,		9,4,0

Charge No.:

Section: BRIDGE

District: NORTHERN

VTRC File No.: 3.2.5

(Petrography: 26. 4. 3. 2. 5 / 91)

Submitted by: BOOKING

Source: BOOKING

Reason for Submittal
(e.g. Type of Deterioration) DEEP SCALING

Analyses Desired if Known AIR VOID & GENERAL EXAMINATION

Description of Samples (core, cylinder, rock, gravel, etc.) Include your Designation of sample numbers	TO BE COMPLETED BY PETROGRAPHY			
	P.E.S.T.#	Sample Prep. (ground slabs thin sec., etc.)	Analyses Performed	Brief Data Obtained
Description - Field No.		LAPPED SLABS		
CORES, CRACKED				
A 1	3033			
A 2	3034			
B 1	3035			
B 2	3036			

Date Data Required: ASAP

Remarks:

Copies: Request Book
Project File
H. E. Brown

B. F. Marshall
Other:

Date of
Report Memo:

Figure 3-3 VTRC REQUEST FOR PETROGRAPHIC SERVICES FORM

3.3 INITIAL EXAMINATION

The first laboratory notes on the specimen are written during the initial examination. The specimen as received may be large, but do not cut it to size until a complete plan of specimen examination has been drawn up. Use a hand lens or magnifying glass for examination until a reduction in size is a scheduled part of the plan. These preliminary notes and the client's request provide the direction for the plan of petrographic analysis.

The initial examination is accomplished in six steps, as listed in Table 3-4.

Table 3-4
PROCEDURE—INITIAL EXAMINATION OF SPECIMEN

1. Note and describe any cracks.
2. Note the location and condition of metal or any other material purposely included in the HCC.
3. Note the condition of the wearing surface.
4. Note the condition of the paste, any reaction products, the general size and distribution of the aggregate and air voids, and any other unusual features.
5. Photograph the specimen.

1. Note and describe any cracks. Pay particular attention to cracks that are on surfaces that were visible before the specimen was removed from the placement; these cracks were the ones first noticed by the client and are probably the ones that occasioned the submittal to the petrographic laboratory. If it appears that cracks on the top of the specimen may be part of a system of cracking and the client has not indicated the extent of this system, contact the client and make arrangements to obtain further information in the form of both verbal description and photographs or by personal visit to the placement.

Become familiar with the material in Chapters 4 and 10 so that you can recognize cracks due to plastic shrinkage, alkali-aggregate reactions, and lack of an air-void system that can provide protection from freezing and thawing. Take precautions to preserve the evidence of these forms of deterioration.

Cracking that appears due to plastic shrinkage (see Chapter 4) is often cause for legal action and must be thoroughly investigated and photographed before any further procedures are performed on the specimen. Cracking that is due to insufficient protection by an air-void system may be cause for litigation if it occurred while the contractor could be held responsible.

Cracking that appears due to alkali-aggregate reactions will usually not be cause for legal action unless the materials used were not those specified. It is probable that the client will want a complete description of the aggregates, the reaction, the reaction products, and how the reaction affects the concrete. Contact the client by telephone, and inquire concerning the required breadth of the petrographic investigation. The study of thin sections, photographs, and photomicrographs may be requested. The client will probably desire sufficient information so that the reaction can be avoided in future concretes.

If the specimen is a cylinder and shows cracks, poor consolidation, or other signs of mishandling (improper storage or fabrication or both) and the client's request con-

cerns low strength, it is possible that the cylinder tested showed low strength because it was flawed. Contact the client, and suggest compressive strength testing of specimens cored from the placement. If petrographic examination is required for other reasons, request replacement specimens (preferably cores). If replacement specimens are not available, try to avoid the flaws when planning the specimen preparation and make sure that the final report describes the condition of the cylinder and mentions the fact that the data obtained were not from the entire specimen.

In the case of a core or other specimen obtained from a hardened placement, try to judge which cracks are indigenous to the concrete of the placement and which cracks can be ignored because they were produced by the sampling procedures employed. Cracks produced by sampling procedures will usually appear fresh and contain no reaction products, but these criteria alone do not mean that the cracks were caused by sampling. If reaction products are present in the cracks or road dirt (dirt not due to drilling the sample) is present in the vertical cracks, such features can be assumed to be indigenous.

A system of cracks parallel to the wearing surface (called scaling), especially if the cracks become wider spaced with distance from the exposed surface, is probably due to freezing and thawing of saturated concrete unprotected by a proper air-void system (see Fig. 3-4). The most important determination to be made on such concrete is an analysis of the air-void system.

Systems of vertical cracks visible on the surface and most closely spaced at joints and pavement edges are called D-cracking. In the midwest, such cracks are usually due to the deterioration of certain impure dolomitic aggregates under the conditions of freezing and thawing (Schwartz, 1987). In the northeast, such crack patterns

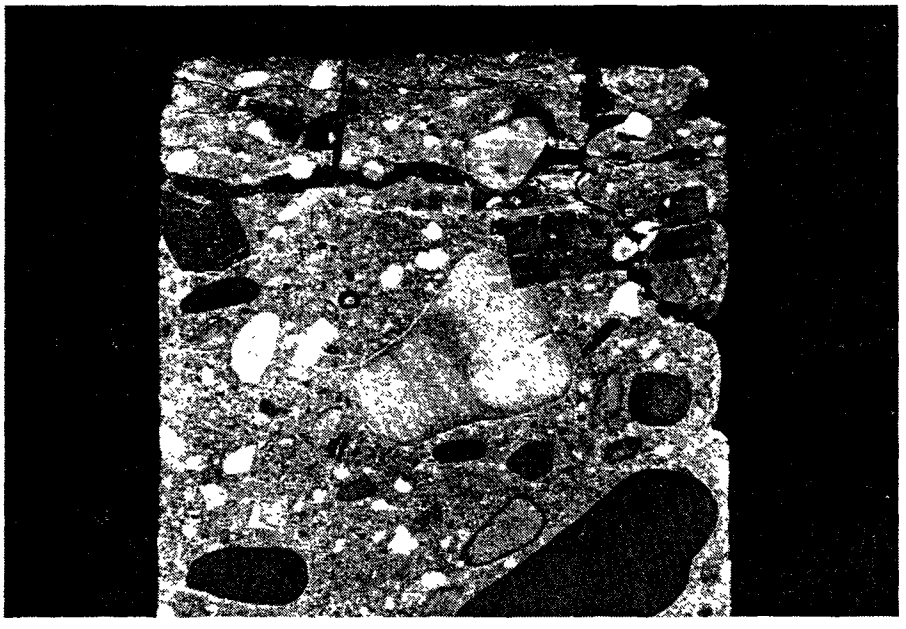


Figure 3-4 SCALING CAUSED BY FREEZING AND THAWING. Occurred in concrete unprotected by proper air entrainment. To photograph the cracking, the specimen was glued back together with a dark glue and then cut vertically across the layered cracking.

have usually been attributed to deterioration of the concrete paste due to the lack of an air-void system capable of providing protection from freezing and thawing (Andrews, 1953). When D-cracking is present, both the aggregate and the air-void parameters should be determined. D-cracking has not been found in Virginia.

2. Note the location and condition of metal or any other material purposely included in the HCC. If the material is not part of a commonly used reinforcement system, contact the client to inquire as to its origin.

Notice whether the location of any of the surface cracking is related to the reinforcement (Figs. 3-5 and 3-6). Note the placement and condition of any reinforcing steel. Check for corrosion products near the steel and any associated cracking. If there is a system of cracks on the concrete surface that appear to lie directly over the reinforcement, this cracking may be due to corrosion of the steel.

Note horizontal cracks on the specimens. Note the depth of occurrence of these cracks. Often, there will be delaminations at the level of the top steel.

The thickness of concrete over the reinforcement necessary to provide protection from the corrosive effects of atmospheric gases with various types of concrete is detailed by Cady (1978).

Examine the bond between the paste and any other materials (such as reinforcement or anchoring pins) that are purposely present. Usually, the bond should be tight and strong and leave no space for the migration of fluids, wobble, or abrasive wear between the concrete and the other material (Lutz, 1978).

Aluminum, zinc, glass, and many plastics may be used in HCC as connectors or electrical conduit. These materials are subject to corrosion when enclosed in HCC



Figure 3-5 CRACKING ON SURFACE AND SIDE OF CORE. With associated corrosion and expansion of the reinforcing bar.

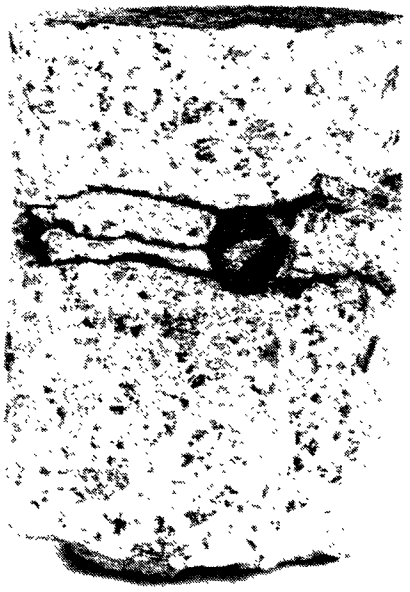


Figure 3-6 DELAMINATION AROUND REINFORCING BARS. There is no cracking on the surface.

in the presence of moisture, corrosive gases, or both. If any of these corrodible materials is present, they may prove quite deleterious (Erlin & Woods, 1978). Note the condition of these materials and any associated cracks and reaction products.

3. If a finished roadway surface (wearing surface) is present on the specimen, note the condition of the texture of the surface. If the surface appears unusual or unable to provide skid resistance, consider the age, amount of traffic suffered by the roadway, original specified texture, and weather during placement. Unless these data are already available, obtain information concerning these factors from the client. Consider preparing a thin section (see 5.3.3).

4. Note all unusual conditions insofar as is possible without the use of a microscope and with no preparation of the specimens (occasionally, washing and partial-to-thorough drying may be necessary). If the client has submitted more than one specimen, be specific and note on which specimen the feature occurs.

Unusual conditions include, but are not limited to, the following:

- condition of the paste (make a preliminary assessment of its friability, porosity, and maturity)
- reaction deposits in voids or in or around aggregate particles
- segregation or alignment of aggregate particles
- areas of abnormal (high or low) paste content
- areas of abnormal (high or low) void content
- textural differences of the paste in different areas of the concrete: near the wearing surface, middle portion, and deepest portion of the specimen

- occurrences of unusually large voids
- pockets of high concentrations of voids
- patches of paste completely lacking in voids
- pockets or aggregate rims of highly concentrated unhydrated cement
- contaminants (such as twigs, parts of shoes, and metal fragments)
- unusual exterior marks made by forms
- unusual dyes, paints, or extra-thick curing compound on exterior surfaces
- evidence of early freezing or later deterioration due to freezing and thawing

Casts of ice crystals are evidence of early freezing. If the concrete is fragmented and casts of ice crystals are abundant on almost all of the fragments (as seen in Fig. 3-7), the concrete was forced apart by the expansion of the ice formed before the paste achieved final setting.

Ice crystal patterns, such as "jack frost" patterns, may be visible on the molded surface of cylinders. Take these patterns at face value; the cylinders were subjected to freezing temperatures, but the patterns do not provide any evidence that the placed concrete was likewise frozen. Obtain a temperature history of the placed concrete and the cylinder concrete from the client before making any conclusions regarding these ice crystal molds. If the cylinders were treated as was the placed concrete, examine them for evidence of curing procedures and molds of ice crystals that may occur in freezing temperatures in fresh, improperly protected concrete (Rhodes, 1978).



Figure 3-7 FRAGMENTS OF CONCRETE DESTROYED BY FREEZING BEFORE FINAL SETTING. The scale is in intervals of 1/8 in.

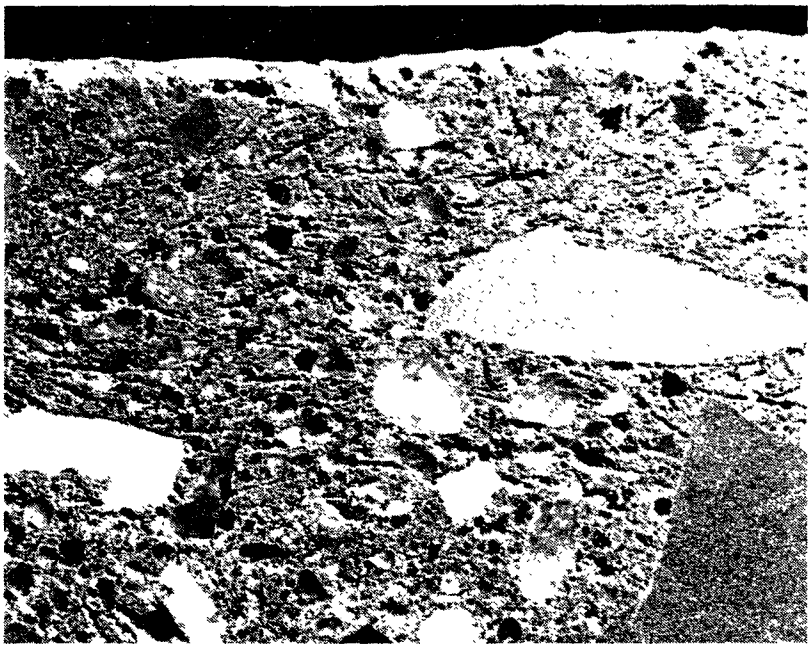


Figure 3-8 SURFACE SAWED THROUGH CONCRETE SLAB THAT FROZE BEFORE FINAL SETTING. The blades of ice were, in general, parallel with the surface, and their orientation can be easily seen. The molds of the ice crystals are now empty and create zones of weakness throughout the concrete. About 3X.

A surface layer of concrete in which molds of ice crystals are prevalent (see Fig. 3-8) indicates that the immature concrete was subjected to extremely chilling conditions (cooled below freezing, often with a wind that removed the heat of hydration). The unhardened concrete can freeze to a depth of several inches and develop casts of the ice crystals, which become zones of weakness and channelways for solutions.

5. Photograph the specimen with a scale and identifying P-number labels. This step may be omitted if previous photographs of the specimen have recorded all visible features that caused concern to the client, the important features, and any differences among a suite of specimens submitted as one sample.

If the initial observations seem to answer the client's questions completely, contact the client and ask if he or she wishes the investigation to proceed further. If the client is satisfied with the data already obtained, prepare all necessary written replies, permanent documentation, and the files as described in 3.5.

3.4 PRELIMINARY PLAN OF ANALYSIS

Preparing a plan to analyze the specimen may be accomplished in four steps, as indicated in Table 3-5.

1. Develop a plan for analysis that will answer the client's questions, explain unusual features, be performed within the specified time frame, and

Table 3-5
PROCEDURE—PRELIMINARY PLAN OF ANALYSIS OF SPECIMEN

1. Develop a plan for analysis that will fulfill the client's needs and explain unusual features.
2. Mark the planes to be cut.
3. Photograph the marked specimen.
4. Prepare and file documentation explaining the plan for analysis.

be consistent with the likelihood of the results of the analysis having to withstand scrutiny by hostile expert witnesses in a court of law. From these assessments, from what is known of the history of the HCC, and with full consideration of the questions asked by the client, develop a plan for the petrographic examination that will reveal further information concerning these features and allow sound petrographic judgments to be made concerning the causes of any distress or unusual features (see ACI 201.1R; ACI 201.2R; Mather, 1978). The sample preparation plan should include deciding which portion of the specimens will be prepared for which test and which portions will be reserved for further reference. If any thin sections will be required, it is best if the location of these sections can be decided as soon as possible and the process of fabricating them begun. Tables 3-6 through 3-8 provide samples of the sort of plans that might be developed. See also Table 3-1.

Table 3-6
SAMPLE OF TYPICAL ANALYSIS

Client's request: Explain the low strength results (strength data enclosed). A very prompt reply is requested. Telephone any preliminary data.

Specimens: Three cores 4 in. in diameter and 5 in. in depth.

Preliminary examination: A visual estimate of the air-void content indicates that two of the three specimens have areas of abnormally high air-void content and there are patches of paste that are low in aggregate.

Telephone report: The low strength is probably due to a high void content. The patchy nature of the concrete may indicate retempering.

Further client request: Conduct an analysis of the air-void parameters, and complete a megascopic and stereomicroscopic petrographic examination.

Plan of analysis:

1. Prepare vertical, finely lapped slices from each specimen.
2. Conduct a brief stereomicroscopic examination of the slices to confirm the original estimate of a high void content in two specimens and a normal content in the other. Consider if it is necessary to have additional slices prepared in order to represent the specimens properly.
3. Conduct an analysis of the air-void parameters of each type of specimen.
4. Conduct a detailed stereomicroscopic examination of each specimen slice to locate all features that might contribute to low strength and especially to search for any features that might indicate retempering.
5. Obtain photographs of the slices.
6. Prepare notes for the report, and report brief results to the client by telephone. Briefly record the results in the logbook. Record the data necessary to relocate negatives and proofs (see 3.5.1).
7. Prepare a memorandum, letter, or whatever is required as a formal report to this particular class of client.

Table 3-7
SAMPLE OF TYPICAL ANALYSIS

Client's request: Examine the specimens fabricated and tested in the VTRC concrete laboratory to determine if the differences in dynamic modulus can be explained by visual evidence.

Specimens: Two beams 3 by 4 by 16 in. prepared for testing in accordance with ASTM C 666. They were subjected to identical treatment, but an experimental chemical admixture was used in one beam. The concrete containing the admixture had a low dynamic modulus after the test.

Preliminary examination: Conduct a cursory examination of the exterior of the beams.

Plan of analysis:

1. Prepare finely lapped slices from the interior of each specimen. Obtain a sufficient slice from each specimen to satisfy the area requirements specified in ASTM C 457.
2. Conduct a visual examination (without any marking of the slices) of each finely lapped slice to survey the deterioration and plan further action in view of the following: aggregates, cement remnants, etc. appear identical; abundant microcracks are noted in both specimens. No comparison between intensity of cracking is usually available at this stage of the investigation.
3. Conduct an analysis of the air-void parameters of each specimen, and report the results to the client by telephone.
4. Obtain photographs of each slice. Record the roll and frame or file numbers of the negatives and proofs.
5. On each specimen slice, using a tape, mark off test areas of similar size and with a similar amount of paste.
6. Conduct a detailed stereomicroscopic examination of and mark in ink all microcracks within the marked test areas and other features (such as reaction products) detectable on the slices.
7. Obtain photographs of each marked slice. Record the roll and frame or file numbers of negatives and proofs.
8. Make a visual megascopic assessment of the comparison between the interior damage in the two specimens.
9. Prepare notes for the report.
10. Oral report: Present the marked slices or photographs to the client with the assessment. Suggest having a technician count the cracks on a series of traverse lines across the slices or the pictures.
11. Written report: Produce a written record of the examination. If the client requires no record other than the pictures, briefly record the results in the logbook and make note of the data necessary to relocate the photographs and negatives (see 3.5.1).

---

2. With a felt marker, mark the first planes to be cut with the diamond-edged saw with full consideration of the need for a detailed examination of the various crack patterns (see Chapter 4) and the likelihood of the need for an analysis of the air-void system. If the cutting plan is complex or if certain determinations must be made before the sawing is complete, it may not be possible to mark the cutting lines for all the planes.

A slice approximately 7/8 in. in thickness is generally required for air-void determinations and general petrographic examinations. This thickness generally yields a slice that is strong enough to withstand normal laboratory wear and handling but is thin enough to permit a comfortably seated microscopist to focus the microscope

Table 3-8
SAMPLE OF TYPICAL ANALYSIS

Client's request: Determine the seriousness of the damage to the concrete caused by a driving rainstorm during placement. A prompt reply is requested.

Specimen: One core 4 in. in diameter, 3 in. in depth.

Preliminary examination of top and sides of core: Visual estimate of depth of damage.

Telephone call by petrographer to client: How much pavement area looks just like the surface of the specimen sent to us? How many intermediate types of surface are there? If the area is extensive and the damage looks serious, send a sketch with the dimensions of the areas involved and a core specimen of each of the different portions of the placement affected by the rainstorm. Send pictures if possible. [Client sent three additional specimens of the surface and sketches.]

Further client request: Detailed comments concerning damaged area and durability prognosis.

Plan of analysis:

1. Prepare vertical, finely lapped slices on which to examine the depth and nature of any distress.
2. Describe the distressed concrete, and measure the depth of the distress in each type of area. Include the condition of texturing, description of any color change between the surface layer and interior of the concrete, any cracking in the affected area, description of the air-void system near the surface (the surface layer is usually too narrow to permit a full air-void determination), and comments on the water-cement ratio near the surface.
3. Obtain photographs of the surface textures (particularly in cross section) showing the depth of any discoloration, layer of excess voids, or other indicator of condition. Record the roll and frame or file numbers of the negatives and proofs.
4. Compose a report including an estimate for each definable area of the depth to which the concrete will quickly wear away or how much concrete should be removed or textured to restore usefulness. If the damage is very slight, so state. Include photographs that illustrate the reasons for the conclusions.

easily without stretching the neck or cramming his or her knees 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 so that the integrity of the slice will be preserved during subsequent handling. In cases where one or two major cracks exist in the specimen in the area to be sliced and the specimen has fallen apart or is about to fall apart, a strong epoxy may be used to keep the specimen together. Take care to prevent the epoxy from being squeezed or dripped into areas where it might obscure important features.

It is often necessary to reconstruct the specimen after cutting; therefore, place marks across all cutting planes (see Fig. 3-9). These marks are called match marks. Match marks should be unique to the cutting plane; one mark for the first plane, two marks for the second, etc. Take care to avoid marking any surface that will be subjected to any form of analysis. Most inks, from pens and markers alike, can sink deeply into porous concrete and will probably not be completely removed by lapping procedures. Ink marks can cause erroneous identification of aggregates and crack features and make the visualization of the paste features difficult.

Plan to cut across (at approximate right angles) any major cracks observed. Lapped surfaces produced on these cuts will enable better observation of the crack pattern.

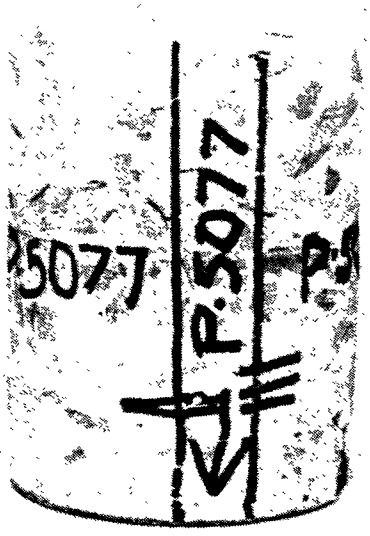


Figure 3-9 CORE MARKED WITH IDENTIFICATION, CUTTING PLANES, AND MATCH MARKS

The surface produced should allow the petrographer to see into cracks from the finely lapped surface.

Most commonly, surfaces are prepared by the technician cutting the material in a direction approximately perpendicular to a formed or finished surface of the HCC, and preferably across the layers in which the HCC was placed, thus producing a slice from the center of the material to be examined. This method is good when the size of the section produced does not exceed the capacity of the lapping equipment available or there are no obvious differences to be seen between the top and the bottom of the cores and the bottom portion can be ignored.

On occasion, it may be necessary to determine the differences between 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.

When the full depth of a core must be examined and the length of the core exceeds the size capacity of the lapping equipment, it may be necessary to saw the core horizontally into two or more pieces before preparing the slices to be surfaced.

3. Photograph the marked specimens (see Fig. 3-9).

4. Prepare and file documentation explaining the plan for analysis. In any document produced concerning the specimen, report the cutting plan used and explain it. Protect yourself in case any litigation should ensue by always reserving some portion of each of the significant parts of the specimen for at least 5 years. Longer storage is recommended if possible.

3.5 FILING OF APPROPRIATE DOCUMENTS

3.5.1 Permanent Case Files

Keep a permanent file in the petrography laboratory for each specimen that enters the system, even if the client's questions have been answered. In the file, place copies of all the original documents, notes on any oral communications with the client, the updated request sheet, laboratory notes, printouts of results of tests, and copies of all correspondence. Correspondence will probably be filed by clerks or secretaries in a more formal filing system, but that system may not have as much accessory data as does the file in the petrography laboratory. Keep a record of all testing procedures, such as air-void determinations. Such files are probably best organized by P-number and may be kept in the laboratory where the test is performed.

If photographing of specimens is performed in the petrographic laboratory, keep the negatives and prints in the permanent file for the specimen. If the photography is performed by another division of the organization that has possession of all negatives, place a record of how the negative is filed and the location of any prints in the file.

3.5.2 Temporary and Archive Files

Keep a temporary file of request sheets of work in progress. As a job is completed, update a copy of the pertinent request form and move it to an archive file. This archive file will be useful when a question comes in concerning an old specimen and the date and P-number are unknown.

CHAPTER 4
CRACKS

4.1 OVERVIEW

Cracks in HCC may have several causes (see ACI 201.1R and ACI 224.1R): plastic shrinkage, settlement, drying shrinkage, thermal stresses, chemical reactions, weathering (freezing and thawing, wetting and drying, heating and cooling), corrosion of reinforcement, poor construction practices (e.g., retempering), construction overloads, errors in design and detailing, and externally applied loads.

For our purposes, eight types of cracks are discussed:

1. microcracks
2. crazing
3. scaling
4. cracks due to insufficient air-void content
5. cracks due to delamination at the reinforcement
6. cracks due to a chemical reaction
7. cracks due to drying shrinkage
8. cracks due to plastic shrinkage

4.2 TYPES OF CRACKS

4.2.1 Microcracks

The term *microcracks* includes all the very fine cracks, in any direction, at the surface or within the mass of the concrete that are not easily visible with the unaided eye but may be seen with a magnifying glass or microscope. Microcracks are often extremely difficult to observe on the textured surface of a placement. Chapter 8 provides instructions on the observation and recording of microcracks.

4.2.2 Crazing

Crazing is a fine, very shallow cracking that occurs in the exposed surface of a concrete placement. Usually, it cannot be seen while the observer is in motion in a vehicle. It may be due to overfinishing, shallow freezing, or curing insufficient to prevent excessive drying, especially in concretes containing fly ash. Frequently, crazing has a very fine pattern, like pattern cracking except the individual uncracked central portions are usually no more than 2 in. across. When crazing causes the surface to flake off, usually to a depth of about 1/8 in., it may be called paper scaling. The crazing depth should be recorded. The underside of any loose flakes of concrete should be examined for the casts of ice crystals that would indicate freezing before hardening.

4.2.3 Scaling

Areas on the surface that appear to be peeling off are scaling. Scaling is usually shallow, less than 0.75 in. in depth, and may be due to imperfectly performed curing procedures so that layers of concrete that differ in water content are formed. The early loss of curing cover (burlap, polyethelene, or both), thus loss of moisture and heat, and freezing of the top layer before hardening are common causes of scaling.

4.2.4 Cracks Due to Insufficient Air-Void Content

Deeper scaling is usually due to an insufficient air-void content and, thus, lack of resistance to freezing and thawing (see Figs. 3-4 and 8-7).

4.2.5 Cracks Due to Delamination at Reinforcement

Cracks at the level of the reinforcement are almost invariably due to corrosion of the reinforcement and expansion. The high pH of the concrete has a passivating effect that protects the reinforcing steel from the corrosive effects of deicing salts and acid rain. The thicker the layer of concrete over the steel, the better the protection against corrosion of the steel. Any vertical cracking that breaks the surface of the concrete or any wearing away of the surface of the HCC lessens the protection that normal concrete will give the steel. The concrete is considered to have only the depth of protection that exists from the bottom of the cracks to the reinforcement (see Figs. 3-5 and 3-6).

4.2.6 Cracks Due to a Chemical Reaction

A chemical reaction may take place between the aggregate and the paste, especially when moisture is plentiful, or may be brought about by the ingress of destructive chemicals. Such cracking is discussed in Chapter 10.

4.2.7 Cracks Due to Drying Shrinkage

The term *drying shrinkage* is commonly used in connection with shrinkage cracking that occurs after the HCC has attained final set and a certain degree of bonding has developed between the aggregate and the paste. After the concrete has hardened and is fully set, the paste is a brittle material because it hardened due to hydration of the cement.

Most HCC shows evidence of drying shrinkage. Cracking can be minimized by good workmanship, proper proportioning of the mixture, and sufficient jointing performed soon after hardening. In jointed concrete, cracks may form if the joints were not formed early enough or are not working properly or the shrinkage in the hardened state is excessive. In continuously reinforced HCC, very narrow, fairly regularly spaced cracks are expected to form. The cracks allow for the changes in volume of the concrete with drying and varying temperatures. These cracks are expected to extend down into the concrete and up from the bottom of the slab, but the reinforcement is expected to keep the crack from extending close enough to the steel to allow deleterious solutions to depassivate and corrode the steel reinforcement.

4.2.8 Cracks Due to Plastic Shrinkage

Plastic shrinkage is a form of drying shrinkage that occurs while the HCC is unhardened and malleable and the bond between the components of the material is very weak. Plastic shrinkage cracking is caused by excessive evaporation of the water at the concrete surface due to improper curing procedures for a concrete under the climatic conditions existing at the time of placement (see Appendix D and Kuhlmann, 1991). Plastic shrinkage occurs in the first few hours after placement before final set (when the rate of evaporation exceeds the rate of bleeding). At this stage, the HCC has some properties of a solid, but no appreciable bond exists between the aggregate particles and the cement paste. This sort of cracking is also called early cracking or morning cracking, the latter because it is often the concrete that was placed in the morning and then exposed to afternoon drying conditions that exhibits this type of cracking (see Figs. 4-1 through 4-6).

Plastic shrinkage cracking often occurs in high-quality HCCs in which the distribution of the various components (such as aggregates and cement and any particulate cementitious admixtures) is often nearly homogeneous throughout the mass. The hydration is usually as complete and well distributed as would be expected of any concrete of this age. There appears to be nothing wrong with the HCC itself. It just cracked.

In modern HCCs with a water-cement ratio of less than 0.47, plastic shrinkage cracking seems to have no relationship to the orientation of the reinforcing steel or other structural features. In HCCs with a higher water-cement ratio, there can be a form of early cracking wherein the cracks are located directly over the steel. This cracking is directly related to segregation and settlement of the aggregate over the steel as the bleed water leaves the concrete and the volume of the paste is diminished. As with HCCs with a lower water-cement ratio, this condition was exacerbated by drying atmospheric conditions. Evidence of this type of segregation can be seen on interior surfaces of specimens of these HCCs. The associated cracking used to be called early morning cracking but is now more properly called settlement or subsidence cracking (ACI 224R).

Occasionally, there may be severe bleed channels in HCC that might be confused with plastic shrinkage cracking. However, upon close inspection, such bleed channels show clear evidence of being a waterway, whereas plastic shrinkage cracks show clear evidence of having been pulled open by the shrinkage of the concrete. Such bleed channels are trains of water voids caused by excess bleed water. Such trains of voids may occur in HCCs with a high water-cement ratio. Some forms of plastic shrinkage cracking may have zones of such void trains. In HCCs with a low water-cement ratio, poor consolidation may cause fold lines and collections of voids. Any cracks occurring in such HCCs can usually be recognized by the nature of the voids and the presence of brittle (as opposed to unhardened) fracture surfaces.

The following may occur in unusual and extreme cases of rapid evaporation causing drying of the HCC before hardening:

- Surface carbonation with associated shrinkage may take place when the relative humidity at the surface falls much below the saturated state.
- As surface paste loses its moisture by evaporation, unhydrated cement may be concentrated in a weak, thin, surface zone.

These are features of extreme drying and are not present in the most common forms of plastic shrinkage cracking. However, the absence of these features does not indi-

cate that the cracking is not due to drying conditions that are deleterious to the HCC.

4.3 DISTINGUISHING BETWEEN PLASTIC SHRINKAGE CRACKING AND DRYING SHRINKAGE CRACKING

4.3.1 Overview

For transportation departments and other purchasers of concrete placed by a contractor (or other agency), it is frequently important to distinguish between early plastic shrinkage cracking and the brittle cracking that may occur due to structural stress or later drying shrinkage. The contractor is obligated to prevent the dehydration (and consequent cracking) of the fresh concrete that can occur when wind, low humidity, or both promote rapid drying. Portland cement concretes with a low water-cement ratio (0.40 or less) and latex-modified concretes are more apt to suffer this sort of failure than are concretes with a high water-cement ratio. When it can be shown that the cracking is due to a failure of the contractor to refrain from placing concrete during unfavorable weather conditions, employ sufficient methods to prevent the drying, or both, the contractor may be obligated to provide a new surface or accept a lower payment.

The usual rule of thumb in the literature is: If the cracks go through the aggregate particles and cause them to break, the cracking should not be considered plastic cracking. Many observers call any crack that goes around the aggregate particles a plastic/early shrinkage crack. This can be in error. There may be other reasons for the crack to go around the aggregate particles.

Some aggregates are more fragile than others, and some may crack during final placing and finishing. It is possible to judge a crack to be a later crack on the criterion of broken aggregate when in reality the aggregate particle just happened to have a zone of weakness in the crack plane and the crack was an early crack. The specimen examined, a cross section of a crack, is a very small portion of a crack. The interior of the crack surface is a very small, nonrepresentative portion of the crack.

When the cracks preferentially go around the aggregate particle, the observer may be observing an HCC in which the bond between the aggregate and paste is very poor at any degree of maturity. The bond may be poor because of any of the following:

- There is excess water or air voids at the aggregate surface.
- There is a coating on the aggregate surface.
- $Ca(OH)_2$ at the paste-aggregate interface was dissolved by percolating waters or solutions, and thus the bond was weakened late in the life of the concrete.

Thus, it is necessary that the criteria for deciding that a specific crack is a plastic shrinkage crack be more than the fact that the crack skirts the aggregate particles.

4.3.2 Analogy with Clay Materials

The difference between drying shrinkage cracking and plastic shrinkage cracking can be explained further by use of an analogy using clay materials.

• **Hard brittle materials (drying shrinkage cracking).** Consider a broken ceramic object (pottery) or rock. If all the pieces can be found and fitted back together, the material will solidly fill the same space as did the original object. Any internal voids will almost invariably be recognizable by either their shape or the nature of the interior surface (different from a fracture surface). If all the chips are available, the expression of the crack on the surface of a hard brittle material will be a thin sharp line.

If the material is a sandy clay that was fired in a kiln ("fired" is analogous to the hardening of concrete), all the cracked surfaces will fit back together if all the fragments are preserved and there are no air pockets present. If the bond and the tensile strength of the ceramic are as strong as the tensile strength of the sand, then the crack will fracture the sand and the crack surfaces will neatly and completely fit back together. If the sand has much greater tensile strength than the fired ceramic, any cracks occurring in the material are likely to detour around the sand grains.

These facts may be directly extrapolated to hardened HCCs. The logic also applies to cracks in hardened portland cement concrete, hardened latex-modified HCC, and many other highway materials. In the case of these materials, the general type of void and the nature and luster of the interior of the voids should be carefully studied so that they can be recognized in the path of any crack under study.

It may be that a macrocrack occurring on the riding surface of HCC that was originally a thin sharp line was worn wider by the abrasive action of traffic. The course of the crack below the surface expression should be examined. Care must be taken to extrapolate the evidence in the light of all relevant facts, including the age of the placement, the amount of traffic, pertinent weather conditions, and the strength and general condition of the concrete placement.

• **Malleable materials (plastic shrinkage cracking).** Now, consider a crack caused by the pulling apart of a piece of modeling clay or other such material. Because of the plastic nature of the material, there may be small "bridges" of the material spanning the crack, there will be deformation of the sides of the crack, and the two sides of the crack will not be able to be fitted back together without reshaping of the crack walls. A crack of this nature starts at the exposed surface. In the case of unhardened HCC, this is the driest portion. There, the crack is the widest because the surface is the origin and there the tension is greatest and there is no adhesion above to resist the pulling apart. The edges of the crack are often rounded back. If the material is a ceramic clay and the material was baked in a kiln in the cracked condition, it would be obvious in the finished piece that the two interior surfaces of the crack would not fit back together. If the material is a malleable sandy clay, then the crack will go around the sand and the crack surfaces will show that deformation occurred while the material was plastic.

This is entirely analogous to the situation in plastic shrinkage cracking in HCC. The crack occurs while the material is plastic and is then "baked" (shape preserved) by the continuing hydration of the cement and complete hardening of the HCC.

4.3.3 Procedures

Distinguishing between drying shrinkage cracking and plastic shrinkage cracking is a five-step procedure, as shown in Table 4-1.

1. Study the intersection of the side walls of the specimen with the wearing surface. Plastic shrinkage cracking may occur before or just after the surface is screeded and the wearing surface texture created. In these cases, it may happen that one or more of the specimens submitted may show that mortar was worked into the top of the crack. Sometimes, portions of the plug of mortar will have fallen out or been chipped off the specimen during coring and handling. It is clear that the plug is a definite indication of early plastic shrinkage cracking (see Fig. 4-1).

Table 4-1
PROCEDURE—DISTINGUISHING BETWEEN PLASTIC AND DRYING SHRINKAGE CRACKING

1.	Study the intersection of the side walls of the specimen with the wearing surface.
2.	Study the wearing surface of the specimen.
3.	Study the expression of the crack on all cut, cored, and lapped surfaces, and focus down into the crack.
4.	Examine the interior surface of the crack.
5.	If necessary, study the type of voids present in other areas of the HCC and compare their surface with various types of surfaces within the crack.



Figure 4-1 PLASTIC SHRINKAGE CRACKING. Was covered up by mortar filling over it in top of a 4-in. core. Mortar was worked into the top of this set of cracks before the concrete hardened. Later, the concrete cracked in the same area. Due to lack of interlock, the new crack follows the mortar boundaries. At the right side of the picture is a depression in the surface where a mortar plug was lost before the photograph was taken. (This specimen had to be glued together to enable the photograph to be made.)

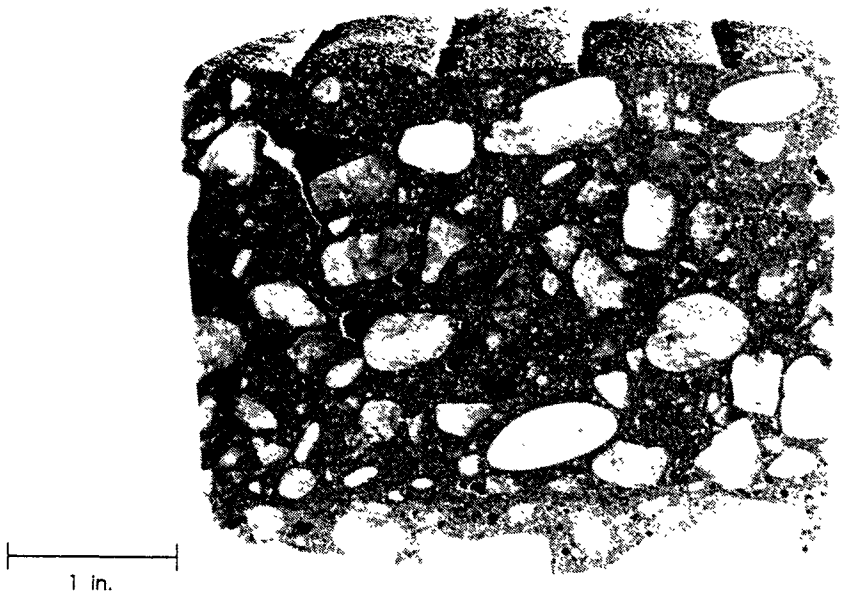


Figure 4-2 PLASTIC SHRINKAGE CRACKING. Occurred before a surface texture was formed. The defect became hidden by the finishing procedures that pushed unhardened mortar over the crack.

If the cracking occurred just before texturing, the creation of the wearing surface would naturally work mortar into the crack (see Fig. 4-2). If the cracking occurred after texturing and was observed by the workers, a deliberate effort may have been made to improve a wearing surface that was seen to be flawed.

2. Study the wearing surface of the specimen. With the microscope, look for features that will define the crack. Check for any features that will indicate the nature of the cracks. If photographs can be obtained of the wearing surface just after cracking but before the surface is worn by any traffic, small bridges of material may be seen stretching across the crack, which will indicate plastic shrinkage cracking (see Fig. 4-3).

3. Study the expression of the crack on all cut, cored, and lapped surfaces, and focus down into the crack. If any little bridges of paste are seen that tie the two sides together or if any uncracked aggregate particle bridges the crack, the crack plane existing all around the particle, and the aggregate firmly attached to the hardened paste on either side, it can be concluded that the crack was caused by plastic shrinkage (see Figs. 4-4 and 4-5). Bridges of paste or aggregate are not found in all plastic shrinkage cracks. The defining feature is the fact that the sides of the cracks have been distorted by the tensile forces due to the shrinkage that occurred while the HCC was still malleable.

The greater the difference between the width of the crack at or near (beneath any mortar plug) the wearing surface and the width of the crack at its deep end and the more quickly it tapers to nearly nothing, the greater is the likelihood of the crack having been caused by plastic shrinkage. The *V* shape of the crack is part of the distortion of the edges of the crack caused by the tensile forces. Commonly, the

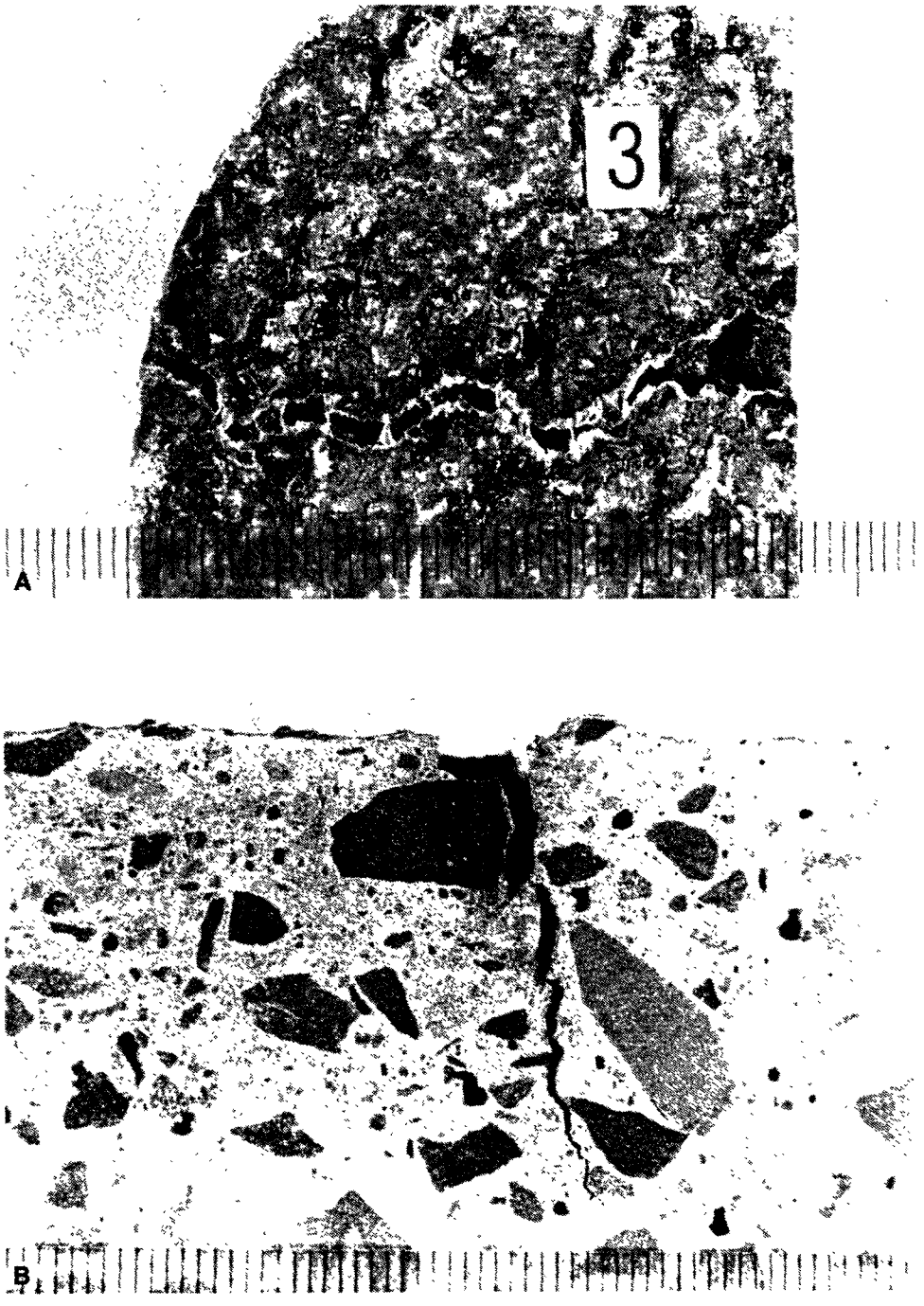


Figure 4-3 PLASTIC SHRINKAGE CRACKING. Occurred in a latex-concrete overlay. The crack shows the distortion that accompanies tension cracking in an unhardened medium. This specimen was collected before traffic destroyed these delicate structures. The scales are in millimeters. **A.** Top view of core. Little threads of latex span the crack. **B.** Little bridges of latex paste connect sides of crack.

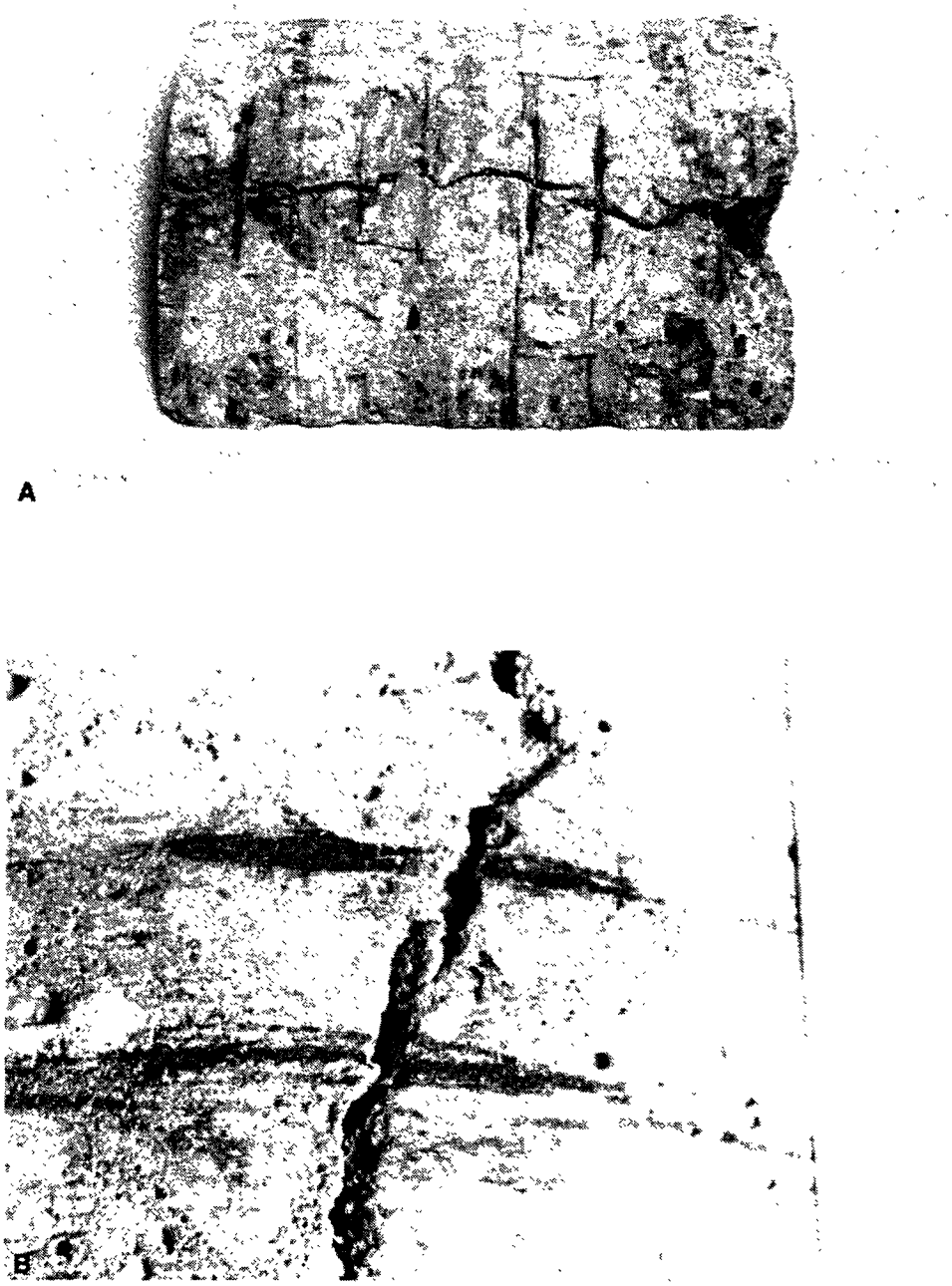


Figure 4-4 BRIDGE OF PASTE ACROSS PLASTIC SHRINKAGE CRACK. A. A little bridge of paste across a crack caused by shrinkage that took place while the ordinary portland cement concrete was unhardened (4-in. core; road surface at right). These little bridges may occasionally be found on broken, sawed, cored, or lapped surfaces. **B.** Close-up of bridge. The scales are in inches.

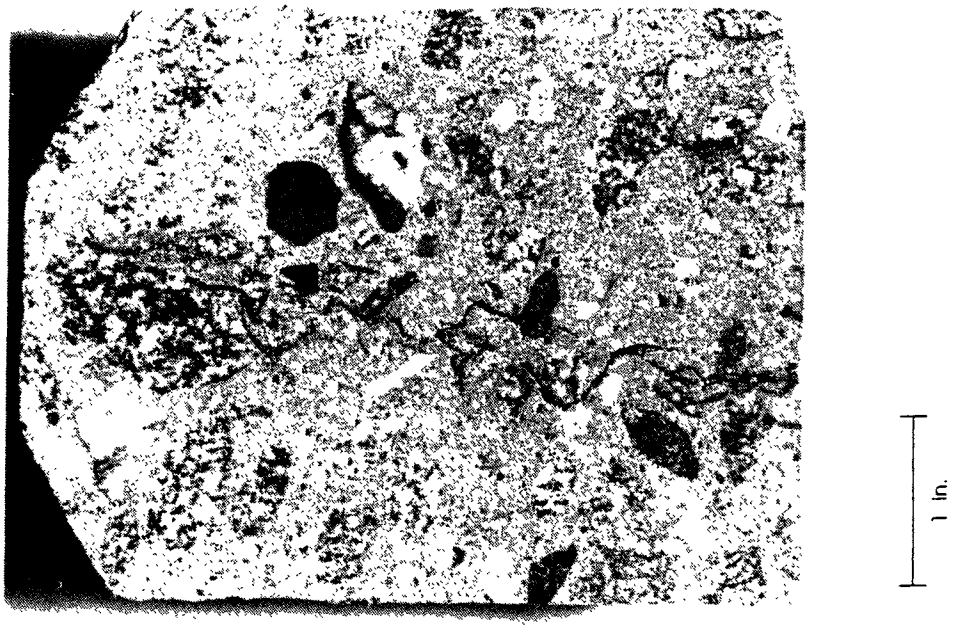


Figure 4-5 PLASTIC SHRINKAGE CRACK. Tortuous path of a plastic shrinkage crack in a concrete that has not completely separated. It is easy to envision how little bridges of concrete form when a crack system of this nature is stretched further. Narrow microcracks are emphasized with ink.

crack is a plastic shrinkage crack near the surface, but with depth, the crack is straighter and the zone of weakness has been extended after the final set by other forms of drying shrinkage or by structural stress, sometimes completely through the slab.

The path of a plastic shrinkage crack on any vertical concrete surface is usually quite tortuous because it is deflected by each piece of aggregate. Typically, the crack is wider at the top than at the bottom, but even at the bottom, it is not the sort of crack found in a brittle ceramic (see Fig. 4-6).

Do not be confused or alarmed by the presence of abundant road dirt or mud created by the drilling of the cores. This mud may appear layered and flaky, but the structures will be fragile.

4. Examine the interior surface of the crack. Delay any destructive procedures until the total surface has been studied and any air-void determinations have been made. Then, break the HCC with a hammer blow in an area away from the crack, and examine the surface produced. In the most common case of a crack produced in an area not including a plastic shrinkage crack, the interior surface will be made up of a multitude of tiny angular planes with the exception of the interior of air voids. There may be an occasional rounded surface of an aggregate particle or of the cast of an aggregate particle, but the major portion of the surface will be a collection of planes.

Break the HCC on the crack, and examine the interior surface. Usually, the interior surface of a crack produced by plastic shrinkage is made up of very tiny globules of cement paste. The surface of the paste inside the crack does not look like a cracked surface. Rather, the paste appears, as one might imagine it would, as if,

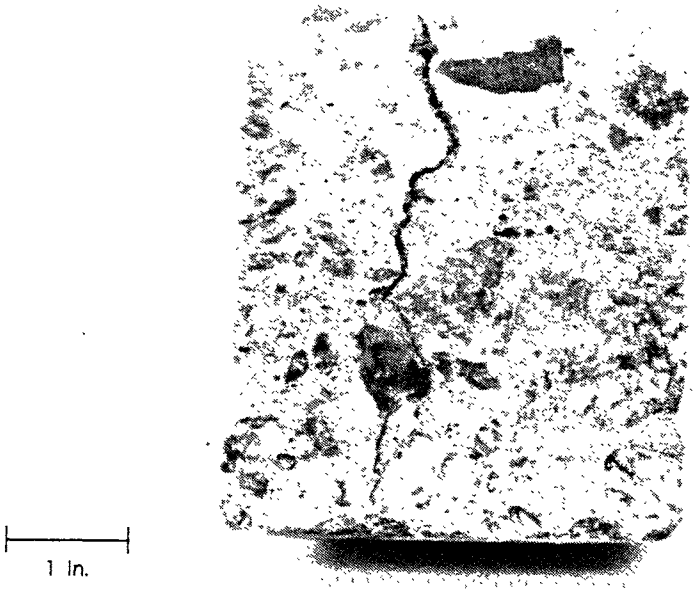


Figure 4-6 PLASTIC SHRINKAGE CRACK. On a lapped surface. The wearing surface is at the top of the photograph.

when the sides of the crack were no longer in contact, minute stringers of paste had momentarily bridged the crack and then as the tensile strength of the stringers was exceeded the stringers coalesced into tiny globules. Each globule appears as if a paste stringer shrunk in on itself and made itself equant. If it is a water-worn sand, none of the sand grains will be broken and they will present a natural rounded surface. A few fragments of the coarse aggregate may be broken, as will occur in the fabrication of all concrete. If the aggregate is shaley or naturally fragile and contains a plane of weakness approximately parallel to the plane of the plastic shrinkage crack, the crack may preferentially traverse this aggregate.

5. If necessary, study the type of voids present in other areas of the HCC and compare their surface with various types of surfaces within the crack.

Chapter 5
PREPARATION OF SPECIMENS

5.1 OVERVIEW

Various methods of specimen preparation are used, depending on the nature of the specimen and the information to be obtained. Smoothly lapped specimens are required for quantitative examination with the stereomicroscope. Small loose particles and many fracture surfaces do not require extensive preparation. Examination with the transmitted light microscopes (i.e., the petrographic and P/EF microscopes) requires very thin specimens mounted on glass or very small grains of the specimen mounted in an index of refraction oil or epoxy. The fluorescence features of the P/EF microscope necessitate that the specimen be not only very thin but also impregnated with an epoxy containing a fluorescent dye. Large specimens of aggregate rock can have their properties displayed best through sawing, lapping, and etching. It is often necessary to crush such material for use in specific tests. Bulk specimens of sand, gravel, and crushed aggregate usually require reduction in overall volume and separation into size-graded fractions.

5.2 SLICES

There are three types of slices prepared for stereomicroscopic examination: (1) basic lapped slices, (2) vertical sections of horizontal slices (or vice versa), and (3) acid-etched slices.

5.2.1 Basic Lapped Slice

5.2.1.1 Overview

The determination of the components, ingredient boundaries, reaction products, and parameters of the air-void system requires the use of a stereomicroscope (see Fig. 2-16). The types of items perceivable with the stereomicroscope are described in Chapter 8. Because it is impossible to focus clearly on a rough surface at the magnifications required for the petrographic examination of HCC with a stereomicroscope, it is necessary that smooth (finely lapped) surfaces be prepared on sawed slices of the specimens. The smoother the saw cut, the easier the lapping routine will be.

Unless specific precautions are taken to avoid it, undercutting is common during the production of finely lapped surfaces. It is the process in which softer constituents are removed more rapidly than harder constituents so that the harder material (usually the aggregate) stands up higher than the softer components (see Fig. 5-1). During the lapping process, the slice rides on the high, hard portions and grinding compound collects under the soft portions. Hard aggregate standing above an undercut paste has a rounded edge, and the area of visible aggregate is increased. There is no sharp line or change in surface texture at the point where the theoretical flat-lapped surface changes to a slope down into the paste. No exercise of logic or mental reconstruction of what the surface should be will allow an accurate

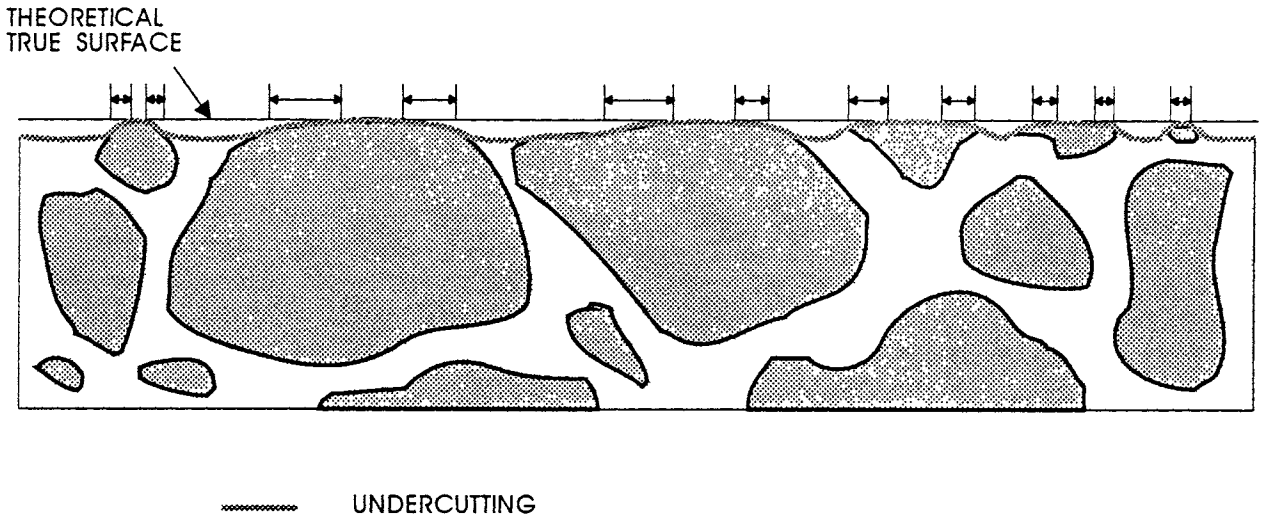


Figure 5-1 UNDERCUTTING. Where the surface falls below the theoretical true surface, the paste-aggregate boundary is rounded over and cannot be defined by a view from above, as through a microscope. The length of each questionable area is indicated by *arrows*.

determination of the paste-aggregate boundary or the paste-aggregate ratio in such a slice.

Undercutting is very common on surfaces that are finished on soft polishing laps. It can also occur on hard, cast iron laps but may be greatly minimized with the use of sufficient weight on the back of the specimen to hold the specimen closer to the lap and create additional wear on the high areas. If the specimen is riding on pieces of high, hard aggregate, the buildup of grinding compound in the low, soft portions of the specimen can cause a general rounding of the surface contours, loss of the ability to distinguish boundaries accurately, enlargement of voids, loss of reaction products, and loss of sharpness and clarity on the edges of cracks and other features.

5.2.1.2 Procedures

Producing a lapped slice is a five-step process, as shown in Table 5-1. Further details on the preparation of surfaces for microscopical analysis may be found in many references, e.g., ASTM C 856 (petrographic examination) and ASTM C 457 (determination of air-void system).

Table 5-1
PROCEDURE—PRODUCTION OF BASIC LAPPED SLICE

1. Cut and shape the slice.
2. Strengthen fragile HCC.
3. Lap the slice.
4. Clean the slice.
5. Check the quality of the lapped surface.

1. Cut and shape the slice. If the specimen is a full-depth core or an irregular fragment of HCC, it may be so large that it cannot be held in the vise of the large oil-cooled saw (see Fig. 2-4B). In this case, bring the specimen down to a more reasonable shape and size by using the large water-cooled saw (see Fig. 2-3).

Cut a slice of the HCC from the specimen according to the plan for petrographic analysis as devised in Chapter 3. The oil-cooled saw produces smoother surfaces that require less lap time than do surfaces produced with a water-cooled saw. As mentioned in Chapter 3, label all fragments of the original specimen to identify their origin and, whenever possible, their orientation. A slice thickness of 7/8 in. is recommended, but inspect the material after the first cut to make sure that a thicker slice is not required because of the presence of a previously unseen crack or other weakness. If the specimen crumbles or cracks into several fragments after the first saw cut, you may have to cement the specimen back together with a strong epoxy before you can shape a slice.

The slice must usually be trimmed to fit the slice holder of the lapping device. Most holders are circular to allow free rotation. Be careful *not* to trim away any portion of the area of greatest interest to the client. Often, the portion of greatest interest will be the top, or wearing, surface. In such cases, any trimming will have to be done on the bottom. If there are no obvious differences to be seen between the top and the bottom of the core and the section does not exceed the diameter of the specimen-holding ring of the lap (6 in. for VTRC equipment) 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.

2. Strengthen fragile HCC. 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 hr. For most intensive carbonation, the cut specimen can be supported for 10 hr over water that is kept agitated by dry ice. If the container is loosely covered, the carbonation will continue until all the carbon dioxide is dispersed, even after the dry ice has completely sublimed away.

Very fragile specimens may require impregnation. A number of materials and techniques of impregnation are available. Carnauba wax is recommended in ASTM C 457, but it must be used with safety precautions to prevent explosion during the heating to remove excess wax. Roberts and Scali (1984) recommended the use of a 1:5 solution of colorless nylon fingernail hardener in acetone. They are presently using a somewhat safer preparation of the nylon nail hardener in methanol (personal communication). They prefer the commercial nail hardener to a solution of pure nylon because some of the softening ingredients in the commercial preparation are valuable in preventing chipping. The nylon solution may be applied to the HCC at any stage of the lapping as seems necessary. The hardener is brushed on the surface until it fills the cracks and thoroughly coats the interior of the voids. The hardener is allowed to dry before lapping is continued. Very fragile HCC may require the use of the hardener during all stages of the lapping; other HCCs will require the hardener during only the last portion of the lapping procedures. Before examination and determination of the air-void parameters, the hardener should be removed by soaking in the appropriate solvent until the plastic shine is dissolved away. Choice of an impregnating agent should include consideration of the desirability and difficulty of removing the impregnating agent from the surface to be examined.

If epoxy or another permanent substance is used for impregnation, it will become a lasting part of the specimen, prevent the detection of subtle textural differences in the HCC, and obscure the viewing of reaction products and internal surfaces. This may or may not be important, depending on the purposes of the investigation for which the specimen is being prepared. Various techniques may be used to impregnate HCC slices with numerous types of substances. The slice may be soaked in a series of increasing concentrations of various types of impregnating agents or vacuum impregnated.

3. Lap the slice. If the surface of the specimen is quite smooth as it comes from the saw, the coarsest abrasive need not be used. If the grooves from the saw are very distinct, any fine aggregate has loosened, or other gross irregularities are present, begin lapping the surface with the coarsest abrasive. Usually, this is done by hand holding the specimen and hand feeding the grit on a small lap (Fig. 2-6).

Once you are satisfied that the surface has been made as flat as possible with the coarsest abrasive and all saw marks are gone, continue the work on the two mechanical laps with the automatic abrasive feed (Fig. 2-7). Place the specimen slice in the slotted specimen holder on the lap. Adjust the yokes so that the grinding slurry does not drop inside the slotted specimen holders and the specimens rotate freely as the lap moves beneath them. Place the cover plate on the back of the specimen, and place any weights on top of the cover plate. Load the abrasive cup with about 2 heaping teaspoons of the appropriate abrasive, and fill it about three-fourths full of lapping oil. Set the clock for 20 min of lapping. Inspect the specimens, and make a judgment concerning the next abrasive. Each abrasive should be finer than the last one used and should be used repeatedly until the marks from the coarser abrasive are gone. A good system is to use one lap for all but the finest abrasive and change to the other lap for the finest one. Lap the surface with successively finer abrasives (see list in Chapter 2) until it is suitable for microscopical observation.

4. Clean the slice. From time to time during lapping, when changing to a finer abrasive, when changing laps, and when lapping is complete, clean all surfaces of the specimen gently and thoroughly to remove the grinding compound and debris. Air voids, cracks, and surfaces of reaction products should be free of such materials.

CAUTION: If the cleaning of lapped slices is not done promptly, the volatile oils will evaporate and the grinding compound deposits will cake and become hard to remove. Therefore, do not allow a specimen that requires cleaning to lose volatile oils or solvents by evaporation.

If cleaning of lapped slices must be delayed, submerge the slice in a solvent, such as 1,1,1, trichloroethane. Caked grinding compound can make viewing of interior surfaces and the crystal forms of the paste and reaction products impossible and contribute to undercutting and destruction of the specimen. At VTRC, a few minutes of ultrasonic cleaning in solvent are routinely used for cleaning all lapped specimens.

CAUTION: 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 (see ASTM C 457).

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

At VTRC, we have not experienced any loss of reaction products or loosening of the aggregates or other surface degradation because of brief ultrasonic cleaning. As an alternative to ultrasonic cleaning, the specimen can be carefully cleaned of grinding compound and loose particles with a soft brush (soft as a baby's hair brush) while it is immersed in a safety approved container of 1,1,1, trichloroethane. It has been reported that a fine, high-velocity spray of solvent (used in an efficient hood) will clean lapped slices efficiently. This procedure has not been tried at VTRC, and we have no evidence concerning the proper pressure and velocity of the spray that would be required to clean without damaging. Whatever cleaning method is used, as much grinding compound and debris as possible should be removed without damaging the surface or any reaction products in the voids.

WARNING: Persons unfamiliar with the hazards of these cleaning compounds are referred to the Chemical Safety Data Sheets published by Manufacturing Chemists Association, Inc., 1825 Connecticut Avenue, NW, Washington, DC 20009, or the Materials Safety Data Sheet that may be obtained from the supplier of the compound.

5. Check the quality of the lapped surface. 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. There should be no noticeable relief between the paste and the aggregate surfaces (see ASTM C 457). Such a surface is shown in Figure 5-2.

If the day is reasonably bright, scan the light from a window as it is reflected at a very low angle from a finely lapped slice. It should be possible to discern objects such as buildings and trees (see Fig. 5-3).

Alternatively, hold the slice 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 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.

The lapped surface needs to show all reaction products, the inner surface of voids, the hydration, etc. in an as-received condition. At VTRC, space and technician time are premium commodities, and the slice used for the air-void determination must be the same surface used for all microscopic evaluations of the concrete sampled and is usually the only slice kept in the archives.

If difficulty is encountered in preparing a lapped surface of the proper quality, a common 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 (as the loose aggregate moves under the surface being prepared) and 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

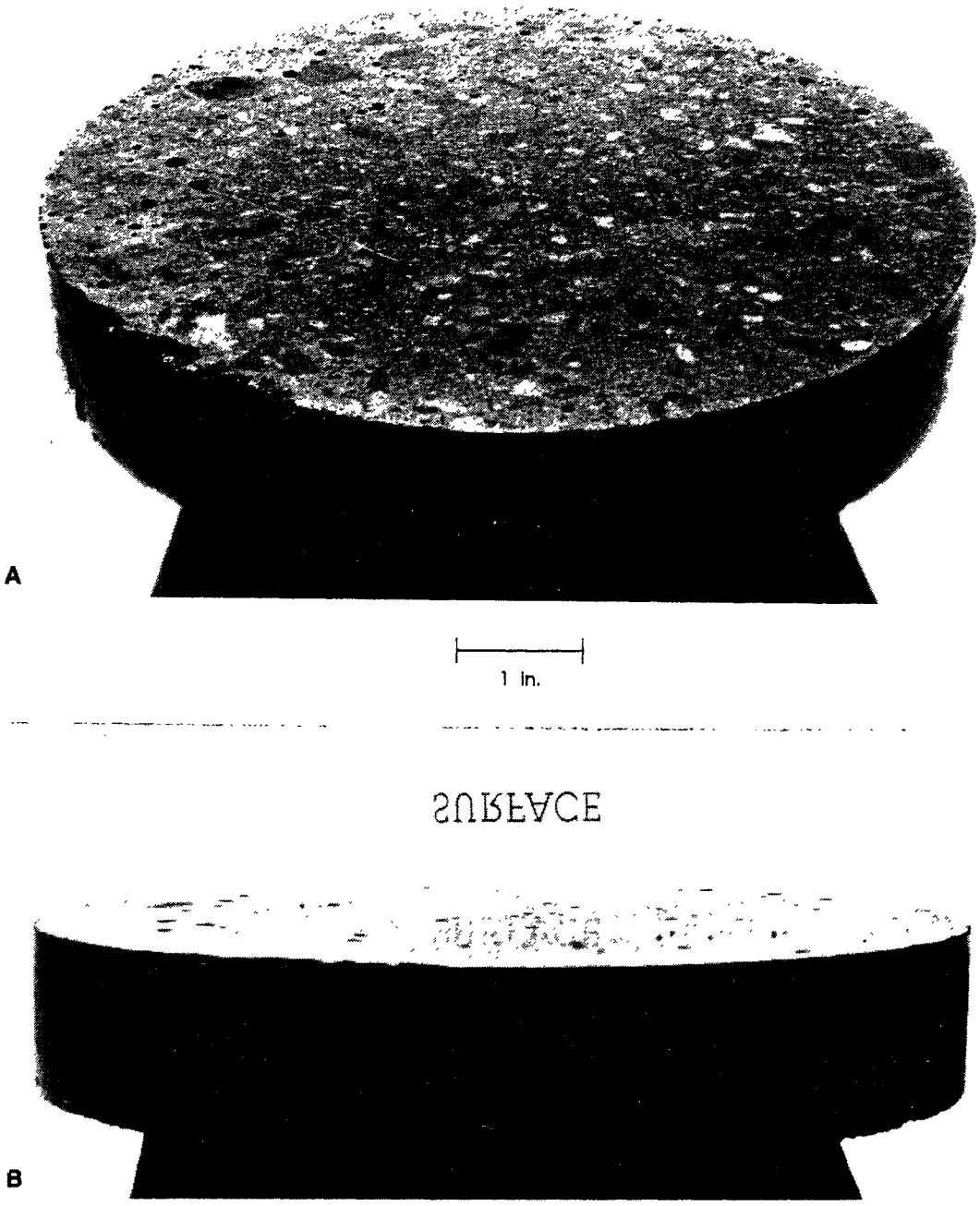


Figure 5-2 WELL-PREPARED SURFACE. Exhibits the quality of the surfaces required for microscopical examination and analyses. **A.** Surface viewed in ordinary illumination. **B.** Surface viewed at a very low angle toward a source of illumination (an illuminated screen). The quality of the reflection 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 5-3 PROPERLY FINISHED SLICE. Same as shown in Figure 5-2. Reflection of outdoor objects as seen on a bright day. The camera was focused on the reflection; thus, to the camera, the slice is out of focus. The human eye sees both in focus.

preparation of the specimen. Often, additional cleaning and more time on the free abrasive laps are all that is required.

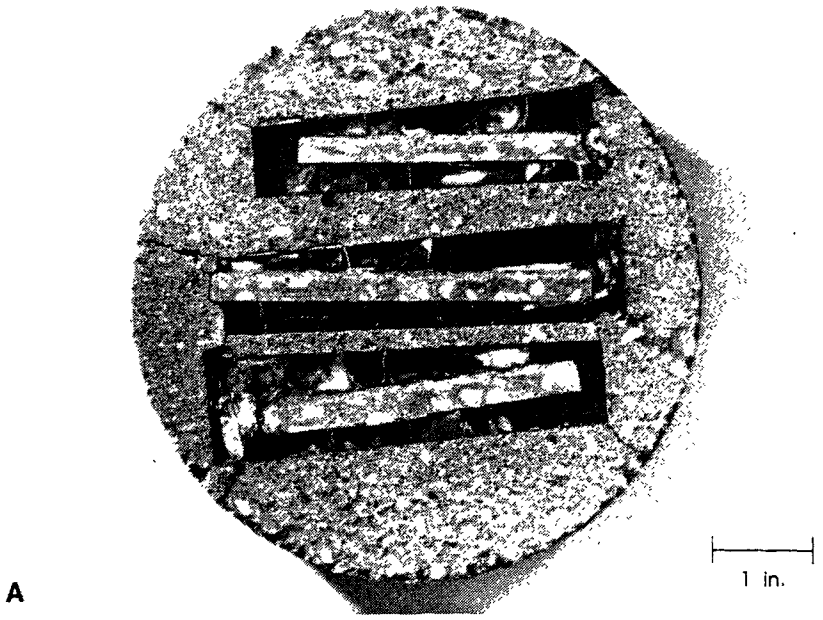
5.2.2 Vertical Section of Horizontal Slice (or Vice Versa)

If a horizontal slice was taken from a core in order to study changes in the air-void system (or other feature) with depth, it may later be desired to lap finely and examine a vertical slice of one or more of the lapped slices already available (or vice versa). In such a case, the slice in question must be secured in an upright position in a mounting medium so that a slice that is at right angles to the direction of the first slice can be cut and lapped (see Fig. 5-4).

5.2.3 Acid-Etched Slice

5.2.3.1 Overview

Etching is performed in order to enhance the differences in acid resistance or solubility that might exist between the phases of a rock or HCC. Some of the phase differences the petrographer might wish to enhance are rhombic dolomite crystals in micritic-carbonate rock, fly ash particles and carbonated areas in HCC, and aggregate particles in HCC paste. Lapped slices of concrete and lapped surfaces on carbonate rock specimens are often etched to make visible and emphasize various phase differences and phase boundaries in the specimen. These differences may be the contrasts between aggregate and paste, various mineralogical differences, and even the differences caused by the depositional and metamorphic history of the rock.



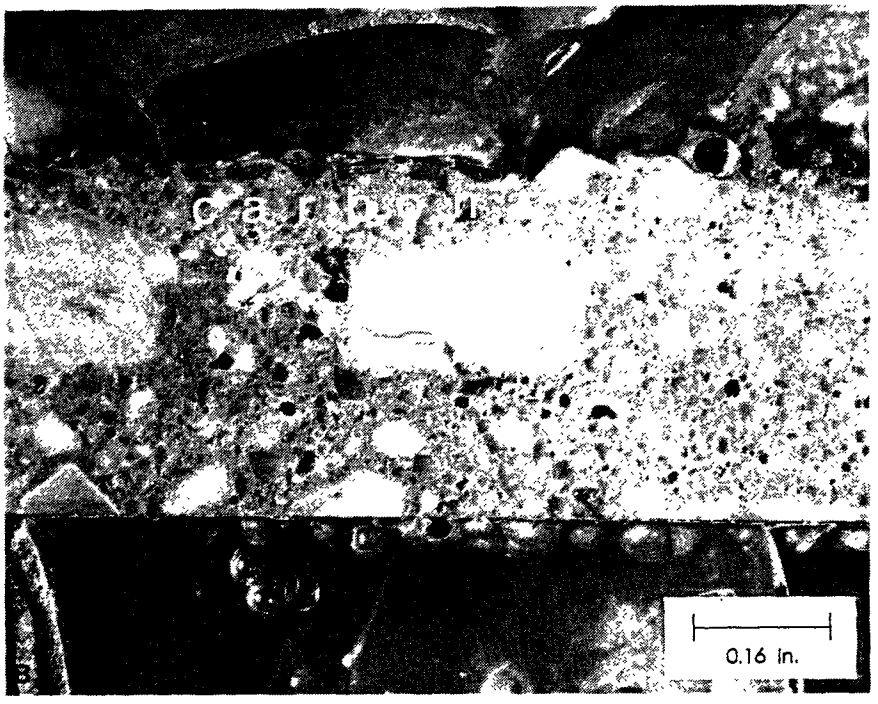


Figure 5-4 SLICE CUT AT RIGHT ANGLES TO ORIGINAL SLICE. A. Edges of thin top surfaces of a concrete core embedded in epoxy and repotted in mortar in a mold 6 in. in diameter and then resliced and finely lapped to allow examination of the vertical surface. In this case, the concrete was slightly etched with weak HCl to delineate the depth of carbonation. **B.** Left portion of middle slice shown in **A** enlarged. The carbonated area is the area above the lower edge of the whiter area near the wearing surface.

The etching of finely lapped specimens of HCC must be delayed until after the specimen has been thoroughly examined and any analysis of the percentage and shape of any constituents that might be changed by the etching is complete (e.g., air). If the etching is being performed to aid in the determination of the paste content for use in the formulas for air-void parameters, the etching and paste determination must be performed on the same surface as was the air-content determination.

5.2.3.2 Procedures

• **Etching a Portion of a Slice.** Etching a portion of a slice is a two-step procedure, as shown in Table 5-2.

1. Ensure that the slice is free of oil. The oil used as a lubricant for the lapping must have been removed from the slice by long exposure or oven treatment. Steam must not form in the HCC; therefore, the temperature in the oven must not exceed 90°C.

2. If the etching of only a small portion of the slice is required and the microscope has a working distance sufficient to prevent splattering of the lenses (or the lenses are otherwise protected), drop 10% HCl acid onto the slice from a rod or dropper while the slice is under the microscope. Observation of the active effervescence of CO_2 from $CaCO_3$ can help locate various mineral species. Take care to distinguish aggregate composed of carbonate minerals from the $CaCO_3$ of the paste caused by exposure to the carbon dioxide of the atmosphere under conditions of moderate humidity.

• **Etching an Entire Slice.** Etching an entire slice is a six-step procedure, as shown in Table 5-2.

1. Ensure that the slice is free of oil. The oil used as a lubricant for the lapping must have been removed from the slice by long exposure or oven treatment. Steam must not form in the HCC; therefore, the temperature in the oven must not exceed 90°C.

2. Flood a shallow, acid-resistant container (e.g., a shallow glass baking dish) with room-temperature 10% HCl acid. Because the acid should be replaced frequently, a shallow bath is recommended.

3. Support the slice (at room temperature) in the acid bath with very small glass, quartz (sand or pebbles), or plastic supports so that the surface does not touch the bottom of the container and the lapped surface is submerged. (If the slice

Table 5-2
PROCEDURE—ETCHING OF SLICE

Etching Portion of Slice
1. Ensure that the slice is free of oil.
2. Drop 10% HCl acid onto the slice while the slice is under the microscope.
Etching Entire Slice
1. Ensure that the slice is free of oil.
2. Flood a shallow container with room-temperature 10% HCl acid.
3. Support the slice in the acid bath.
4. Move the slice around for a few seconds.
5. Rinse, dry, and microscopically determine if the procedure should be repeated.
6. After the etching is complete, rinse the surface with tap water and partially dry it.

is not too large or heavy, it may be hand held. The weak acid will not damage healthy skin.)

4. Move the slice around or vibrate it or agitate the acid while the slice is in contact with the acid for a few seconds.

5. Rinse the slice, partially dry it, and microscopically determine if the procedure should be repeated. If the etching is insufficient, return the slice to the acid bath and repeat the procedure. It is generally satisfactory to perform the etching for 5 to 20 sec. The exact time used depends on the maturity and condition of the HCC and the features to be examined.

6. After the etching is complete, rinse the surface with tap water and partially dry it. Drain the slice onto a paper towel, and pat it lightly with a tissue. The slice will usually air dry sufficiently for microscopic examination in 1 hr but may be dried in a slightly warm oven.

5.3 THIN SECTIONS

Thin sections must be thin enough for sufficient light to pass through them to enable the microscopist by using the particular features of the microscopes (see Chapters 12 and 13) to identify the various parts of the specimen that make up the whole, their nature and present condition, and any changes that have occurred since their original formation. Thus, the sections should be as thin as is practical.

Instructions for fabricating standard thin sections from rock for examination with the petrographic microscope are available in many good books on optical mineralogy or petrology and are usually included with any equipment purchased for this purpose. Special instructions for preparing thin sections of HCC, thin sections for study with fluorescent light, and thin sections of particular aggregates are included here.

5.3.1 Basic Thin Section

5.3.1.1 Overview

The thickness of thin sections fabricated for the usual geologic/petrologic/mineralogic examination of rocks and minerals is 25 to 30 μm. If rock or rocklike specimens are sent to a company that fabricates thin sections without further instructions, the thin sections produced will be 25 to 30 μm. For the determination of the component minerals in coarse-grained igneous and metamorphic rocks, this thickness generally works fairly well. Some rock-oriented petrographers depend on thin sections being a standard thickness so that they can more easily 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 individual particles of interest is less than the thickness of the thin section, the particle boundaries tend to become obscured by the overlap of the particles. The finer the size of the particles, the more difficult it is to observe and define the boundaries. The less the difference between the various optical properties of the particles (index of refraction, color, and birefringence), the more difficult it becomes to identify the boundaries and determine the identity of the particles.

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 thickness. These rocks are composed of rhombic crystals of dolomite (5 to 20 μm across) "floating" in a murky, calcite micrite. The murky appearance of the background is caused by submicroscopic particles of organic material, iron oxides, sulfides, and various other insoluble substances that are included in the micrite. When these materials include a relatively high percentage of opaque material, it can be impossible to discern details in a thin section as thick 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 in thickness, but much of the reactive quartz present in metabasalts, mylonites, and siltstones is exceedingly fine grained and cannot be detected in thin sections of standard thickness.

Most cement is very fine. The greater portion will pass a No. 200 (75 μm) mesh sieve. The cement hydrates into even finer particles of low to negligible birefringence with nearly identical color and low indices of refraction. Observation 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 groundmass. The term *groundmass* has long been used by geologic petrographers to indicate that the material is too fine for determinative microscopy. Examples of groundmass are the submicroscopic clay background of some sedimentary rocks and the material between phenocrysts in lavas or very fine-grained basalts.

Ideally, thin sections should be flat and cover a large portion of the glass on which they are mounted. Unless special equipment (not available at VTRC) is used, the flaws of 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.

5.3.1.2 Procedures

Because these specimens are so thin, they must be mounted on glass microscope slides. Because of the thinness, the chance of lapping too far and losing the entire thin section is great. Table 5-3 lists the necessary steps.

1. Prepare the specimen. Begin fabrication with a number of shaped pieces of specimen so that the likelihood of two or more pieces surviving the procedure is good. Shape these blanks slightly smaller than the size of a petrographic glass slide with a small thin-bladed rotary diamond saw (Fig. 2-5). If ultrathin fluorescent sections are being produced, size the shaped pieces to fit the well of the slide.

2. Prepare the glass slide. Ensure that the two sides of the flat glass slides are parallel and that the slides are all of the same thickness. This is accomplished by

Table 5-3
PROCEDURE—PREPARATION OF BASIC THIN SECTION

1. Prepare the specimen.
2. Prepare the glass slide.
3. Make the first thickness reduction.
4. Make the second thickness reduction.
5. Perform the final finishing.

mounting the slides in the vacuum chuck of the grinder (see Fig. 2-11) and grinding them to a standard thickness as measured on the dial on the right side of the machine. If this is done, it then becomes possible to use the reading on the dial as an indicator of the thickness of the specimen and thus to grind all thin sections to a standard thickness before the hand lapping is begun. The matte surface produced by the grinding of the slide to parallelism and standard thickness may provide a less slippery surface to which the mounting epoxy can cling. If it appears that the cutting and grinding equipment has lost its accurate parallelism between the face of the chuck and the saw or cupped grinder, place a mark on the glass slide indicating the orientation of the glass on the vacuum chuck so that, after the specimen is mounted, the glass may be replaced on the chucks in the same position and thus compensate for any errors in the equipment itself. The 25-year-old equipment at VTRC is still free of this sort of deterioration.

3. Make the first thickness reduction. Mount the piece of specimen for the thin section on the prepared glass slide. Place the glass slide on the vacuum chuck of the cutoff saw (see Fig. 2-11A), and use the handle to move the work into position up against the saw to remove all but 1 μm or less of the specimen. The optimum thickness will depend on the physical stability of the specimen. 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.

4. Make the second thickness reduction. Transfer the specimen chip on glass to the vacuum chuck on the grinder. Gradually advance the thin-section blank to the grinder by using the thickness measuring dial at the right-hand end. An attempt to grind off a great thickness all at once will induce stresses and may cause breakage or debonding of the specimen and the glass.

5. Perform the final finishing. After the grinder has reduced the section to about 30 μm in thickness, hand finish the section with No. 600 SiC and 5 μm Al_2O_3 in a water or oil slurry on a glass plate (see Fig. 2-12) until the section is approximately 25 μm or less in thickness, depending on the material being sectioned and the purposes of the examination. The grinding motion used should be that of making the 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 *8*s should move over the glass surface and rotate, crossing as many different places on the glass as possible.

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

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 fluid lubricant is used.

5.3.2 Thin Section for Detecting Alkali-Reactive Textures in Carbonate Rock

5.3.2.1 Overview

Because the most reactive textures in carbonate rock contain dolomite crystals that are very small (less than 25 μm across), it is necessary to make the thin section used to discern the reactive texture thinner than the usual 25 to 30 μm so that the tiny rhombs of dolomite (often only half the thickness of a standard mineralogical thin section) can be seen without being obscured by other overlapping phases.

5.3.2.2 Procedures

The thin section may be lapped down to zero thickness at one end and wedge toward a more standard thickness at the other (see Fig. 5-5). This may be accomplished by pressing more heavily on one end of the back of the section during the final finishing. By this method, there will exist at least a small area that is of the correct thickness to allow visibility of very small dolomite crystals. The pressure on the back of the section should be kept nearly evenly distributed until the section is down to about 25 μm in thickness.

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. Experienced technicians may thin the entire section to the thickness best suited for viewing, but the likelihood of loosing the entire section is very great.



Figure 5-5 THIN SECTION THINNED TO NOTHING ON ONE END. For observation of dolomite rhombs in micrite as an indicator of possible alkali-carbonate reactivity. The glass mount is 27 by 46 μm.

I estimate that the best compromise thickness for 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.

5.3.3 Thin Section Showing Profile of Wearing Surface

5.3.3.1 Overview

On occasion, the exact details of the texture and the aggregates of the wearing surface of an HCC pavement require examination and study with the petrographic microscope (Webb, 1970).

5.3.3.2 Procedures

Cement two small portions of the surface under study to a slide with the wearing surfaces together in the center (to protect the area of interest from the greatest thinning action), and produce a thin section of the thickness appropriate for the material and the purposes of the study. Figure 5-6 illustrates some of the steps in this process.

5.3.4 Thin Section for Epifluorescent Illumination

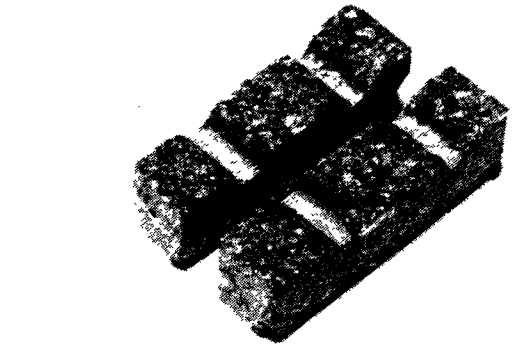
5.3.4.1 Overview

Sections for use with epifluorescent illumination (see Chapter 13) are much like ordinary thin sections. The person fabricating the thin sections should become familiar with the material in 5.3.1. (The welled slides used for fluorescent thin sections require no preparation.) The major difference is that the thin sections are impregnated with a fluorescent-dyed epoxy (Walker & Marshall, 1979; Wilk, Dobrolobov, & Romer, 1974). In these sections, the light emitted by the fluorescent dye is not collimated by the condenser because it is produced in the section, closer to the objective lens than is the condenser. Therefore, the thinness of the section is of great importance in preventing the obscuring of fluorescent details with fluorescent light from greater depths. Because the section is of concrete (usually with considerable porosity) that has been impregnated with a dyed epoxy and properly cured, it is not so difficult as might be thought to produce sections of 15 μm or less in thickness.

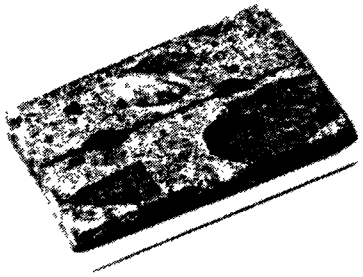
5.3.4.2 Procedures

The methods used by VTRC to fabricate fluorescent thin sections (Walker & Marshall, 1979) were those that would involve the least cost for equipment. The Ingram-Ward thin-section equipment was already available, and the major purchases were the vacuum oven and the Syntron vibratory polishing lap.

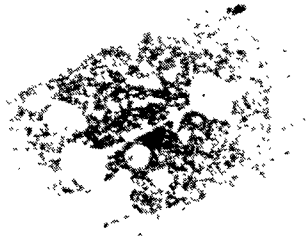
It was found necessary to make sure that the most impregnated side of the thin-section blank was very flat and was cemented down to the final slide to be used because the impregnation was usually only about 1.5 μm in depth. With the Ingram-Ward equipment, the use of a series of work glasses was necessary. Other laboratories could easily devise other methods of producing these sections, depending on the equipment available to them.



A



B



C

Figure 5-6 STEPS IN PREPARING THIN SECTION TO SHOW DETAILS OF WEARING SURFACE. Actual size. **A.** Small blocks of wearing surface. **B.** Blocks cemented to microscope slide. **C.** Thin section of blocks. This section is about 50 μm in thickness because the study for which it was fabricated was concerned with the profile of the wearing surface and not with the identity and interrelationship of the component materials.

The fabrication of fluorescent thin sections is a 23-step process, as follows. When not in use, store the specimen in a dark, airtight cabinet (to prevent fading of the fluorescence). To prevent carbonation, use an effective desiccant, such as Drierite, and a carbon dioxide absorbant, such as Ascarite.

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 by 32 mm to fit the welled area of the final mounting glass.

2. With the small diamond blade saw, and preferably an oil lubricant, cut a small specimen about 4 mm in thickness from the specimen area. Extremely fragile specimens may necessitate thicker specimen chips.

3. Rinse the specimen well in acetone or 1,1,1, trichloroethane, and allow to dry. Carefully, so as to ensure parallelism, mount the specimen on a flat work slide with a mounting epoxy.

> *WARNING: 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, NW, Washington, DC 20009, or to the Materials Safety Data Sheet that should be obtained from the supplier of the compound.*

4. Mount the glass slide in the vacuum chuck of the Ingram-Ward grinder, and grind the specimen chip with the cupped ceramic diamond grinder until the entire surface of the specimen is free of saw-damaged material.

5. Wash by soaking in four changes of acetone over a period of 2 hr to remove the lubricant oil. Air dry.

6. Dehydrate the preparation overnight in a vacuum oven at 80°C. Maintain a vacuum of 10 mm Hg for about 1 hr before leaving for the night. In the morning, turn off the heat and allow the closed oven and slice to come to room temperature before releasing the vacuum.

7. Pot the specimen with the ground surface up (glass down) in a container of liquid fluorescent epoxy to which has been added some 0.3 Linde (see Chapter 2) to protect the edges during later polishing. At VTRC, a disposable plastic Petri dish (5-cm diameter) is used as a potting container. (If the corners of the work glass have to be snapped off to make the preparation fit in the dish, a second work glass will have to be cemented onto the back of the first one before the preparation can be held in a vacuum chuck again.) The use of a larger container would waste epoxy.

8. Place the container in the vacuum oven at room temperature, and bring the vacuum intermittently and slowly down as low as possible (10 mm Hg). Keep it there until outgassing stops. This step is best done in stages to prevent overflow of the bubbling epoxy (see Figure 2-14B) and allow the impregnating epoxy time to penetrate the slice. Release the vacuum, and allow the air pressure to force the epoxy into all portions of the void system. Repeat the procedure as many times as necessary until very few bubbles form while the preparation is under a vacuum. On the last treatment, continue the vacuum until outgassing essentially stops. The entire procedure can take up to 3 hr.

9. Cure the epoxy in a warm oven overnight and then at room temperature for 36 hr or until the epoxy is hard and brittle and does not show any plastic-

ity when tested with a pick. There must not be any possibility that the epoxy in air voids or cracks will become distorted and drag onto adjacent phases. If experimentation concerning the curing is performed, be aware of the fact that subjecting the epoxy to a vacuum may remove some of the more volatile components of the epoxy mixture and may thus change the curing regime required. Treat any experimental curing slices the same as any impregnated material.

It is necessary that the lapping procedures to follow remove the impregnating epoxy by the same infinitesimal chipping method as used for removing the aggregate rock and cement paste.

10. Trim excess hardened epoxy, plastic (from the container), and glass from around the sides of the specimen with the small rotary saw. With a knife, snap off the plastic. Peel the external hardened epoxy from the bottom glass of the impregnated slab. Attach an additional work glass with quick-set glue. (This work glass is required for the next step to ensure that all the ports of the vacuum chuck are fully covered and working efficiently.)

11. Clean the final mounting surface by placing the new work glass on the vacuum chuck of the Ingram-Ward grinder and carefully grinding off the hardened epoxy coating on the surface of the specimen. The sound of the grinding will change when the epoxy layer is gone. Remove as little of the specimen as possible. This highly impregnated area is the best portion to use as the thin section.

12. Lap the mounting surface by hand with a 5 μm Al_2O_3 water slurry on glass.

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

13. Clean the specimen in an ultrasonic cleaner with acetone solvent.

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

14. Place the specimen under a 10-mm vacuum at 50°C for 3 hr or more to remove all oil and water. (Do not use heat. It may soften the epoxy.)

15. Mount the specimen, with the lapped impregnated surface down, in a welled petrographic slide with an undyed epoxy. Perform the mounting carefully to eliminate air pockets and ensure a good bond.

16. Firmly, but without destructive force, clamp the specimen to the slide and allow it to cure overnight. Be careful to limit the forces to those that will clamp the specimen to the glass. Do not twist or slide the specimen relative to the welled glass slide. The clamp stand used for this procedure is shown in Figure 2-15.

17. Cut off the excess thickness of the specimen by mounting the welled slide containing the specimen against the vacuum chuck of the Ingram-Ward saw, as shown in Figure 5-7. The specimen will be made reasonably thin, and the two work glasses removed.

18. Reduce the specimen to about 30 μm in thickness in the Ingram-Ward grinder.

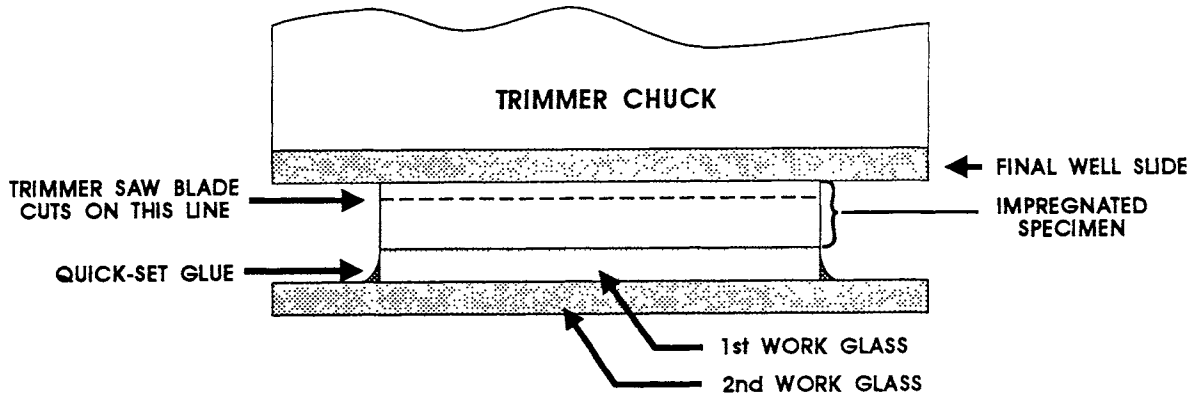


Figure 5-7 SPECIMEN MOUNTED BETWEEN WORK GLASSES AND WELLED SLIDE. The *dotted line* indicates the plane the saw will cut.

19. Rinse the specimen well with acetone or 1,1,1, trichloroethane.

20. Lap the specimen by hand with a 5 μm Al_2O_3 and water slurry on glass. (Refer to instructions in 5.3.1).

21. Clean the specimen in an ultrasonic cleaner with acetone.

22. Attach the glass of the specimen slide to the flat side of the weights of the Syntron vibratory polisher with strong double-sided adhesive tape (carpet tape). Place rubber bumper rings on the weights. Spread the Pellon pad in a bowl on the vibrator with a slurry of diamond compound (the coarser first) and lapping oil. Place the weighted specimen down on the treated Pellon pad (see Fig. 2-16), and activate the vibrator for as long as necessary to reduce the specimen to the required thinness and produce a very finely textured flat surface free of scratches. The vibration may require 8 to 16 hr. Certain types of concrete with especially resistant aggregate will require further polishing to remove scratches. In such a case, thoroughly clean the specimen and weights and repeat the vibratory procedure with 1-μm diamond compound in a second Syntron bowl. The use of a second bowl makes it possible to save the first Pellon pad and associated diamond compound for later use.

23. Clean the specimen carefully with acetone or 1,1,1, trichloroethane.

24. When not in use, store the sections in a dark (to prevent fading of the fluorescence), airtight cabinet. To prevent carbonation, use an effective dessicant such as Drierite and a carbon dioxide absorbant such as Ascarite.

5.4 GRAIN MOUNTS

5.4.1 Overview

Small particles of rock, paste, reaction products, and sand grains can be identified and examined with the petrographic microscope, but they cannot be examined unmounted. The relief between the index of refraction of the substance and the index of refraction of air is so great that the internal reflection within the particle prohibits observation of any useful properties. Therefore, it is necessary to prepare mounts in which these particles are submerged in a transparent substance with an appropriate refractive index. Such mounts are called *grain mounts.* Temporary grain mounts are usually made with oil for the purpose of determining the index of refraction and other optical properties of small particles of material. The oil can easily run off the mount, and, in time, will evaporate. Permanent grain mounts are made with epoxy and can be kept indefinitely for demonstration and archival purposes. Permanent grain mounts can be thinned in the Ingram-Ward equipment as are thin sections (see 5.3.1) to increase the transparency of the particles and facilitate the observation of many of the optical properties of the material.

5.4.2 Procedures

Grain mounts are more satisfactory if the particles in any one mount are nearly the same size. Use the 3-in. sieves to separate the sizes. If the examination is concerned with the relative amounts of the sizes, weigh each sieve fraction.

5.4.2.1 Temporary Mount

1. Crush or size the particles so they may be distributed evenly on a microscope slide.

2. Place a drop of index of refraction oil on or very near the particles and mix the fragments and the oil with a needle.

3. Drop a small thin cover slip on the preparation.

4. Manipulate the mixture to remove bubbles by moving the cover slip around by means of a clean pencil eraser on a hand-held pencil. If the particles are so large that the cover slip is not in contact with the oil across the total preparation, either add more oil or discard the grain mount and begin again with smaller particles. If the grain mount is being made to aid in the determination of the index of refraction, it is best to start with a supply of particles less than 25 μm in diameter. A number of grain mounts using different oils will probably be required, and, in the interests of economic use of the expensive oils and neatness, each should be as thin and small as is practical. The orientation of the individual grains may be altered by moving the cover slip and thus rolling the fragments.

5. Label each preparation. It is easy to forget which is which when several are on the table beside the microscope. Temporary labeling may be accomplished by placing the grain mount over a label on a sheet of paper. Such preparations may also be used for cursory examination of reaction products when the information needed is easy to obtain, such as birefringence.

5.4.2.2 Permanent Mount

Fabricate a permanent grain mount with epoxy, Canada balsam, or a similar substance rather than an index of refraction oil. Such mounting materials may be used purely to preserve the specimen or provide a secure base so that once the mounting material is properly cured the preparation can be thinned and finely lapped as a thin section of the material.

5.5 AGGREGATE SPECIMENS

5.5.1 Overview

Rock as large fragments, crushed and sized for use, or as sand and gravel may be submitted to the concrete petrographer in order to determine its suitability for use as aggregate in HCC. Specimens may be submitted as part of ongoing research concerning some form of HCC deterioration or for comparison with the aggregate found in HCC specimens presently undergoing investigation. The aggregate specimens can be best examined and tested if they are properly prepared for the proposed examination and testing (see ASTM D 75).

Particulate materials become segregated into sizes or into mineralogical portions when they are stockpiled or even when they are shoveled or poured into small piles. The ability of the particles to move over each other and the substrate is affected by the density, geometry, shape, and surface texture of the particles (Mullen, 1978). Air resistance may affect the placement of very fine sizes of materials. Each type of material has its own elasticity and inherent bounce characteristics. These properties can cause both vertical and horizontal segregation within a pile of particles.

5.5.2 Procedures

5.5.2.1 Hand Specimen and Large Fragment of Rock

1. For each type of rock represented, prepare freshly broken surfaces and sawed surfaces in at least three directions at approximately 90° to each other.

2. If the rock contains a large percentage of carbonate minerals, fine lap the sawed surfaces and etch (with 10% HCl) the lapped surfaces to ascertain the direction of the bedding and emphasize the different mineral compositions.

3. Examine the broken and etched surfaces. Describe the color, condition, parting, lithology, etc. Pay special attention to any indications of reactive aggregate.

4. Crush the specimens to the size required for testing. Be careful that the portion selected is representative of all the kinds of aggregate submitted as one specimen. Treat the crushed aggregate as if it had been submitted in the crushed state. Refer to instructions in 5.5.2.2.

5.5.2.2 Sand, Gravel, Crushed Stone, and Slag

1. Ensure that the specimen has been treated (washed or not) and sized as will the aggregate that is being considered for use in HCC. Dust and mud

coatings may wash away. Sizing may allow exclusion of certain types of particles from consideration by indicating that they are such a small fraction that they can be ignored. When the specimen becomes beneficiated by such procedures, these procedures may be required of the aggregate supplier before the aggregate is accepted for use. Refer to Dolar-Mantuani (1983) and other applicable references in the Reading List for more information.

2. Inspect the entire specimen for the presence of the types of particles whose presence in even a small amount can cause the aggregate to fail the client's specifications. Roughly estimate the percentage of such particles.

3. Select a number of small portions to represent the whole for use in tests and examinations. Each portion to be tested must contain the same proportion of lithologic and mineralogic types and the same proportion of sizes, coatings, and impurities as the original specimen (see ASTM C 702). Take care that the reduction in sample size does not change the percentage of the undesirable types of particles mentioned in Step 2. The best method for reducing the quantity of the specimen to an amount usable by any test proposed should include the use of a number of sizes of sample splitters. For the finer sizes of sand, very small sample splitters may have to be constructed in the laboratory from such materials as Popsicle sticks. If the aggregate is fairly dust free and sample splitters of the proper size are not available, the material may be repeatedly halved by the cone and alternate quarter removal method: Construct a cone-shaped pile of the aggregate (on canvas-covered floor, table, etc.). Quarter the sample using a thin board or trowel. Carefully remove and combine alternate quarters. Sweep clean the area beneath each quarter removed, and place the small fragments and dust with the removed material. Repeat the procedure as many times as is necessary to reduce the total specimen of aggregate to the quantity required for whatever testing is to be performed. Do not reduce the specimen below the amount required by the examination or test to be performed.

Chapter 6
THE VOIDS

6.1 OVERVIEW

In hardened HCC, a void is an empty space, other than a crack, in the cement paste that contains nothing but air. The type, size, shape, arrangement, and abundance of the voids are factors controlling many important properties, such as compressive strength, resistance to destruction by freezing and thawing, and resistance to chemical attack on the reinforcing steel and the cement paste. The percentage of air-void volume is generally specified by the design of the mixture. A large number of very small (most not visible without magnification) air voids are desired so that an appropriate amount of air can be distributed throughout the HCC in such a way that the distance between voids is very short and, thus, the paste is protected from freezing and thawing. A ratio of air-void volume to paste volume that exceeds the specified range weakens the HCC and may create channelways for the permeation and circulation of deleterious substances.

The total air-void content (of voids larger than capillary size) of an unhardened concrete mixture is routinely determined in accordance with ASTM C 231 (the pressure method) or ASTM C 173 (the volumetric method). Unit weight determinations (ASTM C 138) may also be determined to provide information concerning the percentage of air in the mixture. These methods do not ascertain the type of voids present; they merely measure the total void content. These measurements are important. As Bartel (1978) stated:

> Tests for air content and unit weight of fresh concrete, carefully made in accordance with the appropriate ASTM test method, will yield an accurate measurement of the amount of air, weight, and volume of concrete being produced. Tests for air content, coupled with intelligently selected specification limits, can ensure the beneficial effects of entrained air in hardened concrete (p. 130).

Specially formed specimens of hardened HCC mixtures may be tested for resistance to the destructive forces of freezing and thawing in accordance with ASTM C 666 (resistance to rapid freezing and thawing). An HCC that is resistant generally indicates that an adequate air-void system is present or that the HCC has not become critically saturated.

It has been variously claimed that the total air-void content increases or decreases as the concrete hardens. It appears that what really happens is that the determination of total air-void content with field equipment made on the fresh concrete does not agree with the total air-void content determined by microscopical analysis of the hardened concrete. Except in the case of hydrogen gas being evolved by the incorporation of aluminum fragments (Newlon & Ozol, 1972) (see Fig. 6-1), no evidence of an equivalent change in the volume of concrete in the field placement or test cylinders has been offered as evidence corroborating the increase or decrease in total air-void content.

Careful investigation by a combination of controlled mixing and sampling procedures and petrographic techniques has shown that the air-void content does not change on hardening but rather may change due to outside influences, such as extreme overconsolidation (unusually long vibration, thus removal of more of the entrapped air than usual) or the further addition of water and retempering. The de-



Figure 6-1 CONCRETE THAT INCREASED IN VOLUME DUE TO INCORPORATION OF ALUMINUM FRAGMENTS. Since it was cast in the cylinder mold, the concrete increased in volume due to the incorporation of aluminum fragments (from an aluminum delivery pipe). Thus, hydrogen gas evolved from the chemical reaction of the aluminum with the alkaline fluids of the fresh cement paste.

termination of the void content in the hardened state should agree within 1 percent with the void content determined in the fresh, unhardened state. When they do not closely agree, either one of the measurements is in error or the two specimens tested do not represent the same concrete subjected to the same influences (see Appendix D and Ozyildirim, 1991).

An air-void content in excess of the amount required for protection against the destructive forces of alternate freezing and thawing that occurs in saturated concrete adds no benefit to concrete expected to bear loads and resist abrasion. (For a discussion of the high-air, cellular concretes, see Lesatski [1978] and Lewis [1978].) An excessive air-void content will lower the compressive strength of the concrete by about 5% for each excess percentage of voids.

In the early days of the use of air-entraining agents, there were some who saw that cracks ended at air voids and interpreted this to mean that the cracks started at air voids and were caused by them. In many parts of the United States, there was (and still is) a fear of an air-void content that exceeded the minimum specified. This may be interpreted as a fear of not meeting the compressive strength requirements. By nature of the bell-shaped probability curve, avoidance of high air-void contents can lead to air contents that are below the amount required for resistance to the destructive forces of freezing and thawing in saturated concrete. Conversely, there now seems to be, at least in this area of the United States among some concreting contractors, a fear of low air content. When HCC containing a percentage of air near the top limit of the specified amount is altered by the addition of retempering water (which can cause air-entraining agents to be more active), an HCC with an

air-void content considerably higher than the specified quantity is frequently produced: the result is concrete of insufficient strength (see Appendix D).

6.2 TYPES OF VOIDS

The overall void content in HCC is composed of four general types of voids, as listed in Table 6-1.

Table 6-1
TYPES OF VOIDS

1. **Capillary voids.** Capillary voids are irregularly shaped and very small, less than 5 μm on the lapped surface of the slice examined. They represent space originally filled by mixing water, remain after the hydration of the cement gels, and are an integral part of the paste. Although they contain air at the time of examination, they are not considered part of the air-void system.
2. **Entrained air voids.** Entrained air voids are defined at VTRC as spherical voids larger than the capillaries but less than 1 mm on the lapped surface of the slice examined. They are formed by the folding action of the concrete mixer, and their shape and size and abundance are influenced by the addition of surface-active air-entraining admixtures to the mixture.
3. **Entrapped air voids**. Entrapped air voids are voids that are larger than the entrained voids but have internal surfaces that indicate they were formed by air bubbles or pockets. They may be spherical or irregularly shaped.
4. **Water voids.** Water voids are irregularly shaped voids whose shape, location, or internal surface indicates that they were formed by water. Usually, they are larger than entrained air voids.

6.2.1 Capillary Voids

The smallest class of optically visible voids in HCC are the various sizes of capillaries. A very few of the larger capillary voids may be seen at the higher magnifications used to determine the parameters of the void system, but they are generally not that large. Capillary voids are spaces formed by the shape of the hydrated cement gel structures and spaces left between the gel structures as water is used in the self-desiccation of the hydration process. They were occupied by water or gas when the concrete was fresh and are larger and more abundant in concretes with a high water-cement ratio. The magnitude of the capillary system is controlled by the water-cement ratio and the degree of maturity of the concrete. The evenness of the distribution of the pores and capillaries is controlled by the distribution of the water. As the concrete hydrates, the water in the pores is used in the hydration of the cement. As the concrete matures, much of the capillary space becomes filled with the products of hydration and the products of any reactions occurring between the chemicals of the paste and the aggregate rocks. Some of the finer capillaries are spaces created by differential crystal growth. (See Chapter 13 and associated figures and note how the quality of the fine aggregate affects the distribution of moisture and thus pores and capillaries in the paste.)

The capillaries are detected only when specialized methods are used. In laboratories so equipped, the various types of electron microscopes may be used to view the capillary void system. In the VTRC laboratory, the abundance and location of the capillary voids are detected by use of the P/EF microscope in the study of fluorescent thin sections of the specimen concrete (see Chapter 13). Rarely, capillary voids

may be noted during the determination of the parameters of the void system. In that event, capillary voids are considered to be paste.

6.2.2 Entrained Air Voids

Entrained voids are small spherical voids enfolded by the mixer. Surface-active, air-entraining agents are added to the mixture to stabilize a specified percentage of these voids and thus protect the hardened HCC against the destructive forces of freezing and thawing. Thus, the entrained air void is a desirable void. Entrained air voids are generally considered to be larger than the capillaries (at least 5 μm in diameter) but smaller than the voids considered to be entrapped voids (Verbeck, 1966, 1978). Entrained air voids have so much surface tension relative to their volume that they are distorted very little by the shape of nearby particles. Distortion occurs in these small voids only when external forces distort the concrete after the beginning of hardening.

The presence of the proper quantity of well-distributed entrained air voids can prevent deterioration of the concrete (even when saturated) by the mechanisms of freezing and thawing (Helms, 1978; Newlon, 1978) and facilitate the placement of the concrete because the entrained air voids act as additional fluid, almost as though the entrained voids were ball bearings. In Virginia, the proper quantity of air voids is defined for each class of HCC by *Road and Bridge Specifications* (1991). Entrained air voids allow the relief of pressures caused by the freezing and thawing of saturated HCC and thus protect the HCC from destruction by these mechanisms. The exact method by which they perform this function has not been determined and agreed on by all concrete technologists, but all agree that the empirical evidence demonstrates that the presence of a sufficient quantity of sufficiently small (entrained size), properly distributed air voids protects the cement paste in the concrete from deterioration by freezing and thawing.

Very irregularly shaped small voids (maximum dimension less than 1 mm) cannot be properly called entrained voids because the surface tension caused by the air-entraining agent is lacking. It is not known if such voids function to protect the concrete against the deterioration caused by freezing and thawing. Small, irregular voids, particularly if not at an aggregate boundary or a wearing surface, may be evidence of retempering (see Appendix D).

Figures 6-2 and 6-3 show varying percentages of air voids.

6.2.3 Entrapped Voids and Water Voids

All voids, regardless of shape, that have a maximum dimension (on the surface examined) of more than 1 mm are defined at VTRC as entrapped voids (large spherical) or water voids (large irregular). If voids occur flattened out at the boundary between the aggregate (usually coarse aggregate) and the paste, they are a class of entrapped voids called boundary voids.

All voids larger than entrained voids have no appreciable beneficial effects and weaken the HCC. Such voids are partially controlled by the efficiency of whatever system of consolidation is in use. Certain voids may be caused by too much water in the HCC, a strong affinity of a particular aggregate lithology for water, improper consolidation, and occasionally by the dissolving away of $Ca(OH)_2$. Irregularly shaped voids, regardless of size, may be caused by water pockets or air pockets that

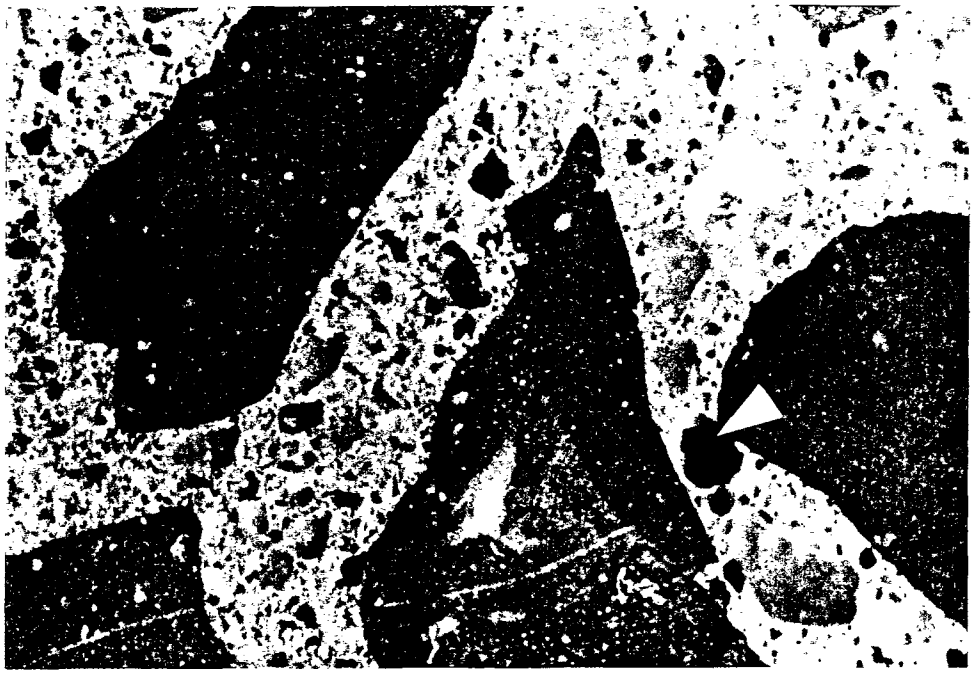


Figure 6-2 SURFACE OF FINELY LAPPED SLICE OF CONCRETE CONTAINING 5.6% TOTAL AIR VOIDS. The void content of this concrete is in the middle of the specification range. The large void marked with an *arrow* is about 2 mm across (larger than an entrained air void). Notice the very fine voids throughout the paste.

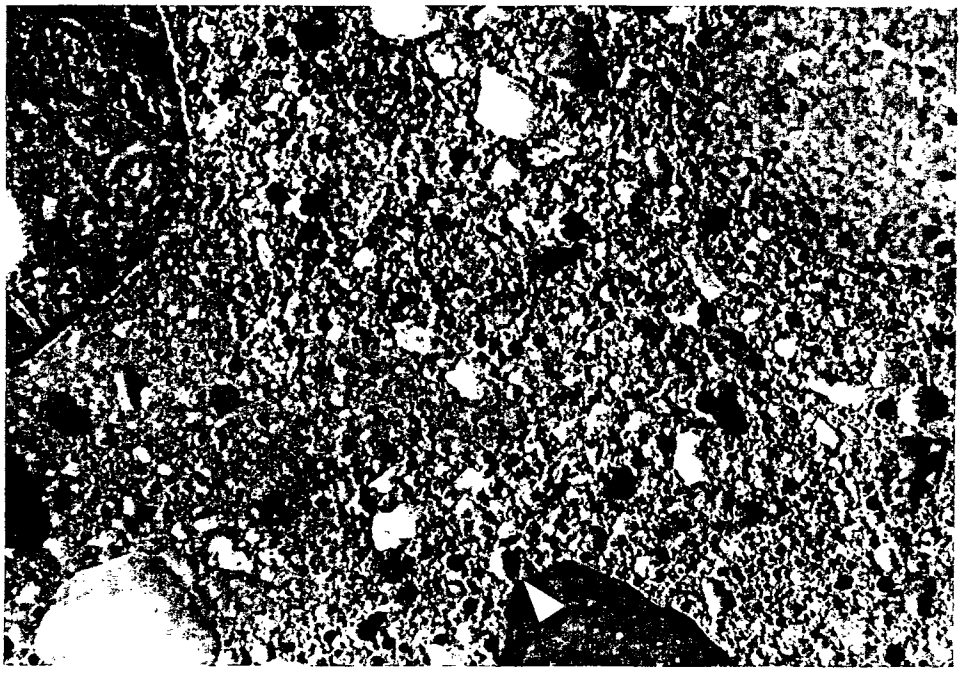


Figure 6-3 SURFACE OF FINELY LAPPED SLICE OF CONCRETE CONTAINING 17% TOTAL AIR VOIDS. The void content of this concrete is way above the upper limit of the specification range. The void indicated by the *arrow* is about 1 mm across. The area of darker paste (lower left) has a lower void content. An HCC that contains more than one kind of paste generally indicates that the mixture had begun to hydrate before additional water was added (see 8.4 and Appendix D).

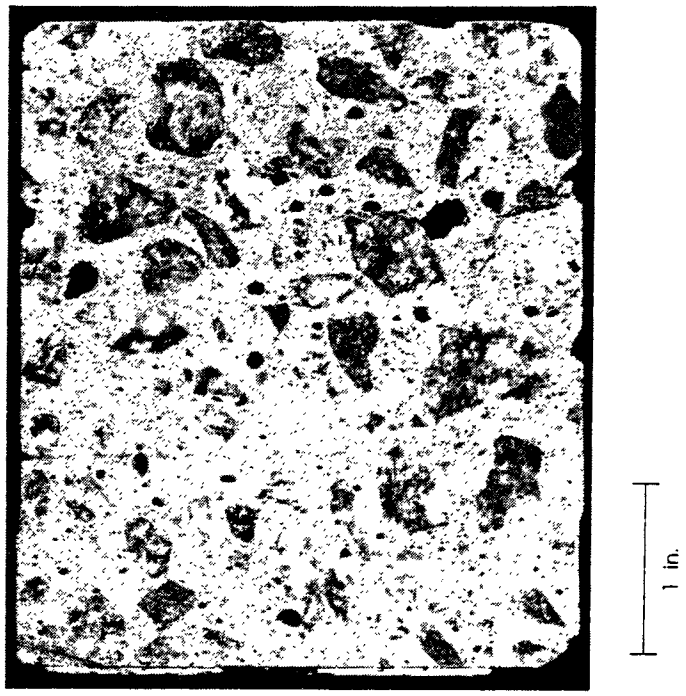


Figure 6-4 CONCRETE CORE WITH ABOUT 4% LARGE IRREGULAR VOIDS. In this instance, the concrete, which had not yet been consolidated, became hard and unworkable while repairs were being made on the paving equipment.

the consolidation procedures did not remove (see Fig. 6-4). Water voids are irregularly shaped voids created in the HCC by bleed water prevented from rising to the surface by an aggregate particle or the hardening of the paste. Water voids contained water when the HCC was fresh and unhardened. In the hardened state, these voids are filled with air and might be more properly termed *water-formed air voids.*

6.3 QUANTITATIVE DETERMINATION OF AIR-VOID PARAMETERS

6.3.1 Overview

In hardened concrete, the parameters of the air-void system may be determined by obtaining the data and performing the calculations specified in ASTM C 457. The parameters calculated include:

1. *Air-void content* (symbolized in ASTM C 457 by A). It is a percentage by volume. A minimum amount of air voids are required to protect the concrete from the expansion of water during freezing. Excess air-void content will cause the concrete to have less than the intended compressive strength.
2. *Void frequency* (symbolized in ASTM C 457 by n.) It is the number of voids per unit length of traverse. The void frequency is required in the calculation of the average chord in the modified point-count method.

3. *Average chord length* (symbolized in ASTM C 457 by $\bar{l}$). It is the length of the sum of the chords of the air voids divided by the number of voids encountered in the traverse.

4. *Specific surface* (symbolized in ASTM C 457 by α). It is the surface area of the average void divided by the volume of the average void. It is calculated from the average chord. The unit involved can be expressed as squared units divided by cubed units or as units to the minus 1 power. The higher values (higher void surface area per void internal volume) indicate smaller voids. Small voids (with shorter average chord) are desired because they disperse throughout the concrete with small unprotected volumes of paste in between. If the same air-void content was present in larger voids, the unprotected volumes of paste would be much larger.

5. *Spacing factor* (symbolized in ASTM C 457 by $\bar{L}$). It is calculated from the specific surface, the percentage of air voids, and the percentage of paste (see 7.1) that must be protected. It is expressed as a decimal value of the measurement unit. The spacing factor is a theoretical measure of the average distance water, ice, or expansive force must to travel in HCC before it contacts an air void, i.e., half the average distance between air voids. The smaller the spacing factor, the more completely the air-void system can protect the concrete against deterioration by freezing and thawing. Regardless of the ratio of air-void volume to paste volume, the higher values for void frequency and the concomitant shorter average chord length result in smaller spacing factors and a more desirable air-void system.

6.3.2 Methods and Equipment

6.3.2.1 Overview

New methods and equipment are continually being devised to monitor and determine the air-void parameters of hardened concrete. It is part of the job of the petrographer to assess the value of new methods and equipment and decide which method is of value in which situation and, therefore, which equipment is worthy of a place in the budget of the organization. If the results of an air-void determination are to be presented in court and the testimony of opposing expert witnesses will be heard, any deviation from the principles of ASTM C 457 that has not been agreed on by the client may invalidate the results of the analysis. Within an organization, certain deviations from the strict interpretation of ASTM C 457 may be acceptable.

According to ASTM C 457, air-void system analyses can be efficiently performed with several methods and kinds of equipment. Suitable equipment for the determination of air-void parameters in hardened concrete includes, but is not necessarily limited to, (1) linear traverse, (2) modified point-count, and (3) image analysis equipment. In common, the types of equipment to be used permit or facilitate the movement of the specimen of HCC on the stage of a microscope so that data may be collected over the specified area and from the specified length of traverse. In common, the data collected are:

1. *The total length of traverse over which the determination is made.* In the modified point-count method, the total number of points examined and the distance the equipment moves between the points are required for the calculations.

2. *The portion of the traverse that is across air voids.* In the linear traverse method, this portion is the sum of the chord lengths across air voids; in the modified point-count method, this portion is the number of points that occur in air voids multiplied by the distance the equipment moves between points.
3. *The number of voids occurring in the traverse examined.* The accuracy of the determination of the specific surface and spacing factor is completely dependent on the accuracy of the count of the number of voids on the line traversed. In the linear traverse method, the number of voids in the traverse is the number of chords collected; in the modified point-count method, it is the number of voids counted along the traverse line.

The procedures detailed in ASTM C 457 are those to be used with nonelectronic types of equipment (see ASTM C 457, Figs. 2 and 5). When equipment is used that includes automatic devices for moving the specimen, electric or electronic counters or totalizers and calculators, or measuring devices, the equipment must allow adherence to the principles of ASTM C 457 and permit or perform the calculation of the same parameters of the air-void system from the same data. The exact procedures followed for the operation of the equipment must be those described and specified by the fabricator of the equipment.

It is not known which type of equipment produces the most accurate results or how accurate the 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 for research purposes. Image analysis is favored by those who desire speed and the ability to collect a lot of data and manipulate it on a computer in many different ways. Image analysis can strain the equipment budget but requires less operator time since the specimen is not examined by the human eye. Image analysis equipment is not available at VTRC. Research laboratories will usually require either point-count or image analysis equipment for speed in making routine determinations and linear traverse equipment for its ability to determine chord length distribution on a surface unaltered by the fillers and dyes required by image analysis.

> ***NOTE:*** *The air-paste ratio method of calculation detailed in ASTM C 457 is to be used ONLY (1) when proportions of the ingredients in the mixture are known with some certainty, (2) it can be assumed that no change in mixture proportions has occurred (e.g., retempering has not occurred; i.e., the amount of paste can be closely calculated), AND (3) because of the lack of exposure of a generalized specimen of the HCC or because of the extremely large size of the aggregate it is impossible to obtain a specimen of the HCC for microscopical analysis with an aggregate distribution that is representative of the placement. The air-paste ratio calculations use the aggregate-paste ratio of the design of the mixture to transform mathematically the air-paste ratio determined microscopically to percentage air voids, specific surface, and spacing factor. In these situations, it is convenient to select a specimen of HCC that is low in aggregate so that the microscopist will not have to spend excess time moving over aggregate.*

6.3.2.2 Linear Traverse

Using the linear traverse equipment (see Fig. 6-5), the operator tabulates the chord lengths across all phases of interest and records them for later analysis (Walker, 1988). This sort of data permits the straightforward calculation of the void parameters by the summing of the lengths of the chords and counting of each occurrence of

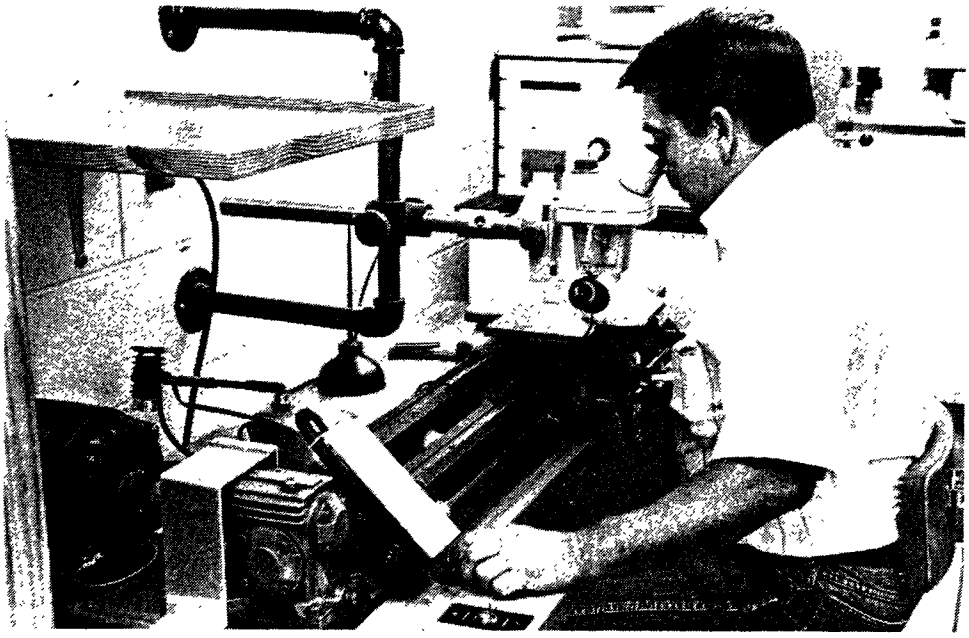


Figure 6-5 PARTIALLY AUTOMATED LINEAR TRAVERSE EQUIPMENT FOR DETERMINING AIR-VOID PARAMETERS

a phase. Because the calculations are extremely sensitive to errors made in the determination of the number of voids traversed, the method of deciding whether a void is or is not touched or transected by the line of traverse must be carefully employed in any case of doubt. If the individual lengths of the chords of the air voids are recorded and certain shape assumptions are made, a graphical representation of the chord lengths will indicate the size distribution of the air voids. The collection of the air-void data requires one pass of the microscope along the traverse line. The data necessary for calculation of the paste content may be collected at the same time or a separate determination can be made for the paste content. This procedure is further discussed in 7.1.2. With some types of linear traverse equipment, all the air-void parameters are automatically calculated; with others, the calculations must be performed on a calculator or computer.

6.3.2.3 Point Count

With the point-count equipment (see Fig. 6-6), the operator records the type of substance (air void, paste, or aggregate) appearing at the index point of the reticle at a large number of points as provided by a click stop on the stage. The points may be randomly distributed or regularly distributed on a randomly placed grid or a traverse line. Data concerning the relative amounts of all the phases can be collected from one pass along the traverse line. Calculation of voids per unit length, average chord length, specific surface, and spacing factor usually requires that a second pass along the traverse be used to count the number of voids occurring on the traverse line. Although the majority of users of the point-count apparatus collect the information concerning the abundance of paste during the same pass on which that concerning the abundance of air is collected, they may sometimes find difficulty in

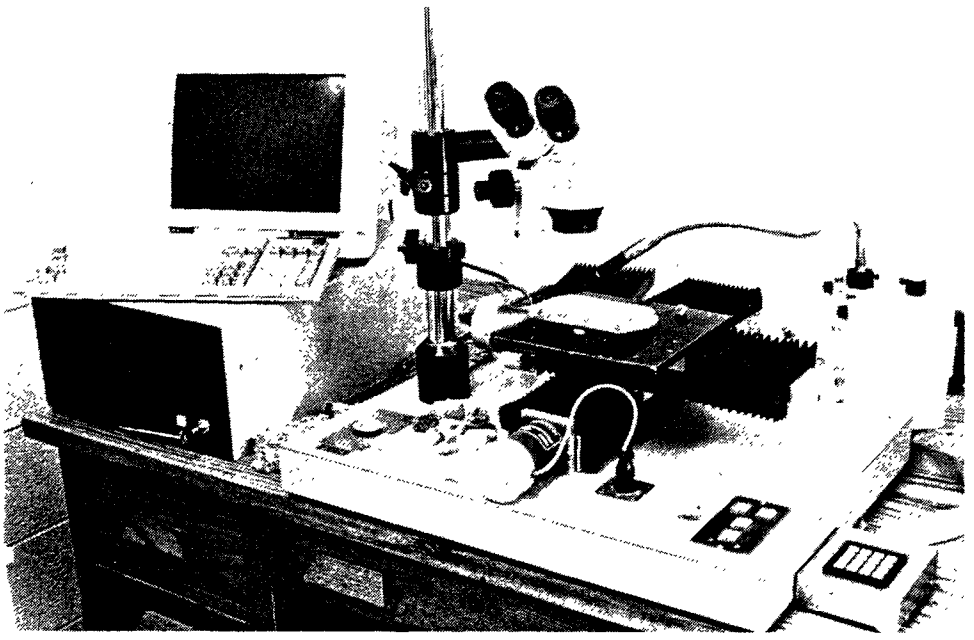


Figure 6-6 FULLY AUTOMATED EQUIPMENT FOR DETERMINING AIR-VOID PARAMETERS. Either linear traverse or point-count software can be used to control the computer and the motion of the stage.

distinguishing the exact paste-aggregate boundary and might wish to consider a separate pass over a lightly etched surface to collect these data (see 7.1.2). The air-void parameters may be calculated by the analysis equipment or separately calculated using a calculator or computer.

6.3.2.4 Image Analysis

Image analysis equipment (see Fig. 6-7) requires that the specimen be specially prepared so that each of the three major phases of interest (voids, paste, and aggregate) is a distinct tone (e.g., white, black, and medium gray). The presence and shape of areas of the three selected tones are determined by electronic eye, and the data are automatically recorded, sorted, and calculated. The specimen preparation methods for image analysis can be exacting and make the surface used useless for ordinary stereomicroscopic examination (as described in Chapter 8). If it is desirable to examine the distribution of phases with the human eye later, when the specimen is sawed, the surface facing the surface that is to be colored and filled should be finely lapped for microscopical evaluation. Both of these surfaces should be kept intact and safe until all controversy regarding the concrete is over. Automatic systems that require filling the voids (thus hiding their interior surface) cannot be used to make certain distinctions possible by a human operator. The human operator can often mentally reconstruct what the surface examined would have been if this or that flaw had not occurred. The human operator can judge if a void observed is an air void, a fly-ash cenosphere, or the hole left where a small round grain of sand has fallen out. These distinctions are generally possible by study of the reaction products and the luster of the interior of the void.

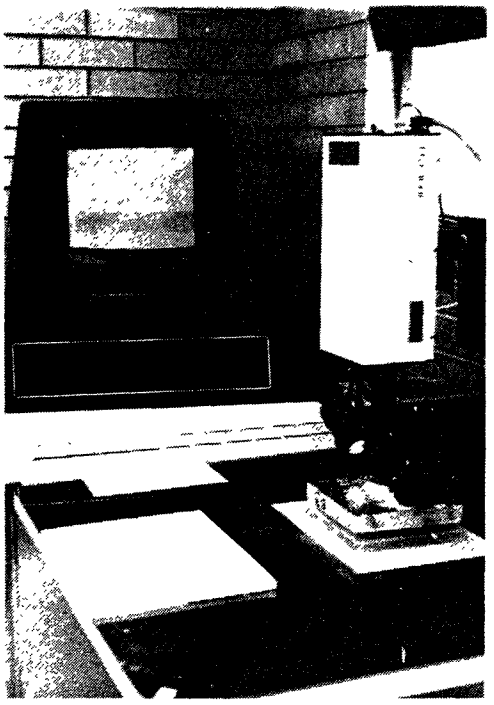


Figure 6-7 IMAGE ANALYSIS EQUIPMENT. The instrument is shown in the process of analyzing the air-void system of a slice of concrete. The screen in the background shows the progress of the analysis. (Photograph by R. H. Howe, courtesy of PennDot.)

6.3.2.5 Other Considerations

At VTRC, it has long been recognized that the accuracy of a linear traverse determination of the air-void parameters is as dependent or more dependent on the number of voids encountered and measured along the traverse as it is on the length of the traverse. Once 1,000 voids have been measured and counted, the results from the data obtained subsequently change very little. Snyder, Hover, and Natesaiyer (1991) made an analytical investigation of the effect of the number of voids and the length of the traverse on the minimum expected error that can be encountered in a linear traverse determination of the void parameters in hardened HCC. Their work supports our belief that little additional accuracy is achieved if the determination includes more than 1,000 voids and that almost no additional accuracy is achieved with more than 2,000 voids.

For intradepartmental purposes, for ordinary determination of the air-void parameters, it has been our practice to estimate visually the number of voids per unit length of traverse, consider how long a traverse is required in order to obtain data on 1,000 voids, and then plan how to spread the traverse length evenly over the specimen surface available. Under circumstances when ASTM C 457 requires 100 in. of traverse, we estimate that only 50 to 70 in. is required for the collection of data from 1,000 voids in ordinary within-specification concrete. Should some legal controversy arise concerning the subject concrete, any traverse length deficiency can be made up by collection of data from the lacking inches of traverse (also evenly distributed over the surface). If the traverse direction and starting point are randomly chosen, in both cases, the randomness of the data collection will be maintained. It is our view that spreading the data collection area over as large a surface

area as possible so that any irregularities of void distribution (any clumping or areas devoid of voids) become part of the data recorded and examined is more important than the length of the traverse line.

The corollary is that if the number of voids is very small due to low air content or large voids, the length of traverse recommended in ASTM C 457 is probably not sufficient to obtain accurate air-void parameter data (Snyder, Hover, & Natesaiyer, 1991). Under such circumstances and with borderline compliance with specifications, it may be wise to use an additional length of traverse to ensure accuracy. In most cases, the small specific surface and large spacing factors caused by the lack of sufficient small voids will decisively indicate lack of compliance with the specifications.

The method of deciding whether a void is or is not touched or transected by the line of traverse must be a simple rule that is firmly adhered to throughout any particular analysis. Pleau, Plante, Gagne, and Pigeon (1990), using the point-count method, recommended: "A simple way to guarantee a random choice is to systematically choose the constituent located in a given quadrant (of the field viewed), say the upper left corner of the cross-hairs." A similar method can be devised for whatever type reference point, reticle, and counting method are in use. Other researchers (Mather for one [1989]) have suggested that points in dispute be collected in a separate register and later distributed to the totals of the constituents in the same proportion as are the data concerning which there is no dispute.

6.3.3 Preparation of Specimens

The importance of the proper preparation of the surface of the slice of concrete cannot be overemphasized (see Figs. 5-2 and 5-3). In most laboratories, specimens are prepared by skilled, highly trained technicians. A poorly prepared specimen can cause a determination of the percentage of air present in a specimen to deviate from the true value by as much as 2 percentage points (20% to 50% of the true value). A rough surface makes it impossible to detect small voids. This will have the effect of lowering the detected percentage of air, decidedly lowering the specific surface, and thus raising the spacing factor. Quantitative determination on a surface that is undercut and wherein the edges of the voids have been chipped or worn away can provide data that indicate the presence of more air than really exists.

The preparation methods used by Pleau et al. (1990) can be questioned. They advocate soaking the slices (slabs) in water for a few days, presumably to complete the hydration and produce a more stable material for lapping. (Chapter 5 has several suggestions for the treatment of weak or immature concrete before lapping.) However, water can wash away reaction products, liquify expansive alkali-silica gels, dissolve calcium hydroxide crystals, loosen aggregates in their sockets, change the appearance of the inner void surfaces, and weaken thin paste walls between voids. The void walls and remnants of void walls serve to define the void boundaries and facilitate the recognition of the individual void structures. If the inner void surfaces are in their original condition, the luster, surface texture, and asperities on these surfaces can help distinguish the differences among entrained voids, entrapped voids, water channelways, and aggregate sockets. Thus, water should not be used in sample preparation. It is common practice in concrete laboratories to use a saturated solution of calcium hydroxide as the water bath whenever specimens of concrete are soaked in water (for testing absorption etc.) to prevent weakening the concrete by dissolving of the contained calcium hydroxide that is an important part of

the structure of most concretes. The calcium hydroxide solution may have undesirable effects on specimens prepared for microscopical analyses and is not recommended for this purpose.

Each method of producing a finely lapped specimen surface for microscopical study will probably produce different effects on different types of concrete (different water-cement ratios, different kinds of aggregates, different degrees of maturity and deterioration).

In certain concretes in which the shape of the air voids has become distorted (see Appendix D), all manner of overlaps and crushing of voids may occur; the operator should be alert and ready to record the data for each void in a logical and consistent manner.

6.3.4 Technician Considerations

The linear traverse and modified point-count methods are tedious and hard on the eyes. A single determination of the air-void parameters of a concrete by means of the linear traverse method can take up to 7 hr, depending on the size and quantity of the voids. A technician cannot spend more than 4 hr a day doing this sort of work on a day-to-day basis. Everyone who has tried has found that the ability of their eyes to focus has deteriorated on the following day. The training and keeping of good microscopical technicians can be a major undertaking requiring tact, skill, understanding, and a flexible schedule of rest periods.

Image analysis systems do not require that the operator be with the equipment after the initial adjustment; thus, eye fatigue and the need to train technicians to perform microscopical analyses are eliminated.

The following points are important considerations in the hiring and training of technicians for the microscopical analysis of air-void systems:

- Try to avoid hiring operators for linear traverse and point-count determinations of air-void parameters who do not have good binocular vision.
- Keep available standard specimens of concrete with a range of different types of air-void systems. Air-void contents of 2% to 14% are recommended. These should be specimens that have been analyzed by a number of different operators. The results previously obtained should be kept in a secure place by the supervisor. Each new operator who is trained for this work should be tested on the standard specimens, and training should continue until the results of the new operator are comparable to the range of results recorded in the past.
- Make sure that each operator knows how to adjust the positioning of the specimen so that it is flat and so that the specimen can move under the microscope and remain in the same focal plane. This procedure can be a tedious nuisance and may be neglected if its importance is not sufficiently emphasized during the training of the operators.
- Make sure that each operator knows how to adjust the binocular vision spacing; the height of his or her chair; and any other items available for greatest visual acuity, comfort, and convenience. The operator must understand that these adjustments are not emphasized for his or her personal comfort but rather because proper adjustment adds to the accuracy of the determination. An operator suf-

fering from a headache or backache is not as able to produce good data as a comfortable, healthy operator.

- Make sure that the operators understand the need for good focus and how to achieve good focus on the reticle for their main eye and simultaneous good focus on the specimen for both eyes. Each person has one eye that looks straight ahead (the main eye). The other eye observes things at an angle. Whenever an optical technique requires a reticle in one eyepiece of a binocular system, the reticle should be placed in the lens system used by the main eye.
- If the microscope is used by more than one person, make it a routine practice for each operator when beginning work to check the focus of the reticle and the focus of the surface being examined. Emphasize that the surface should be in focus throughout an entire traverse across the specimen. If focus is lost, errors will be great and the ability to judge the type and origin of a flaw in the finish of the surface being examined will be seriously diminished.
- Observe the actions of the operators and determine if they are following instructions. From time to time, have the work of the operators checked by having another operator redo a specimen, an operator redo a specimen done some months ago, or an operator redo one or more of the standard training specimens.
- Teach operators that great care must be taken to include in the count every void crossed by the traverse. The air-void count should be performed slowly and accurately. Very small voids and voids that are just barely touched by the traverse line must be counted. When the linear traverse procedure is used, it may be necessary to slow the motion along the traverse almost to a stop (if not completely) to register very small voids in the count. If it is realized that a void with an essentially zero chord length (because the traverse line is tangent to the void or because the void is very tiny) has not been counted, it is possible with some equipment to bring the motion along the traverse to a halt (so that zero chord length is recorded) and press momentarily the button that registers the presence of the appropriate void type. The location of a void along the traverse line is not a matter of concern, and the operator can record it anywhere. In the modified point-count method, no automatic motion is usually used while air voids existing along the traverse line are counted; therefore, this error will not be made in the same way.

6.4 CLASSIFICATION OF VOIDS

6.4.1 Overview

Determination of the abundance of the various types of voids is very useful in concrete research. It can make data available that can change various practices in the mixing and placing of HCC. As an example, it was once thought that the speed of the screed pulling the vibrators through freshly placed concrete did not affect the degree of consolidation. This did not seem logical to some. The Ballenger Construction Co. of North Carolina set up a series of test sections of pavement in which the speed of the screed was carefully controlled. A detailed petrographic laboratory analysis of the abundance of the various sizes of voids in the air-void system of 24 cores that had been removed from these test sections showed that there is a good inverse relationship between the speed of the screed and the degree of consolidation

(Walker, 1972a). As a result, the maximum speed of the screed is now limited in many specifications.

The quantitative determination of the abundance of various types of voids can be an important part of the petrographic analysis of a specimen. In the normal usage of linear traverse equipment in the VTRC petrographic laboratory, the abundance of each of the three types of air voids (entrained, entrapped, and water formed) is routinely determined (see Table 6-1). With equipment designed for this purpose, this determination may be performed concurrently with the determination of the other parameters of the air-void system.

In a finely lapped slab or a thin section, the size and shape of voids can be used as indicators of void origin and type. The luster and texture of the interior of the voids may sometimes be used in the recognition of voids caused by accumulations of water and passageways for water. The properties on which distinctions may be made between the various types of voids are arbitrary and may vary from one laboratory to the next. Because these distinctions are made on the appearance of a void on the surface of a slice (where the third dimension of the void cannot be seen), many large voids will be classified as entrained voids when they are really entrapped voids. As indicated in Figure 6-8, a small section through a large void can, in two dimensions, be indistinguishable from a large section through a small void. A cross section that is larger than the defined maximum for entrained voids must be a section of an entrapped air void or a water-formed void. A large number of large cross sections indicates a large number of large voids.

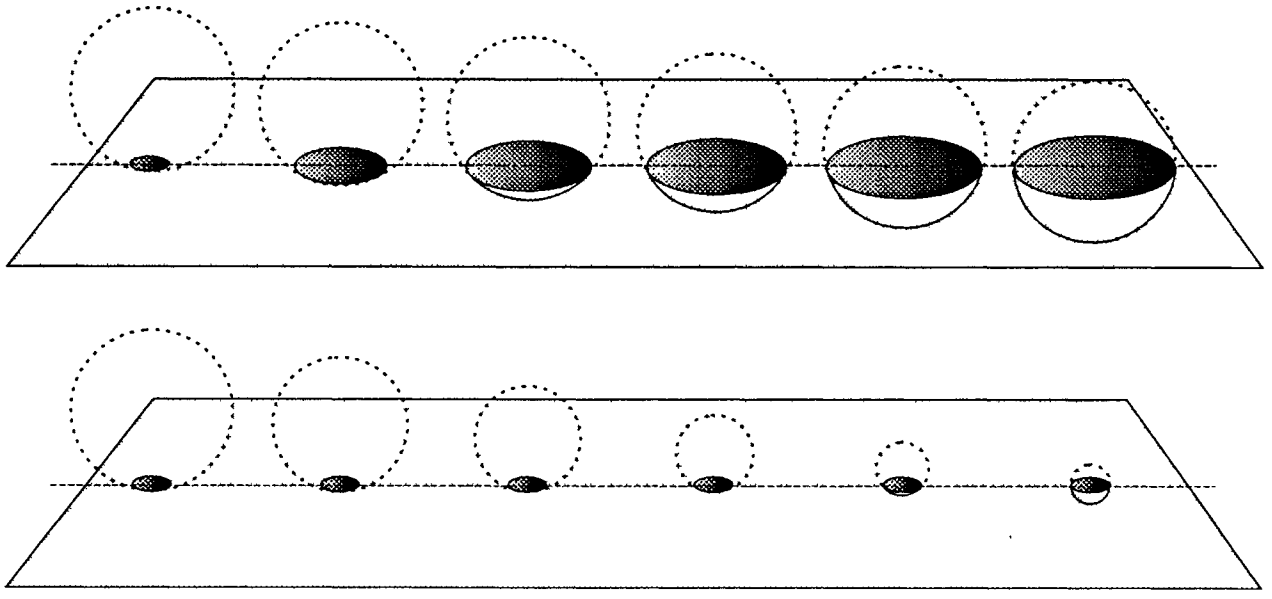


Figure 6-8 ILLUSTRATION OF VARIOUS SIZES OF SECTIONS THAT MAY BE EXPRESSED ON RANDOMLY PLACED PLANE

6.4.2 Distinguishing Between Entrapped Voids Caused by Air and Those Caused by Water

The petrographer can often distinguish between entrapped voids caused by water pockets and entrapped voids caused by air pockets. The estimate will necessitate careful observation and some extrapolation. In general, the interior surface of an air void will appear smoother, sometimes even shiny. A water void will usually have a dull interior that appears to have had small particles and precipitates deposited on it. In the case of water-formed voids, the shapes of the bounding aggregate particles are often visible in the interior of the void. Water voids may have an interior showing water movement patterns, may be interconnected bleed water voids, or may show by nature of the internal deposits and asperities and by position that they are water pockets trapped by aggregate particles.

6.4.3 Determination of Size Break Point Between Entrained and Entrapped Voids

The determination of the size break point between entrained and entrapped voids varies from laboratory to laboratory and must be interpreted in light of the method of measurement. For example, if the voids whose maximum chord on the surface examined is less than 1 mm are defined as entrained voids, then some voids whose true diameter is larger but not observable because the diameter is not in the plane of observation will be classified as entrained voids. The petrographer should maintain a clear idea of the meaning of the methods of determination in the size sorting of the voids.

A random line of traverse through HCC has a greater chance of traversing a large void than a small one. The probability ratio is as the ratio of their volumes (see Fig. 6-9). The calculations detailed in ASTM C 457 are designed to be used on the sums of the chord lengths and on the count of the voids regardless of the desirability or relative amounts of the various sizes. If the large voids are not counted and measured as part of the overall determination of the void system (suggested by Sommer [1979]), the control against large voids provided by the determination of the specific surface and spacing factor will have been blocked and the apparent precision of the method spuriously improved (Walker, 1980).

Unless the method and the criteria used to obtain data concerning void size are rooted in statistics, the data are only rules of thumb and valid only when compared with data obtained by the same methods. Calculations can be made on the distribution of the void sizes from chord data if certain assumptions are made concerning shape, heterogeneity, and isometric distribution of air voids.

At VTRC, the diameter of the section of the void as seen on the finely lapped surface examined must be equal to or less than 1 mm for the void to be considered an entrained void. In other laboratories, the length of the chord on the traverse line across the void is the parameter measured. The latter method makes it possible to set up an automatic electronic classification and counting system for entrained versus entrapped voids. In some European laboratories, the chord must be 0.3 mm or less for the void to be considered an entrained void (Wilk, Dobrolubov, & Romer, 1974). A void viewed in a lapped surface may be transected by the surface either above or below its true diameter, and there is no known way to measure an actual internal diameter. Efforts have been made to peer into a void to try to get an estimate of the true diameter, but in my view these efforts serve only to confuse the is-

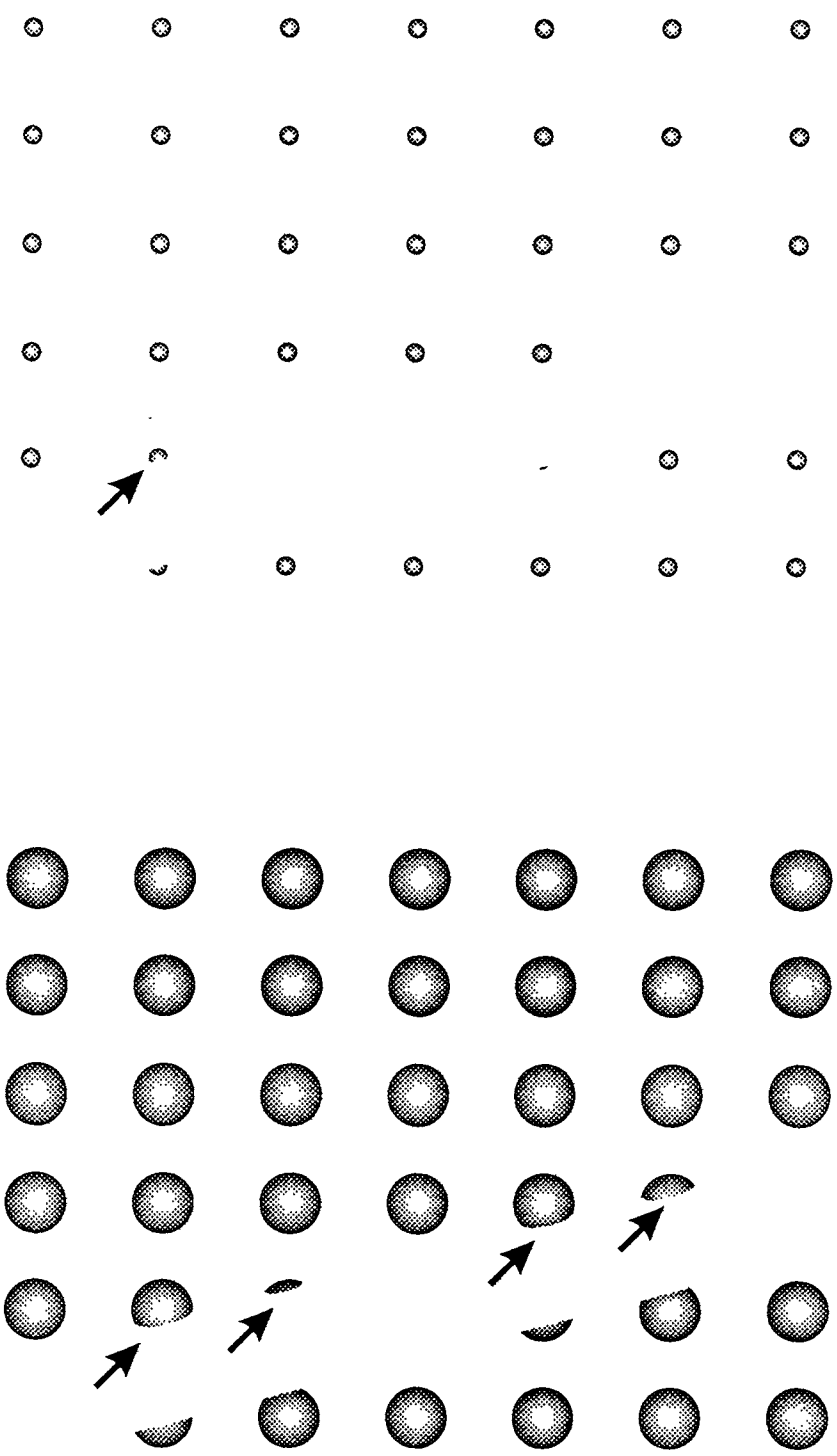


Figure 6-9 TWO EQUALLY SPACED ARRAYS OF VOIDS. Each is crossed by a randomly oriented plane. There is the same number of voids in a unit area in each array. Note that the plane touches more voids (see *arrows*) when the voids are large than when the voids are small.

sue. In many laboratories, decisions on individual void size are made on the lapped surface as viewed. A large void, more than 1 mm in diameter, may be so oriented that the surface examined truncates only a small portion of the void, the extreme top or bottom, when considered from the finely lapped surface. Thus, there will always exist a larger proportion of large voids than can be recognized on the surface examined (see Fig. 6-8).

6.4.4 Procedures

The procedures given here are for the linear traverse method when the chord lengths are collected by an operator depressing an electric recording device and either a paper printout is produced or three collecting devices are available (Walker, 1988). The point-count method does not survey every void on the traverse during the percentage portion of the examination and, therefore, does not allow a classification of every void. Image analysis procedures are not discussed in detail because such equipment is not available at VTRC.

1. Examine each void when the void's leading edge comes to the index point (usually the center of the cross hairs), and determine which type of void is present. With the wide-angle lenses and a magnification of 100X or less, voids of less than 1 mm in diameter will be completely visible in the field of view. Most voids can be classified at a glance as either entrained, entrapped, or water formed. When borderline cases occur, use a finely marked metric ruler on the slice, in the field of the microscope, to determine void size (at low magnification, an eyepiece micrometer may be used).

2. Record the presence of the void and the length of the chord across it in the usual manner for linear traverse (by pushing down a button and holding it down until the trailing edge of the void is at the index point). If three buttons are available for the three types of voids, each with their own totalizing devices that separately measure, total, and count the voids, depress the button appropriate for the type of void determined in step 1. If the chord lengths are recorded by using only one button and are individually printed on paper and the void encountered is not an entrained void, stop the motion of the traverse stage and mark the paper tape at the chord measurement with a symbol to indicate the classification of the void measured. Continue with the analysis; repeat from step 1 for each void.

3. When the analysis is complete, add the lengths of the chords for each type of void (if not added by the linear traverse device employed) and report the percentage by volume and the count (individual voids per specified inches of traverse) of each in the total concrete.

6.5 MEANING OF AIR-VOID PARAMETERS

The major parameters of the air-void system are interdependent. Some specifiers of concrete will require only that the air-void content is within certain limits; others will require that the spacing factor be below a certain limit or the specific surface be above a certain limit. Because one-sided limits on spacing factor and specific surface do not indicate the presence of a too high air-void content, the air-void content should be required to be within an upper as well as a lower limit.

The following are some of the issues the air-void parameters will determine:

- *Resistance to deterioration caused by freezing and thawing.* When ASTM C 457 is followed with care and the report is as instructed therein, the numerical data

obtained will clearly indicate the ability of the cement paste in the concrete represented by the specimen to resist the destructive forces of freezing and thawing. A specific surface of more than 600 $in.^{-1}$ ($in.^2/in.^3$) and a spacing factor of less than 0.008 in. indicate an HCC with a paste having an air-void system of the type that will resist severe winter weather conditions in a mature HCC containing few microcracks. Certain concretes with a very low permeability (and usually a very high strength) may resist the forces of freezing and thawing without meeting these requirements either because they never get critically saturated or (less likely) they lack sufficient freezable water when saturated.

- *Use of specific admixtures.* The petrographer is often asked if certain admixtures or an excess amount of a certain admixture has been used in a specific concrete. Extraordinarily low spacing factors accompanied by a high specific surface can indicate either excessive air-entraining agent or (if the total air content is within specifications) the use of a highly specialized admixture. During the first trials of some of the high-range water reducers, the paste was exceedingly compact but many of the voids were large. This created a specific texture, as illustrated in Figure 6-10. The high-range water reducers used in present-day mixtures do not create such a high content of large voids, but some concrete with this texture is still in service and requests to examine these concretes can still come in.
- *Flaws.* An unusually large number of voids that appear to have held water when the concrete was fresh indicate flaws in either the proportioning or the workmanship. An abundance of entrapped voids indicates either poor consolidation or early loss of slump.

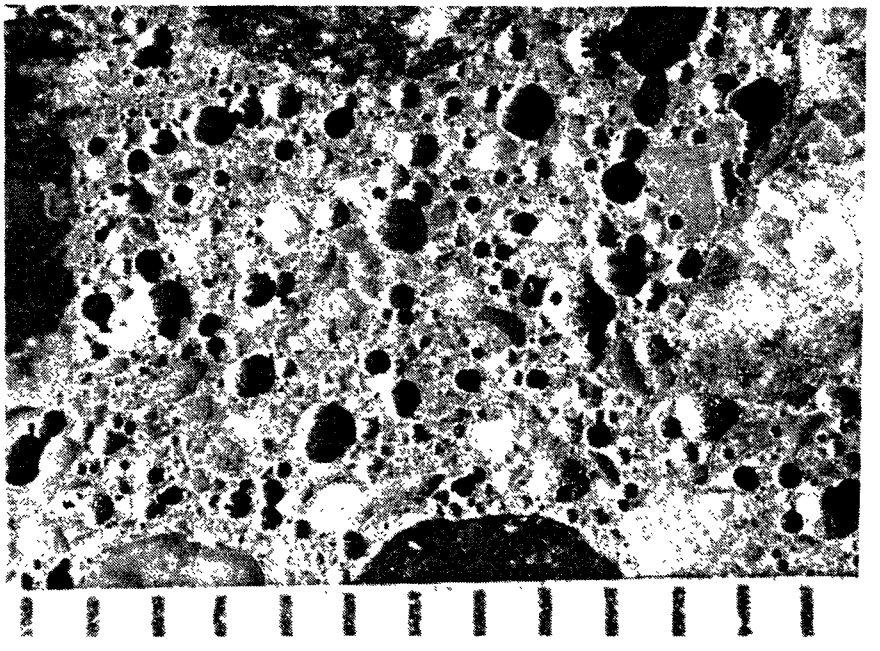


Figure 6-10 TYPE OF VOIDS AND PASTE TEXTURE PRODUCED BY EARLY TYPES OF HIGH-RANGE WATER REDUCERS. Such large voids do not add to the resistance of the concrete to freezing and thawing but do lower the compressive strength. The scale is in millimeters.

The data concerning the distribution of the types of voids as detailed in 6.4 can be used to discover certain placement conditions, such as the efficiency of the consolidation and reasons for various nonstandard conditions (such as low compressive strength or high permeability). Sometimes, when hand-held vibrators are used, there is excess entrapped air because of the persistent but erroneous belief that the specified amount of vibration will cause a loss of a portion of the required entrained air. It has been demonstrated that vibration even 2 or 3 times as long as the required amount does not reduce the entrained air in properly proportioned mixtures (R. H. Howe, personal communication, October 24, 1991). Although the maximum allowable quantity of large voids and the mathematical expression of a large quantity of large voids in a high spacing factor and low specific surface are not parameters required by the specifications of VDOT (thus difficult to argue in a court of law), it is important to consider these parameters and be able to explain their meaning. In general, it is much easier to talk about a large quantity of large voids than to explain the mathematical derivation of the specific surface and the spacing factor. If the percentage of the large voids exceeds 1.5%, it is considered high. More than 2% is considered excessive, and explanations for the prevalence of the large voids is sought. Does the concrete appear to have been retempered? Improperly mixed or consolidated? What is the reason?

Chapter 7
PERCENTAGE ANALYSES OF PASTE, AGGREGATE & OTHER SUBSTANCES

7.1 PASTE

7.1.1 Overview

It is necessary to calculate, estimate, or determine microscopically the percentage of air-free paste in an HCC specimen in order to calculate the specific surface and the spacing factor by means of the equations in ASTM C 457 (see Chapter 6).

7.1.2 Procedures

7.1.2.1 Calculation from Design of Mixture

When the design of the mixture is known and it is fairly certain that all additions of water or other changes in the mixture have been properly documented, the percentage of paste can be calculated from the design of the mixture by adding the volumes of water, cement, and particulate admixtures and determining the percentage this total of paste components is of the total volume of HCC produced by the mixture (ACI 211.1; ACI 211.2; ACI 211.3).

7.1.2.2 Estimation

Slight variations in paste content used in the calculations of air-void parameters do not cause much change in the values obtained. Therefore, at VTRC, for ordinary intradepartmental work, for preliminary work, and whenever the accuracy of the spacing factor and specific surface is not required to be defended against opposing expert witnesses, the paste content is estimated. The normal range of paste content is 23% to 32%; 27% has often been used as a good estimate of paste content in normal concrete (ACI 211.1; ACI 211.3; ACI 221R). This figure should be weighted by the judgment of the petrographer of the appearance of the specimen (see Fig. 7-1). If the quantity of paste appears to be far from normal (see Fig. 7-2), a microscopical determination of the percentage of paste should be made. If an estimate is to be used, the calculations may proceed and the data and method recorded in the notes for the report.

7.1.2.3 Microscopical Determination

The amount of air-free paste present is most easily determined by making a microscopical determination (linear traverse or point count) of the amount of aggregate present. The aggregate occurrence (sum of aggregate chords or sum of points falling in aggregate) is calculated by dividing by the total (total traverse length or total number of points) to determine the percentage of aggregate. Most laboratories using point-count equipment perform paste content determinations concurrently with the air content determinations; therefore, separate paste determinations are

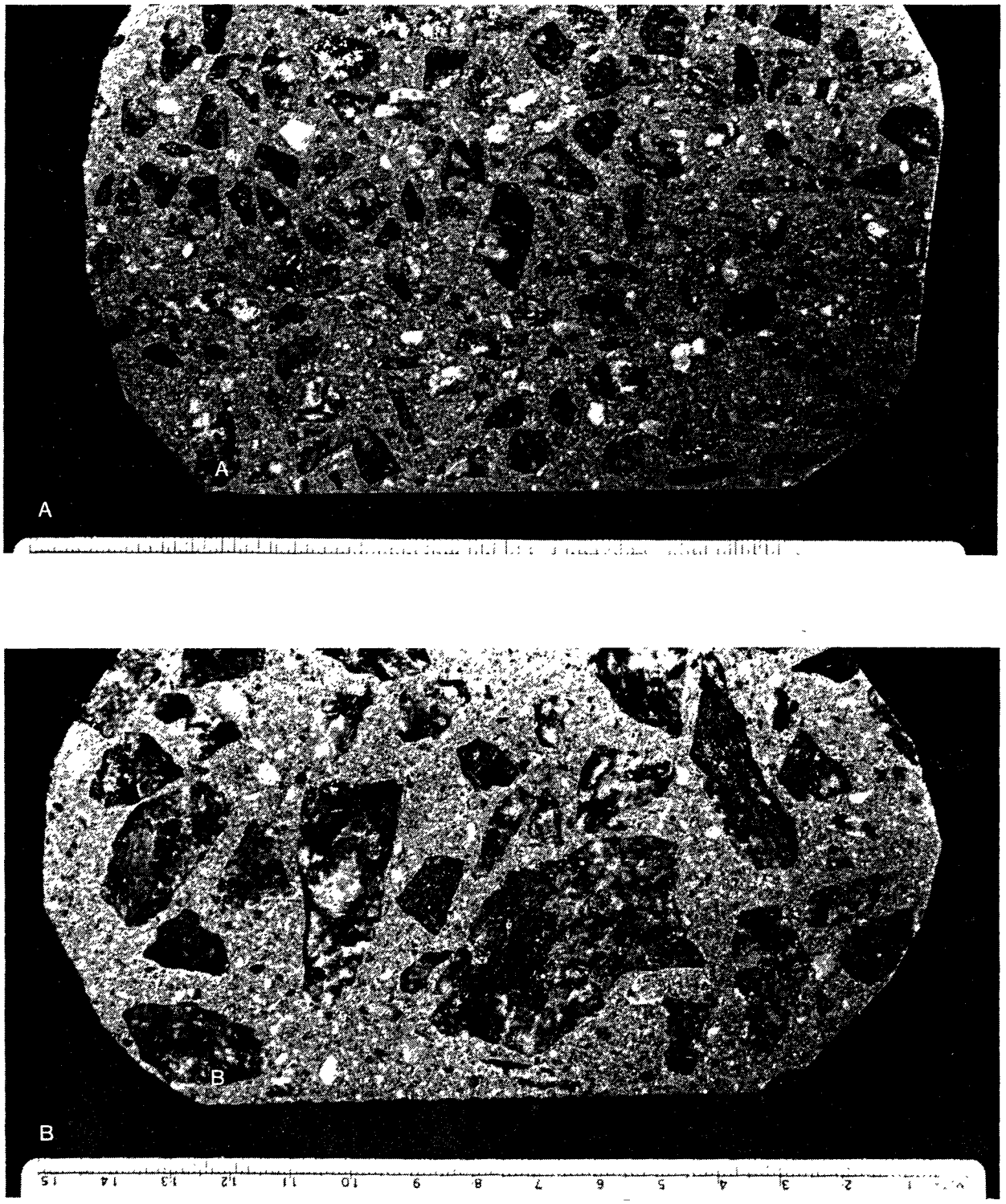


Figure 7-1 FINELY LAPPED SLICES OF CONCRETE WITH NORMAL PASTE CONTENT. Actual size. **A.** Rounded quartz gravel coarse aggregate and sand fine aggregate. **B.** Angular crushed granite coarse aggregate and sand fine aggregate.

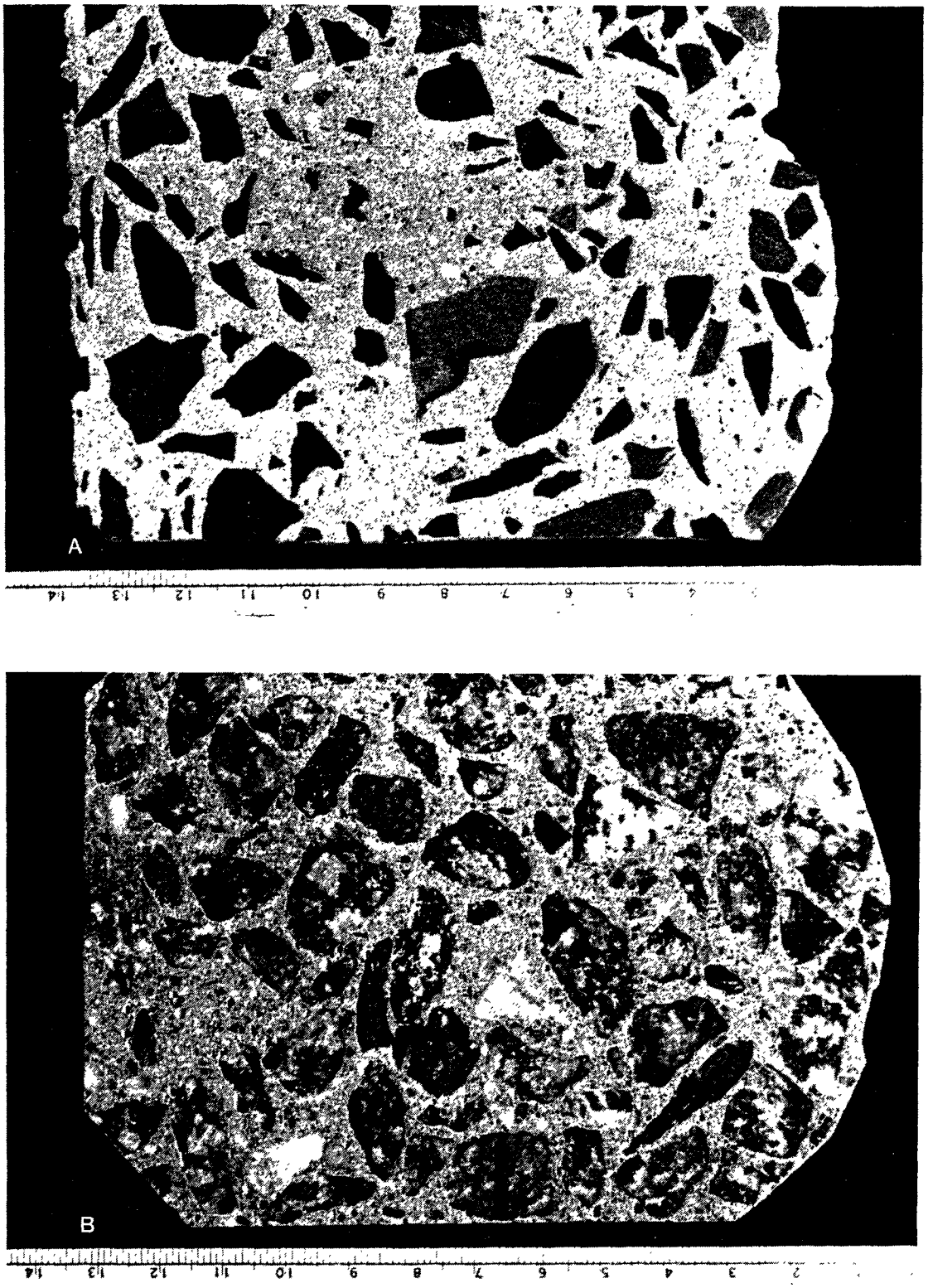


Figure 7-2 FINELY LAPPED SLICES OF CONCRETE WITH NONSTANDARD PASTE CONTENT. Actual size. **A.** High paste content. The coarse and medium-sized aggregate is a fine-grained metamorphosed shale, and the fine aggregate is a quartzitic sand. **B.** Low paste content. The coarse aggregate is a granitic gneiss, and the fine aggregate is a river sand.

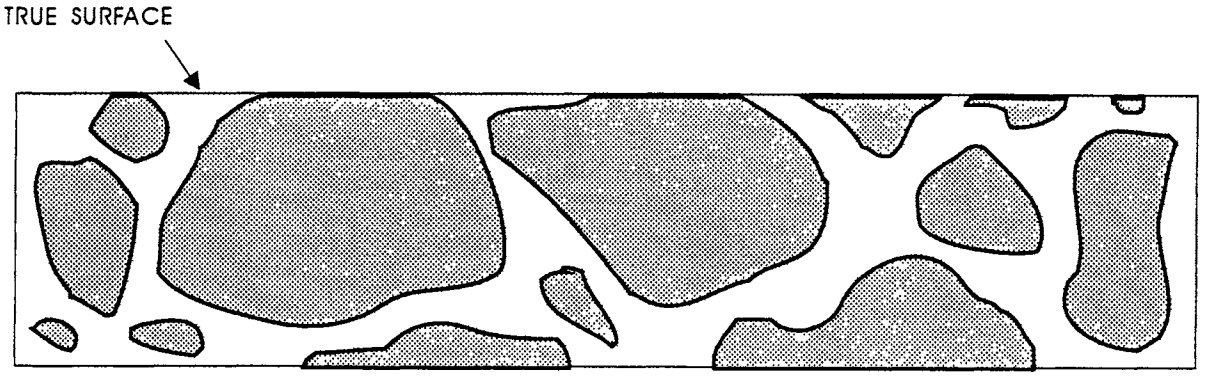
most often performed with linear traverse equipment. The percentage of paste is determined by subtracting the percentage of aggregate and the percentage of air from 100%.

To obtain the same accuracy in a microscopical determination of the aggregate as that of the air voids, at least 1,000 occurrences (fragments) of the aggregate must be counted (point count) or measured (linear traverse). To determine the percentage of a substance of which there are only 200 occurrences in the traverse of the surface on which the percentage of air was determined, five such slices would have to be prepared and examined. Examining such a large number of surfaces is often economically impossible, and unless the air-void determination included these surfaces, the air content determined may not be relevant to the paste content. When there are fewer than 1,000 aggregate particles along a microscopical traverse, the percentage of paste or aggregate determined should be considered an estimate.

At VTRC, we find that paste can be distinguished from aggregate on the basis of color, luster, internal structure, and the sort of surface produced by the lapping procedures. However, we have seen aggregate particles that so closely matched the paste in color and luster and finely lapped texture that it was possible to miss small corners of coarse aggregates and fragments of sand and be quite uncertain about the exact location of the boundary between these phases. Strangely enough, carbonate aggregate is usually distinguishable by color, luster, translucency, and crystal structure. It is usually the light brown to creamy gray quartz pebbles and sands that are the most difficult to distinguish from paste.

When aggregate particles are lapped in a slice of concrete, they are leveled off to the level of the paste and a fine-matte surface is produced on the aggregate surface remaining in the slice of concrete (see Fig. 7-3). This matte surface will be a little different for each lithology of the aggregate in the surface, but it is generally sufficiently different from a broken surface or a natural water-worn surface of the type of aggregate for a technician to be able to distinguish a matte-lapped surface from all others. Because of the higher capillarity of the paste and greater hardness of the aggregate, the matte surface on an aggregate particle is usually quite different from the surface of finely lapped paste. The lower the water-cement ratio, the denser the paste becomes and the more the lapped surface on the paste becomes like the lapped surface on the aggregate. When the problem of distinguishing paste from aggregate becomes difficult, the differences in solubility in weak acid or the differences in porosity indicated by dye absorption can be used to differentiate between aggregate and paste.

The problem cannot be solved by adhering to such statements such as: "The paste content calculated from the known mixture proportions is approximately 12 percent higher than the one obtained from the ASTM C 457 measurements" (Pleau et al., 1990). Pleau et al. called this sort of error "an unavoidable artifact of the measurement process." In their work, no effort seems to have been made to enhance the paste-aggregate boundary and lessen their error. They used the point-count method, determining paste concurrently with the analysis of the air content. They explain the difference between their mixture proportion and their point-count results by stating that there are errors made in determining the proper outlines of aggregates when the aggregate occurs close to the surface being examined. In their specimens, the paste seems to be more translucent than in our specimens. Reportedly, their operators frequently see through the paste and count an aggregate particle be-



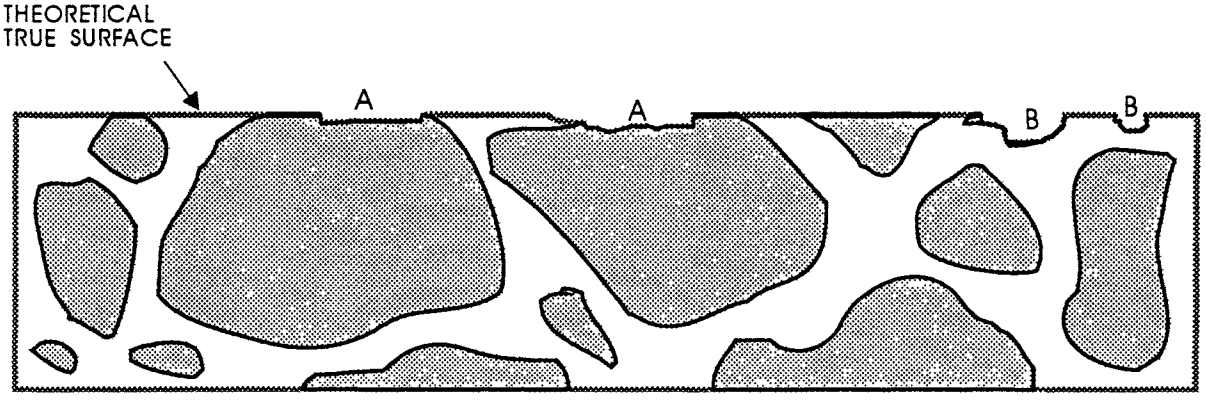


Figure 7-3 FLAWS IN PASTE. Illustrates the type of flaws caused by aggregate that is fragile and recognizable by the broken aggregate surface (marked *A*) and the type of flaw caused by complete or nearly complete loss of an aggregate particle (marked *B*) and recognizable by the shape of the cavity remaining and the texture of the paste surface within the cavity.

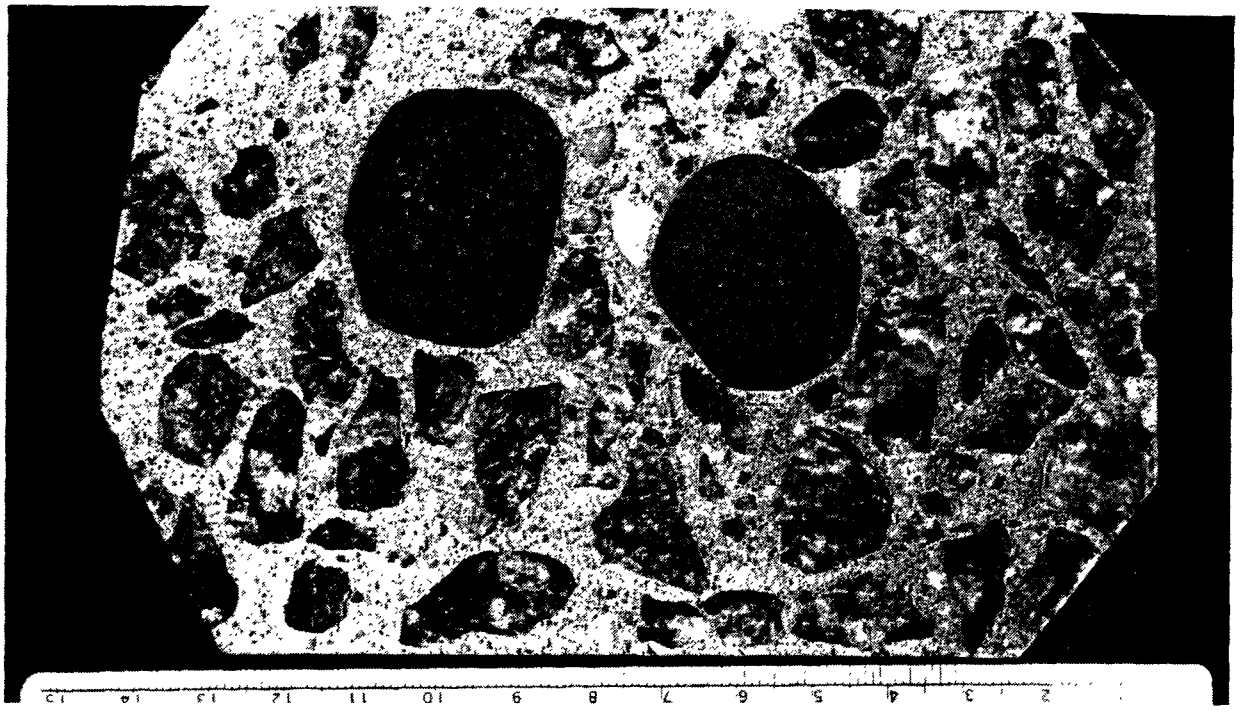


Figure 7-4 KNOTS OF CEMENT EXPOSED ON FINELY LAPPED SLICE. Actual size. The rounded shape is caused by tumbling in the mixer.

neath. Also, they have occurrences of discolored portions of the paste that appear to be aggregate particles and become counted as such.

Other than the dark blue-green blotchy appearance often seen in pastes containing GGBFS, the only such "discolorations" seen in the VTRC laboratory have, when studied in thin section, been determined to be lumps of cement (see Fig. 7-4) caused by either exposure of the cement to moisture during storage or an improper mixing sequence. In retempered concrete (see Appendix D), some of the aggregate may have coatings of dense, partially hydrated cement. Obviously, counting a cement lump or coating as aggregate in the point-count method will decrease the ratio of paste to aggregate determined. Although Pleau et al. (1990) found a consistent 12% shortage in the microscopically determined paste content in the laboratory-mixed specimens they used, they did not present any evidence that indicates that all paste contents determined microscopically should be increased by such an amount.

The percentage of paste is determined in six steps, as listed in Table 7-1.

1. Etch the slice if necessary. Determine if etching the slice will enhance the visibility of the boundary between the aggregate and the paste. Test etchings can be performed by dropping a small quantity of acid on companion surfaces or even on the slice under examination. Most users of point-count or image analysis equipment make the paste determination concurrent with the air-void determination and forgo the greater definition of boundaries available by etching. When the color of the paste and the aggregate match and the lapped surfaces are similar, the determination of the exact boundaries between aggregate and paste is very difficult. If the results of the determination will have to be presented as legal evidence and perhaps

Table 7-1
PROCEDURE—DETERMINING PERCENTAGE OF PASTE

1. Etch the slice if necessary.
2. Examine the etched slice, and become familiar with the way the paste and aggregates reacted to the acid.
3. Prepare to recognize the true lapped surface of the aggregate.
4. Survey the slice to become familiar with any features that could cause confusion.
5. Perform the microscopical determination of the amount of aggregate in the slice.
6. Calculate the percentage of paste.

questioned by opposing expert witnesses and sufficient specimen material is available, VTRC subjects the specimen surface to a *very* short etch (see 5.2.3) so that the exact aggregate-paste boundaries can be distinguished by the different solubilities of the phases in the dilute acid. Avoid etching the specimen to determine an accurate paste percentage unless sufficient slices can be obtained so that the slice etched will not have to be used for other purposes and an unetched slice will remain available for archival purposes.

At VTRC, we find it more accurate and efficient to perform a count of a single substance at a time and almost always etch the surface before performing a paste determination.

> *CAUTION: Perform the etch procedure after the air-void determinations have been made because the acid will round off the void edges and make the voids appear larger. Therefore, if an acid etch is to be used, determining the paste content must be a separate procedure from the air-void determination.*

2. Examine the slice, and become familiar with the manner in which the paste and the various lithologies of aggregate reacted to the lapping and the acid. Usually, the paste is more soluble than the aggregate and is etched down to a lower level (see Fig. 7-5A). Occasionally, there may exist pieces of pure calcite in the aggregate. Calcite itself is much more soluble in weak HCl than concrete paste and will be dissolved away to an even lower level. Thus, there exists a solubility difference, and the boundary can be distinguished. Unless the aggregate rock used is exceptionally rich in pure calcite, this should not occur often. Impure carbonate (calcitic and dolomitic) rocks are not removed to such a great extent. The pyrites, clays, and other minerals included in the more complex carbonate rocks remain at the level to which the specimen was lapped even if a layer of the carbonate is removed (see Fig. 7-5B).

3. Prepare to recognize the true lapped surface of the aggregate as distinguished from any broken or water-worn natural gravel aggregate surfaces. Whether the paste determination is made on a flat lapped surface or an etched surface (whether the quantity of paste is determined at the same time as the quantity of voids or not), think clearly about the fact that the matte surface on the aggregate is the portion of the aggregate that exists on the plane on which the determinations are being made. Other surfaces of the aggregate will have a natural broken or water-worn surface. Figure 7-3 illustrates (1) the type of flaws caused by aggregate

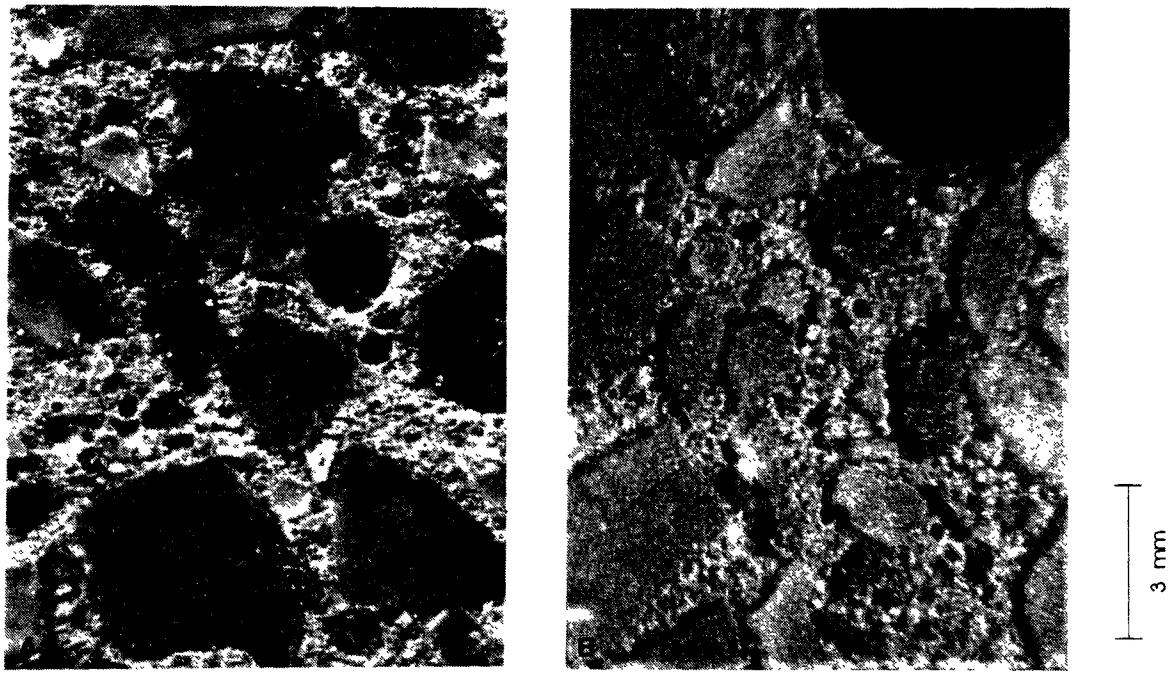


Figure 7-5 ETCHED SLICES. A. Etched surface on a concrete fabricated with quartz sand fine aggregate. **B.** Etched surface on a concrete fabricated with crushed limestone fine aggregate.

that is fragile and recognizable by the broken aggregate surface and (2) the type of flaw caused by the complete or nearly complete loss of an aggregate particle and recognizable by the shape of the cavity remaining and the texture within the cavity. These flaws are not a common occurrence, but when present, a mental reconstruction of the surface will usually indicate that they should be counted as aggregate. If these flaws are common and a mental reconstruction of the true surface does not indicate the proper location of the boundaries between paste and aggregate, the slice should be refinished or replaced with one of better quality. For highest accuracy, the air-void determination should be made on the better prepared slice. In cases other than the flaws indicated in Figure 7-3, refrain from recognizing as aggregate any area that is below the finely lapped surface. If a part of the aggregate that should be covered with paste is exposed, count such areas as paste. If the paste surface has been removed from over an aggregate surface (chipped off or etched off), the aggregate particle will not exhibit the finely lapped matte surface of the properly exposed aggregate particle and it is likely that at a magnification of 100X (or larger) there will be a decided difference in the location of the planes of focus between the true lapped surface and any aggregate surface beneath. This difference in focus (which must be adjusted for if the surface is to be kept in focus) should alert you to the fact that you are viewing a surface that is further from the objective lens than was the surface on which the focus was originally located.

None of these problems should cause errors if you think clearly about the view seen and mentally reconstruct the view that would be seen if the paste area on the lapped surface accurately indicated the true proportion of paste present. Avoid errors by allowing sufficient time for the analysis. Figure 7-6 illustrates some of the flawed and etched artifacts that may be observed in surfaces during the microscopi-

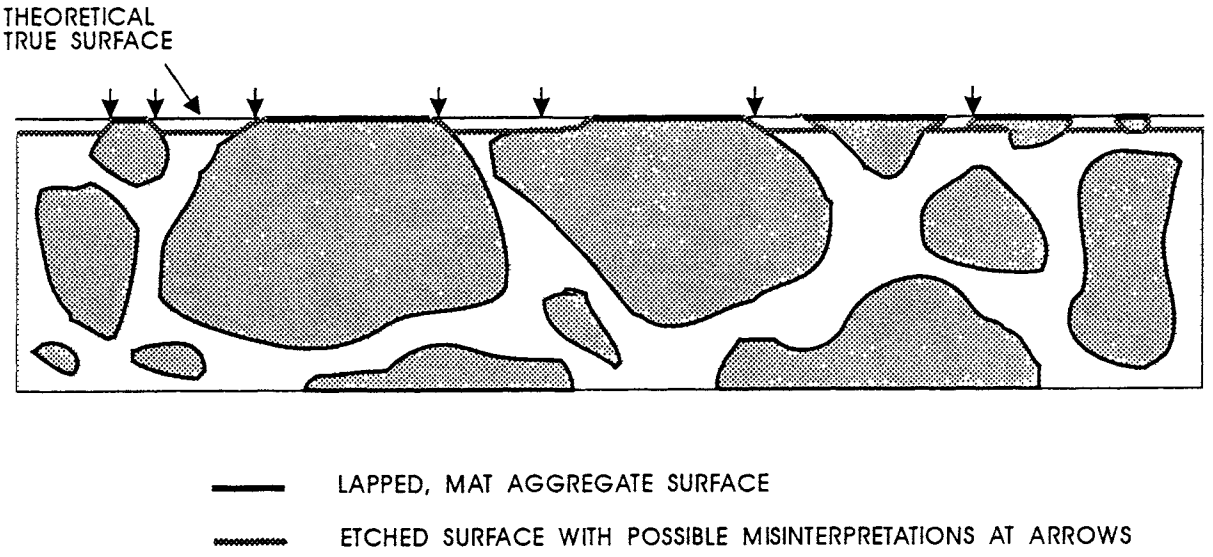


Figure 7-6 CROSS SECTION OF SURFACE DEMONSTRATING PROBLEMS OF BOUNDARY DISTINCTION. This is an acid-etched surface that shows the need for counting as aggregate only the matte-lapped surface of the aggregate.

cal determination of the paste-aggregate ratio. These surfaces are different from the undercut surfaces discussed in 5.2.1 (see Fig. 5-1). The undercut surfaces have a somewhat similar high relief, but the features are rounded and there is no way a mental reconstruction of the surface will yield a good estimate of the position of the paste-aggregate boundary. Undercutting must be overcome by proper surface preparation.

4. Survey the slice and become familiar with any portions of the paste that could be mistaken for aggregate because of coloration, carbonation, or other factors. Paste that is dark because of concentrations of unhydrated cement or GGBFS or paste that has been carbonated and therefore not etched as deeply as the surrounding paste will not have a lapped matte surface as the aggregate particles do. Although the calcite in carbonated areas is soluble in HCl with effervescence, these areas are seldom etched as deeply as uncarbonated paste. The calcite is completely dissolved, but usually there remains a porous layer of material that is not soluble in the acid and is often as high as was the original lapped surface. It appears that the siliceous components of the uncarbonated paste are more soluble than the siliceous portion of the carbonated paste.

5. Perform the microscopical determination of the amount of aggregate in the surface. With the exception of the kinds of flaws shown in Figure 7-3, be careful to count as aggregate only those portions of the aggregate that were at the surface when the air-void determination was made, i.e., the high matte-lapped surfaces of the aggregate.

6. Calculate the air-free percentage of paste according to the following formula:

% Paste = 100% – (% Aggregate + % Air voids).

7.2 AGGREGATE AND OTHER SUBSTANCES

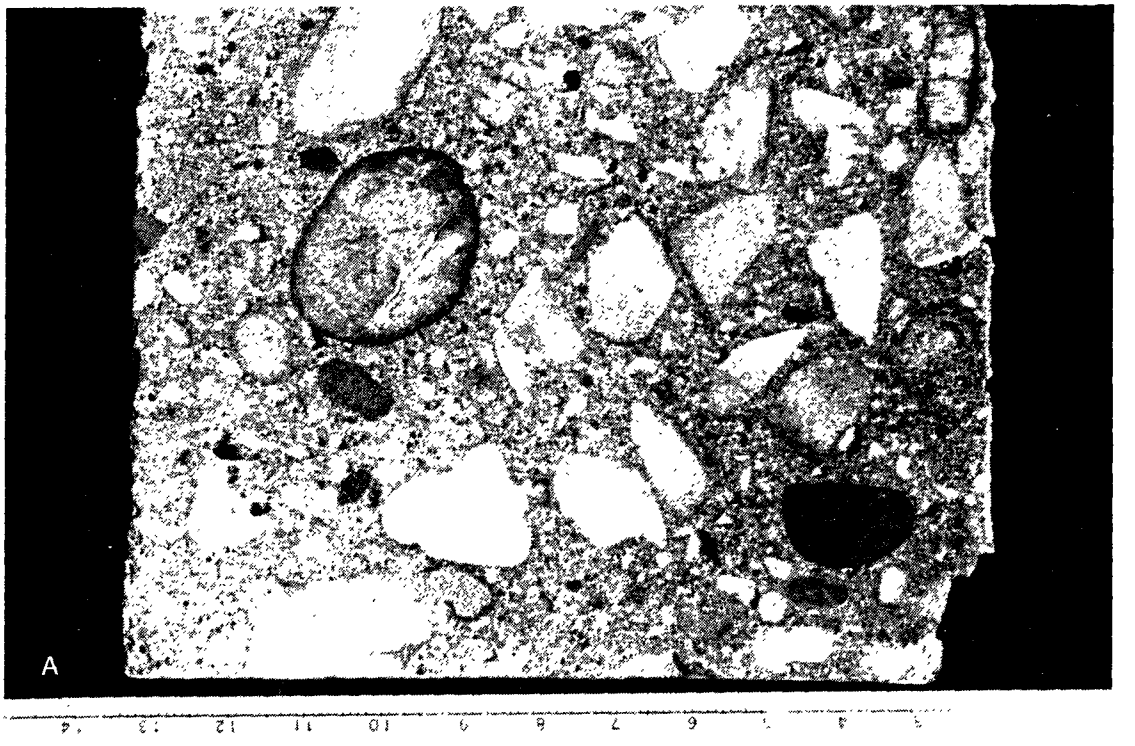
The percentage of a specific type of aggregate or other substance should be determined when deemed important by the client or the petrographer. Any substance that can be recognized when seen on the surface of the slice as being composed of a particular material can be counted by point-count or measured by linear traverse equipment and an estimated volume percentage of the substance determined. The accuracy of such determinations is dependent on the frequency of occurrence of the particles of the substance, as discussed in 7.1.2.

Substances cannot be distinguished on the basis of size. It is possible to know only that the cross section exposed of an object indicates that the object is large enough to have the particular cross section. The object may be much larger.

Occasionally, the petrographer is requested to determine the relative amounts of coarse aggregate and fine aggregate. If there is a distinct lithologic difference between the two (e.g., a fine-grained greenstone coarse aggregate and quartz sand fine aggregate), an estimate of the percentage of each can be made using point-count or linear traverse equipment. If there is no readily recognizable lithologic difference (most often the case when crushed limestone is used for the fine as well as the coarse aggregate), it is impossible to make a percentage determination of the relative amounts of these materials by readily available optical methods (see 12.3). Calculation of diameters from chords such as that of Lord and Willis (1951) that are dependent on the spherical shape of the item measured cannot be used for aggregate particles. The distinction cannot be made on the size of the area of the particle exposed on the lapped surface because a piece of coarse aggregate may be almost hidden, with only a tiny corner showing. At present, the only method available is the removal of the paste with an acid and the sieve analysis of the aggregate. An acid that attacks the aggregate cannot be used. Limestone is usually soluble in acid.

An experienced petrographer will usually be able to tell by comparison with other specimens of concrete if an unusual amount of either coarse or fine aggregate is present. The original design of the mixture should indicate the sizes intended to be used (see Fig. 7-7).

If the petrographer feels that the aggregate is not sized according to the intended grading, an investigation of the sizes of the materials in the stockpiles can be made. If the sizes in the stockpiles are within specification, the problem may be in the proportioning of the aggregates during fabrication of the concrete mixture.



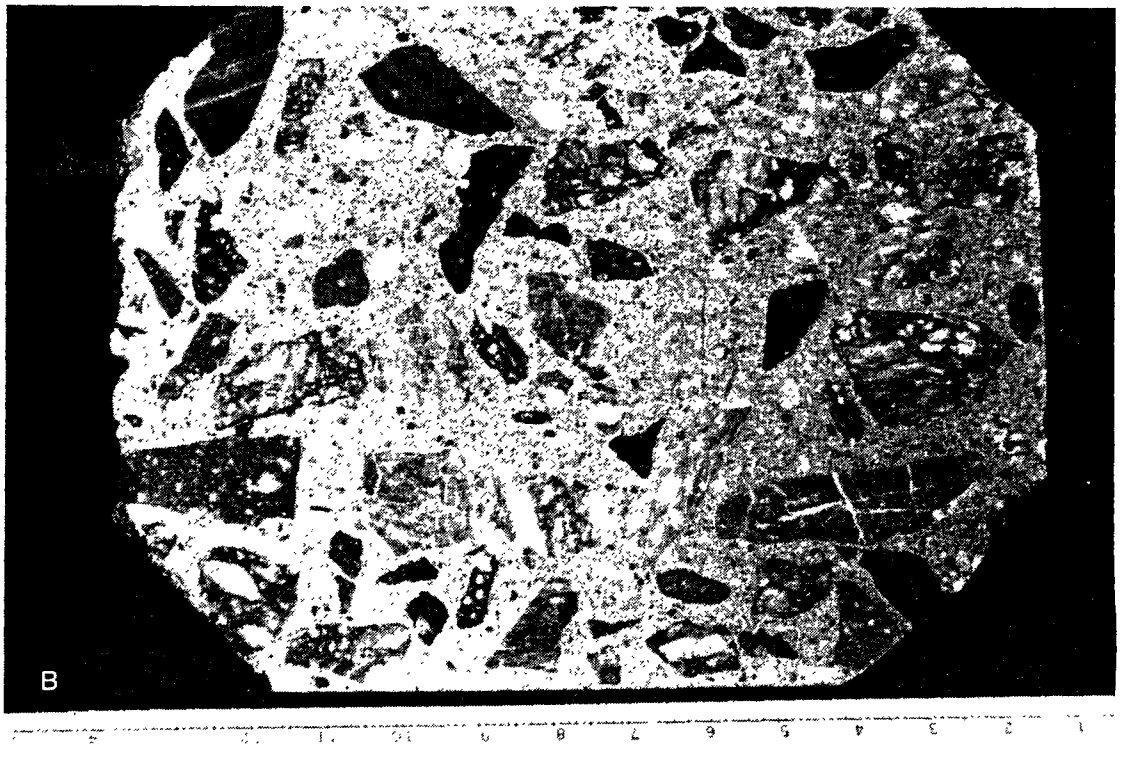


Figure 7-7 VARYING AMOUNTS OF AGGREGATE SIZE FRACTIONS. Actual size. **A.** Concrete fabricated without larger sizes of coarse aggregate. **B.** Concrete fabricated with coarse aggregate that is larger than is now normal for bridge deck concrete.

Chapter 8
EXAMINATION WITH THE STEREOMICROSCOPE

8.1 OVERVIEW

The stereomicroscopic examination of lapped surfaces of the specimen is usually the procedure that controls the course of the analysis of the specimen. If the specimen was submitted for a specific analysis and the petrographer is fairly sure that the other parameters of the concrete do not require investigation (as may be the case when the concrete has been fabricated in a concrete laboratory), the stereomicroscopic examination of the concrete may be omitted. Usually, the petrographer cannot be sure that the examination requested will provide sufficient information to discern all of the possible problems of the concrete submitted and will perform a general stereomicroscopic examination to allow analysis of all the features of the specimen.

The concerns of the client must be considered throughout any examination of the specimen. However, the observations made should not be confined to these concerns: all the features of the entire suite of specimens should be inspected and examined in detail. Often, the petrographer is asked to confirm or deny the presence of a certain form of distress but then finds that there are other kinds of problems present as well.

After any planned quantitative analysis of the constituents (see Chapters 6 and 7), five procedures are performed in a general examination with the stereomicroscope, as listed in Table 8-1.

Table 8-1
PROCEDURE—EXAMINATION WITH STEREOMICROSCOPE

1. Review the data.
2. Prepare the equipment.
3. Examine the slice, and mark and label it appropriately.
4. Enhance the marked features.
5. Photograph the slice, and make photomicrographs.

8.2 REVIEW OF DATA

In the normal course of events, the preliminary examination (described in Chapter 3) is performed and then the specimen is prepared according to the preliminary plan of analysis. If the plan of analysis includes the determination of the air-void parameters, the procedure is usually performed by a technician. Thus, several days may have elapsed between the original preliminary examination by a petrographer and the stereomicroscopic examination. Occasionally, the plan of analysis may have been sketched out by a different petrographer than the one performing the stereomicroscopic examination. If the petrographer's workload is heavy, the time when it is convenient to perform the stereomicroscopic analysis of the specimens may be a

few weeks after the receipt of the specimen. Therefore, it is usually necessary that the petrographer review the complete history of the HCC and be aware of anything unusual about the design of the mixture and any unusual procedures or occurrences during placement. With this information, the microscopist will be best able to notice and report on any features that might be attributable to new methods, materials, or admixtures or that might have been caused by the incidents that occurred during the placement of the HCC. Therefore, the petrographer should review the following: (1) the history of the concrete being studied as reported by the client either in accompanying documents or orally, (2) any unusual methods of sample preparation that were required, (3) any features noted by the technician during the preparation of the specimens and the air-void analysis, and (4) the results of the air-void analysis.

8.3 PREPARATION OF EQUIPMENT

The stereomicroscope (see Fig. 2-17) should be placed on a table or stand that is a convenient height for the microscopist. A variety of microtools (see Fig. 2-18), bottles, and droppers to supply water and 10% HCl and a variety of marking equipment to make both temporary and permanent marks on the surface of the lapped slice should be available. A variety of pens; soft, colored pencils; and sticky paper arrows, dots, or other labels for use on the surface of the lapped slice should also be available.

Various pens should be tested on some fine microcracking on an unimportant lapped, oil-free slice of HCC. No particular brand of pen is recommended because inks and pens change as manufacturers find new ways to please the public or economize their operations. The soft or fiber-point disposable pens that have a medium-thick fluid ink work well. While using the stereomicroscope, the microscopist positions the pen on a microcrack and causes a very small portion of the ink to flow on the visible expression of a crack. If the ink enters the crack (as opposed to beading up on the surface) and by capillary action is drawn a short distance along the crack (without bleeding into the mass of the paste), this small leading portion of the ink, visible with the stereomicroscope, indicates the next direction of the crack. By the use of the proper pen to trace the direction of the crack with ink, it is possible to detect and mark microcracks and connections between cracks that cannot be seen against the general paste background at the magnifications of the stereomicroscope. If such pens are available in several colors that will contrast with the HCC, various features can be marked with different colors.

Other pens and pencils are for marking on aggregate surfaces. Light pencil marks are not easily seen on the lapped surface of paste, and heavy pencil marks may damage the surface or fill cavities with graphite or colored flakes.

8.4 EXAMINATION AND MARKING OF SLICES

Table 8-2 is a checklist of the features that should be examined. The following procedure should be used:

1. When there is any possibility of the labeling obscuring your ability to see details in the paste (usually the case when using ink to emphasize microcracks), delay any permanent marking and labeling on the paste until you examine the slice for all the other items on the checklist. When

Table 8-2
CHECKLIST FOR EXAMINATION WITH THE STEREOMICROSCOPE

Item	Remarks
____**Cross sections of exterior surfaces** (quality of original surface texture, present quality of surface texture)	
____**Foreign objects**	
____**Reinforcing bars, supports, or both**	
____**Voids**	
____General appearance	
____Shape	
____Distribution	
____Size	
____**Aggregate**	
____Lithology and mineralogy	
____Orientation	
____Aggregate-paste ratio	
____Distribution	
____Cracks	
____Coatings	
____Aggregate-cement reactions	
____**Paste**	
____Water-cement ratio	
____Discolored areas	
____Carbonation	
____Cementitious particulate materials (GGBFS or pozzolans)	
____Cracks at aggregate bond	
____Cracks within paste	

marks and labels can be placed on aggregate surfaces or the labeling can be easily removed, perform such marking as instructed under the various features (concurrent with the general stereomicroscopic examination). Features that may be easily observed with the stereomicroscope may be difficult or impossible to see with the naked eye and are often impossible to record photographically unless they are enhanced by some form of marking or emphasis that can be seen without magnification.

NOTE: Most of the features of the lapped slice are easier to see if any shine caused by the lapping oil has been removed from the slice by evaporation (sufficient exposure to room air or overnight treatment in a warm oven). Check the slice during the last stages of the evaporation process to make note of any areas that have absorbed extra oil. Such areas may indicate cold joints, overlay bonds, boundaries of carbonated zones, or transitions between various qualities of HCC.

2. Examine any cross sections of formed, finished surfaces or wearing surfaces on the slice. The surface of a concrete placement that is not against a form and is generally smoothed and finished by a float, trowel, or texturing device (such as a tine rake or burlap drag) is the finished surface. Ascertain if the paste within a few millimeters of the finished surface is about the same color as the rest of the paste and if the air-void content of the surface layer is not abnormally high. (If the concrete was too stiff to finish easily, the contractor may have added water to the top layer and thus changed the consistency.) A light-colored surface layer (may be no more than 1 mm thick), often with a froth of very fine air voids, may be evidence of the use of excess water during the finishing procedures or the occurrence of rain during the finishing process. Such a froth will not have much strength and will wear away rapidly, but, fortunately, such zones are usually very shallow and do not greatly affect the durability of the placement.

Overworking with the finishing equipment can cause a thin layer at the surface to be exceedingly rich in paste and low in aggregate and air. Excess water will weaken the concrete and sometimes cause cracking and crazing. Excess and misshapen voids, zones of streaked paste, and zones low in air are additional evidence of problems occurring during finishing (see Fig. 8-1).

Unless the concrete is old and worn by heavy traffic, the texture of the wearing surface or that of any finished surface should conform to the texture specified. Thin sections that show the profile of cross sections of the surface can be fabricated (see 5.3.3). Skid resistance is aided by the production of asperities and the provision of channelways in the surface for the escape of water in order to prevent skidding and hydroplaning. If the specified texture is missing or the grooves and lands are sloppy and misshapen, a heavy rain during the finishing procedures or poor



Figure 8-1 EXCESS AIR AT SURFACE OF CONCRETE (*Top*). Water was added to facilitate finishing. The elongated, angular coarse aggregate caused the mixture to be difficult to place. Notice the angular voids that exist down to a depth of 10 mm. The scale is in millimeters.

workmanship is the probable cause. Texture is important for any surface on which wheeled traffic travels. If the texture is insufficient, the petrographer should consider suggesting the sawing of grooves or, in the case of old concrete, replacing the layer by adding a textured overlay.

Report the condition of the texture on any surface on which traffic moves. Report any differences between the microstructure of the major portion of the paste and the paste adjacent to any exterior surface.

3. Note the presence of any foreign objects in the concrete. Such objects might be pieces of glass, wood, metal, or fabric. If such objects are common in the concrete under study, the cause is either a massive accident occurring nearby while the concrete was fresh, poor workmanship, or sabotage. Look for the evidence of any chemical reactions between such foreign objects and the chemicals of the paste. Some glasses will react with the highly alkaline paste and cause deleterious expansion. Fine aluminum fragments will cause the evolution of hydrogen gas, concomitant voids, and zones of weakness. Other substances may cause other reactions.

4. Report any portions of reinforcing bars or portions of the supports for the bars (called *chairs*) present in the slice. Rust-colored portions of the lapped surface may indicate nearby corrosion of ferrous metal. Check the back of the slice, the opposing sawed surface, and the remaining (unsliced) portions of the specimen for corroded reinforcing bars and related material. Report their presence.

5. Examine the voids:

- *General appearance.* Examine the appearance of the void system. It should be in conformance with the data obtained by the air-void analyses. Consider the number of voids and their size. If the voids are unusually small and numerous, the air content may be greater than it first appeared. If there is an apparent discrepancy, (1) check to make sure that the air-void analysis was conducted on the slice being examined and the analysis was performed properly and spread over the entire surface of the slice, or (2) request that the air-void analysis be repeated and/or conducted on additional slices. In the report to the client, discuss an apparent conflict between data obtained in accordance with ASTM C 457 and that seen with the unaided eye.
- *Shape.* The hydrostatic pressures within the unhardened HCC paste cause the small voids to tend to a spherical shape. Most of these are the entrained air voids. They are sometimes mentioned in the literature as if the sphericity defined them. In extreme circumstances, even these small voids can become distorted by various forces. The shape and distribution of the various types of voids are important. Note any areas of unusual void angularity, and describe or label the location.

  Voids that are within the thin zone that becomes worked as the concrete is finished and textured can be quite angular. It is only the extremely small voids that maintain their individual integrity when they have been distorted by these procedures. The larger voids are usually broken and thus expelled from the surface, but many of the tiny ones remain, often angular and distorted. A minimum magnification of 100X is required to discern the presence, shape, and condition of very small voids.

  Rarely, retempering (see Appendix D) that occurs after the paste has started to harden and the individual voids have developed a "skin" will cause the ma-

jority of the medium and small voids in a concrete to take on an ovoid shape. Most commonly, the ovoids will have their long dimension vertical. This is a very rare condition and must not be considered necessary evidence of retempering. Much retempered concrete exists without the presence of any ovoid or otherwise distorted entrained air voids.

- *Distribution.* When the voids are unevenly distributed (see Fig. 8-2), the location of large concentrations of voids can be important, particularly if the voids are consistently associated with certain other features, such as a particular type of aggregate. If these concentrations are thought to be an important indicator of the condition of the concrete, mark or otherwise make note of their location on the slice. (See the description of types of air voids in Table 6-1.) Concentrations of voids that do not appear to be related to any other specific feature may often be attributed to retempering or incomplete mixing.
- *Size.* The overall void system (including the size of the voids) is measured and documented by the analysis of the air-void parameters as detailed in Chapter 6. However, if there is anything unusual about the size of the voids that can be seen with or without magnification, document it. For example, if one extremely large void (e.g., 50 mm by 15 mm) is present in the slice being examined and it is decided that the void is not typical of the overall concrete, the area of the slice in which the void occurs is excluded from the area examined in accordance with ASTM C 457. Such a void should be measured, and its shape and internal surface described in the notes and report on the specimen.

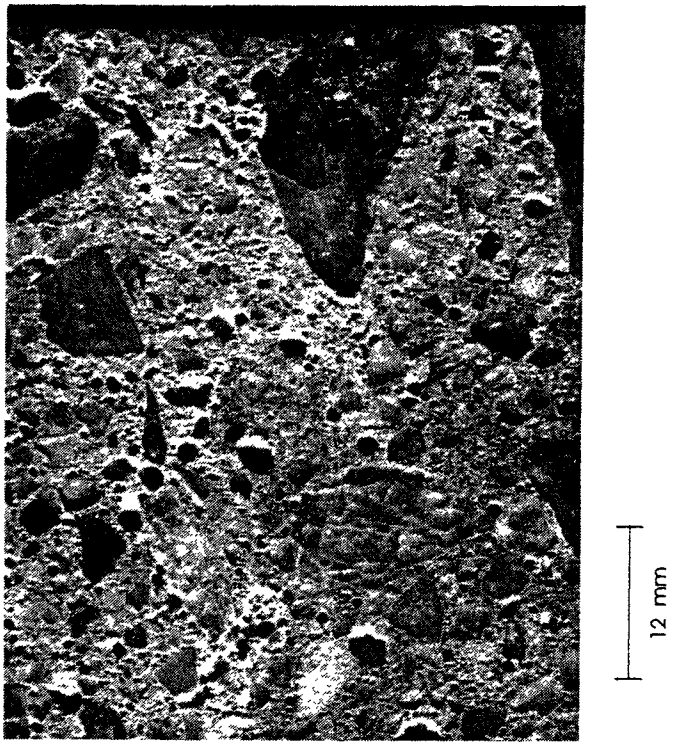


Figure 8-2 VOIDS OCCURRING IN BUNCHES. In this case, the bunching is apparently due to excess air-entraining admixture and incomplete mixing.

6. Examine the aggregate:

- *Lithology and mineralogy.* Briefly describe the general types of rock and minerals seen in the aggregate of the HCC being examined. Include particle shape and color in the description. This description can be very helpful when you are trying to find a particular lapped specimen among all the specimens in the laboratory. If a particular type is present in only a very small amount, it may be a contaminant and should be so noted. As an example, if the major portion of the coarse aggregate is crushed limestone and a few rounded pebbles of quartzite are present, the quartzite is probably a contaminant and may indicate poor workmanship or an unusual incident occurring during mixing or placement.

  If the aggregate does not seem to be the aggregate specified in the design of the mixture as reported in the accompanying documentation, contact the client to resolve this inconsistency. Are the specimens received the ones that were meant to be sent? Is the documentation incorrect? Is there some other explanation? To achieve assurance in the identification of the aggregate, it may be necessary to request bulk specimens of the specified aggregate and perhaps specimens of aggregate from other sources; perform a detailed study of the frequency of occurrence of the various lithologies; determine particle shape, soundness, staining, natural rims, and cleanliness; and study the microstructure and optical properties of these aggregates in thin sections with the petrographic microscope.

  Take note of any deterioration of the HCC that is associated with any particular lithology. Such deterioration includes microcracking; popouts on the surface; reaction rims; and any deposits, particularly those that might indicate an expansive reaction (see Chapter 10).

- *Orientation.* In some HCC specimens, the preferred orientation of the long dimension of the aggregate particles or general parallelism of flaky aggregate particles can be observed. Such orientation may indicate an abnormally high water-cement ratio, improper mixing or placement procedures, or both. Note any patches of paste that seem particularly devoid of aggregate and, conversely, any areas in which the aggregate is crowded together. These evidences of segregation of the unhardened concrete mixture may indicate an abnormally high water-cement ratio, improper timing of the placement sequence, or just generally shoddy workmanship.

- *Aggregate-paste ratio.* An experienced concrete petrographer can usually tell at a glance if the aggregate-paste ratio is within the normal range of 75-25 to 59-31 (see Figs. 7-1 and 7-2). A more exact determination can be made with linear traverse, point-count, or image analysis methods. The precision of the determination of this ratio with linear traverse or point-count equipment is not well known. In the case of image analysis, if the area of surface examined is sufficient and representative and the toning and other sample preparation procedures are carefully carried out, the results should be quite accurate.

- *Distribution.* If the heavier and larger pieces of aggregate have settled to the bottom of the placement, it can usually be assumed that the segregation took place because the mixture contained an excess of mortar or the paste was too fluid because it contained excess water. Occasionally, it has been found that

such segregation should be attributed to excessive vibration during consolidation of a mixture having more water than desirable.

If there is a zone at the top of the placement that is nearly devoid of aggregate but the aggregate distribution appears normal in the remainder of the placement (as in Fig. 8-3), the cause of this distribution is probably the addition of water to the mixture after placement. This water may be added by a rainstorm or possibly even purposefully added to facilitate placement.

- *Cracks.* During the study of cracks in the aggregate of hardened concrete, give continual thought and recognition to the fact that the aggregate has withstood the stresses of concrete fabrication.

  If the aggregate cracks are narrow (or do not seem to separate the aggregate into fragments) and the cracking seems to be in a regular pattern that echoes the surface of the aggregate, the cracking may be due to the intrinsic cleavage or parting of the aggregate. Shaly rocks and monomineralic aggregate particles of certain minerals might be good examples of this type of cracking.

  Cracks in the aggregate that do not appear to be due to the structural nature of the material of the aggregate, break the aggregate into separate pieces, or start in the aggregate and extend out into the paste may be due to reactions and forces to which the concrete has been subjected since hardening. For an example of this, refer to Chapter 10.

- *Coatings.* Note any coatings on the surface of the aggregate particles (such as clay, iron oxide, or manganese oxide). These coatings may help identify the environment, source, and degree of beneficiation of the particles (Ozol,



Figure 8-3 OVERWATERED CONCRETE. Rain and snow occurring after the concrete was placed caused overwatering near the surface (*top*). The aggregate sunk out of the overwatered zone.

1978). Such coatings, if porous, may act as reservoirs of water that cause weak zones of high capillarity in the hardened concrete.

- *Aggregate-cement reactions.* Make special note of the presence of evidence of chemical reactions of the aggregate with the paste, i.e., rims, cracks, or reaction deposits, such as gel. Refer to Chapter 10.
- *Specialized aggregate materials.* Refer to Appendix E for descriptions of specialized aggregate materials.

7. Study the paste:

- *Water-cement ratio.* Estimate the water-cement ratio of the paste as either high, medium, or low by means of the following factors: (1) the observable texture of the paste; (2) the distribution of unhydrated cement grains; (3) the speed with which water sinks into a clean, oil-free surface; and (4) the manner in which the paste reacts, as observed with the microscope, when scratched and picked at. The petrographic evidences of the water-cement ratio in HCC or mortar are very subtle. Generally, they cannot be considered as hard firm evidence but may be used to indicate the necessity for a chemical determination of the cement content. Further details may be found in Chapter 9.

  Because of retempering (see Appendix D) or other mixing conditions, HCCs may have areas of low water-cement ratio and areas of high water-cement ratio in the same placement and even in the same specimen or slice. The strength of the HCC will depend on which type of concrete is most prevalent and most continuous through the mass.

- *Discolored areas.* Take special note of areas of the paste that are a different color than the remainder of the paste. If the concrete mixture contains GGBFS, is quite young, and has been kept moist and the dark areas are a dusky bluish green, the areas of concern are probably nothing more than the wetter zones of the GGBFS-bearing HCC that are commonly this color until the material has dried and oxidized for the first time (Mather, 1957). Later moistening of GGBFS HCC will not recreate the color.

  Areas of a darker color than the major portion of the paste are probably zones with a high cement content and hence a lower water-cement ratio and were probably formed before all the water was added. If sufficiently hydrated, these areas may be very strong. If dry cement is encapsulated in these areas, it will have very little strength and the area will be a zone of weakness. Such areas may indicate retempering (see Appendix D) or improper mixing. They occur in two general types: (1) as coatings on and in reentrant angles of the aggregate particles, and/or (2) as knots or blobs of various sizes in the main area of the paste (see Fig. 6-3). If such areas are present, mark the slice in such a way that a number of these areas will be noticed in a photograph.

  Occasionally, there are zones near the aggregate that are made up of cement that contain little water. These are caused by moist aggregate having come in contact with dry cement and having picked up a cement coating (see Fig. 8-4). If these coated aggregates are used in a concrete mixture, the very outside of the cement coating becomes moistened in its new environment; if the coating is thick, the inside can be preserved in a dry condition. If this concrete becomes subject to stress before water permeates the cement shells and hydrates them, these dry cement coatings will be a zone of weakness.



Figure 8-4 CEMENT COATING ON AGGREGATES. Damp aggregate came in contact with dry cement and picked up a layer that did not become hydrated. This layer is much denser than the zone surrounding the aggregate in Figure 6-3. Actual size.

Knots of cement (see Fig. 7-3) may be caused by water reaching the cement before use or by certain batch procedures. These knots can be so large and dense that they may be mistaken for aggregates. These areas are usually rounded by the action of the mixer. If a large portion of the cement is caught into balls or aggregate coatings, the remainder of the concrete will be depleted in cement and will have a high water-cement ratio and be weak.

Variations in the color of the paste may indicate variations in the water-cement ratio (see Chapter 9).

If the surface of the slice is rusty looking, it may be because the slice, while it contained a little water, was allowed to set for many hours on a nonmoving iron lap. Question the support staff concerning this and, if necessary, remind them that laps become pitted by rusting and then must be demounted, resurfaced or replaced, and remounted on the motor assembly.

- *Carbonation.* Carbonation of the paste may be indicated by very slight color or textural variations within the paste or differential absorption of lapping oil. Occasionally, a narrow zone of the paste that is nearest to the carbonated area has been depleted of calcium hydroxide and is sufficiently porous to absorb a larger amount of lapping oil. This absorbent area may be seen as a dark line before the slice is completely oil free. Lightly etch the portion of the slice suspected of being carbonated (see 5.2.3). Carbonation is most prevalent near the surface, along cracks, along "cold joints," and at cracks and aggregate boundaries.

  Carbonation is a process wherein certain constituents of the HCC paste chemically combine with the CO_2 of the atmosphere. The calcium ions be-

come part of the mineral calcite: $CaCO_3$. $Ca(OH)_2$ within the affected zone may completely alter to calcite. Some loosely held calcium ions in the calcium silicate hydrates of the cement paste alter to fine crystals of the carbonate minerals, mostly calcite. When the paste is treated with acid (see 5.2.3), the $CaCO_3$ dissolves with effervescence. The uncarbonated paste is very soluble in the acid, and often the carbonated zone (now with calcite removed) appears porous but remains higher than the etched uncarbonated paste. After acid treatment, the portion that had been carbonated is usually cream or white in color. There is often a ridge of especially high relief at the line of contact between the carbonated area and the uncarbonated area (see Fig. 5-4).

If the etching does not clarify the difference between carbonated and uncarbonated paste, fabricate thin sections for examination with the petrographic microscope. The high birefringence and the fine crystallite size of the products of carbonation, when viewed through crossed nicols, will indicate the presence of this alteration of the paste (see Figs. 13-8 and 13-9).

- *Cementitious particulate admixtures.* Examine the paste for evidence of any cementitious materials other than portland cement. Fly ash can often be seen on a finely lapped slice of the specimen. Consider using an acid etch (see 5.2.3) to enhance the visibility of the fly ash (see Figs. 8-5 and 11-3). Some of the particulate admixtures of cements, such as GGBFS, cannot be seen on a lapped surface but may be detected in a thin section when studied with the petrographic microscope. GGBFS can be suspected when the color of the concrete is either mottled with the dark blue-green or is the light cream color associated with aged GGBFS concrete, but the GGBFS particles can be seen only in thin section. Particulate materials other than portland cement are discussed in Chapter 11.

- *Cracks at aggregate bond.* Bond cracks are the narrow separations occurring at the bond between the coarse aggregate and the paste. Mark bond cracks by placing a small arrow on the aggregate. These separations are particularly common with quartz-rich rock and gravel aggregate. Water has an affinity for the quartz surface, and this layer of water may remain on the aggregate uncombined with cement as the cement hardens around it. Aggregate surfaces that attract water are often called hydrophilic. Surfaces that repel water are called hydrophobic.

  These bond cracks are more apt to occur in mixtures with a high water-cement ratio. The layer of water attracted to the aggregate creates a boundary void that often becomes filled with calcium hydroxide. Pockets of calcium hydroxide are not as strong as calcium silicate hydrate, and thus weak zones are created. In very old porous concrete subject to the action of running water or ground water, calcium hydroxide in bond cracks may leach out and leave an empty boundary crack.

  Conflicting evidence exists concerning the prevalence of bond cracks in concrete containing expanded aggregate. In some, the aggregates seem to have developed a surface that repels water and makes a tight bond with the cement paste. In others, they seem to have developed as many, if not more, bond cracks as do quartz gravel aggregates (see Appendix E). Be continually on the alert for bond cracks and for any means of explaining their existence.

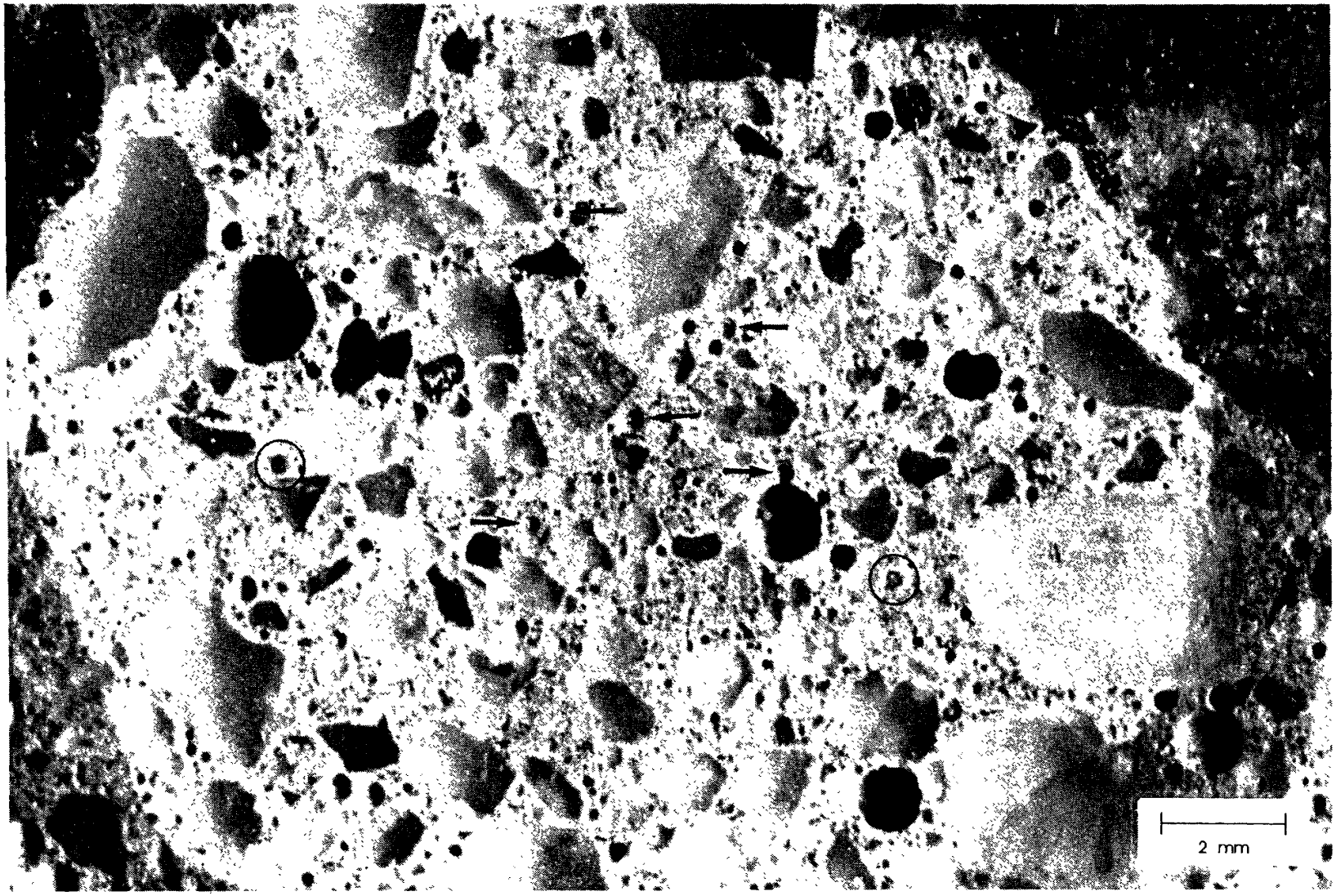


Figure 8-5 FLY ASH PARTICLES ON SURFACE OF LAPPED SLICE OF CONCRETE. They can be recognized by their glassy interiors. The lighter colored fly ash is marked with *arrows*. The black fly ash is *encircled*. If the slice was etched, more fly ash particles would be detected.

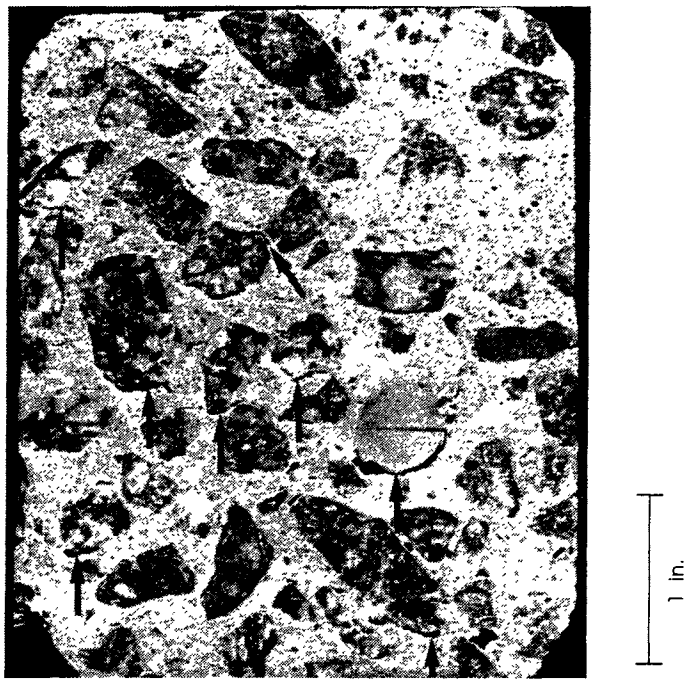


Figure 8-6 CRACKS AT BOND BETWEEN AGGREGATE AND PASTE (See *arrows*). In this instance, the bond cracks occur most frequently on the underside of the aggregate and, therefore, can probably be attributed to bleeding or poor consolidation.

Bond cracks can be serious zones of weakness and contribute significantly to permeability (see Fig. 8-6).

- *Cracks within paste.* Interior cracks in concretes that can be easily seen with the unaided eye are usually observed only in HCC that has been badly deteriorated by structural cracking, drying shrinkage cracking, plastic shrinkage cracking (see Chapter 4), alkali-aggregate reactions (see Chapter 10), or freezing and thawing (see Fig. 8-7). In the early stages of deterioration, they were much smaller. The interior cracks that become visible with close observation and with magnification are called microcracks. Note all cracks within the paste.

  Microcracking and similar fine details are very difficult to see on a rough surface and usually cannot be observed on anything but a well-prepared finely lapped surface. Sometimes, the microcracking seen on a finely lapped interior surface will indicate the general location of the cracks on a nearby rough exterior surface. The thinner the slice, the more relationship will be found between the nearly invisible surface microcracks and the microcracks found on the lapped surface (see Fig. 8-8).

  The field of view seen through a microscope is very limited, and it is very difficult to construct a mental image of how one feature relates to another and be constantly aware of which portion of the specimen is under observation. A crack seen in one view may be followed across the specimen until it disappears, gets to the edge of the specimen, or abuts another feature, but the overall relationship of all cracks to each other can be obtained only by marking the cracks with ink and examining the specimen without magnification.

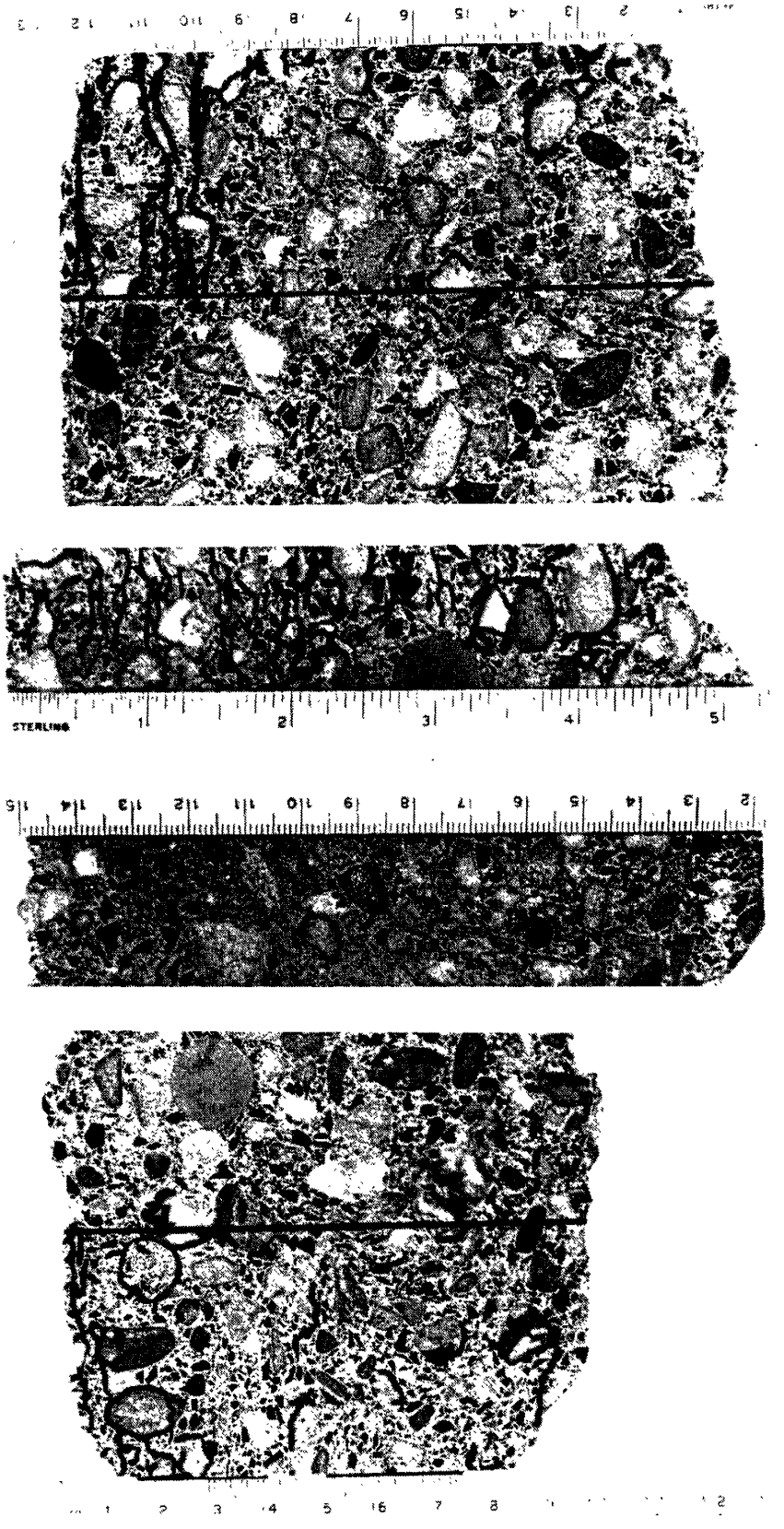


Figure 8-7 TYPICAL CRACKS DUE TO FREEZING AND THAWING. Such cracks occur in non-air-entrained concrete. Cracks are emphasized with ink.

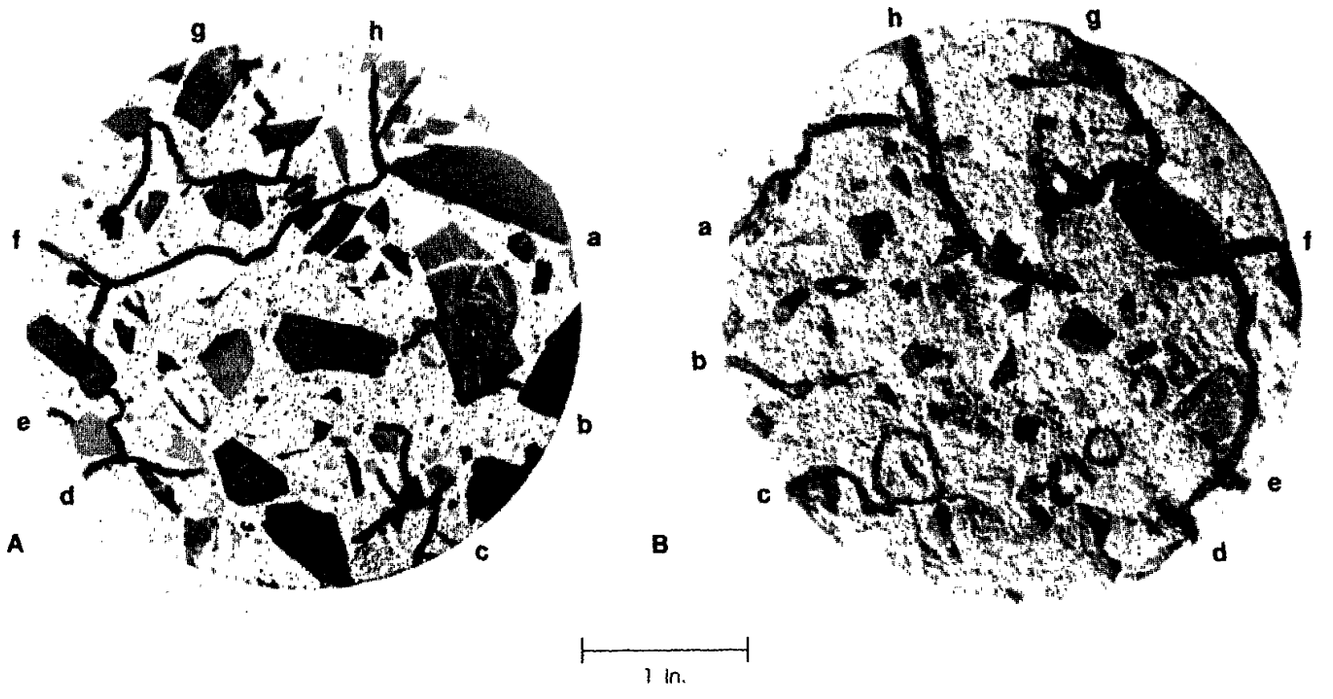


Figure 8-8 MICROCRACKS. A. Smoothly lapped surface with ink-marked microcracks. **B.** Wearing surface near view **A.** The cracks were followed over the edge of the slice. The crack pattern seen in view **A** was used to guide the finding of the cracks in view **B**. The crack lettering system allows comparison of the two photographs. Study these with regard to the fact that these views back up to each other.

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The procedure for marking microcracks in the paste is as follows: While viewing the slice with the stereomicroscope, carefully mark the microcracks with a pen. Use the movement of the ink as it follows the minute cracks by capillary action to find all the connections between microcracks (Walker, 1988). Despite many years of experience, I have to teach my eyes what a microcrack looks like each time I do this task. At first look, I see very few microcracks, but as I start to study the few I see, more and more of them become visible. The visibility of microcracks depends a great deal on the angle of the illumination and the angle of viewing. As the specimen is turned and moved under the microscope, more and more cracks become visible. The mind and eyes become concentrated on the view in the microscope, and the hands become totally engaged in the tasks of marking and moving the slice of HCC. Thus, a sort of hypnotic state is created that leaves no part of the mind free to relate the area being marked to a larger view of the slice as a whole. (I find I cannot talk to anyone or truly listen to conversation without pulling myself away from the microscope.) Many operators find that they are quite startled when the crack being marked extends to the edge of the slice and the crack falls into oblivion.

When control or other comparison specimens of HCC are available, mark the microcracks in at least one slice of the specimens of the control concrete and one slice of any specimen of an intermediate degree of distress (see Appendix B).

This method can be very time-consuming if there is a great amount of microcracking. Moving the slice of concrete under the microscope in an effort to check and mark the cracks and the connecting cracks of the total area can be a seemingly endless task. To get the work done in a reasonable amount of time, a possible procedure is to mark off a randomly selected portion of surface on each slice to be examined and compared. The size of the portion should be governed by the uniformity of the cracking and its frequency (2 by 2 in. might seem like a lot of area when magnified). Do not use a pen to mark off the portion. Ink may follow capillaries into the area to be examined. Use a narrow adhesive tape or a soft pencil of a bright startling color. The marking of cracks and other important features can then be performed on these smaller portions of the surfaces (see Fig. 8-9).

Once the microcracks are marked, the patterns of microcracking will be visible to the unaided eye and varied HCCs can be compared and photographs of the microcrack pattern may be prepared and used as evidence of the true condition of the HCC, as shown by Figure 8-10.

If the placement has been overlaid with another material, such as latex concrete, the client may ask if there is a crack at the bond line. In such a case, study a vertical lapped surface and mark all microcracks with ink. Often, any separation between the overlay and the substrate concrete is not at the bond line but is in the weaker material of the substrate (see Fig. 8-11). This just-below-the-bond cracking is often discontinuous, but freezing and thawing and traffic may well cause complete delamination. This type of cracking indicates that more of the substrate should have been removed before overlaying if maximum durability was to have been achieved. Report all cracks.

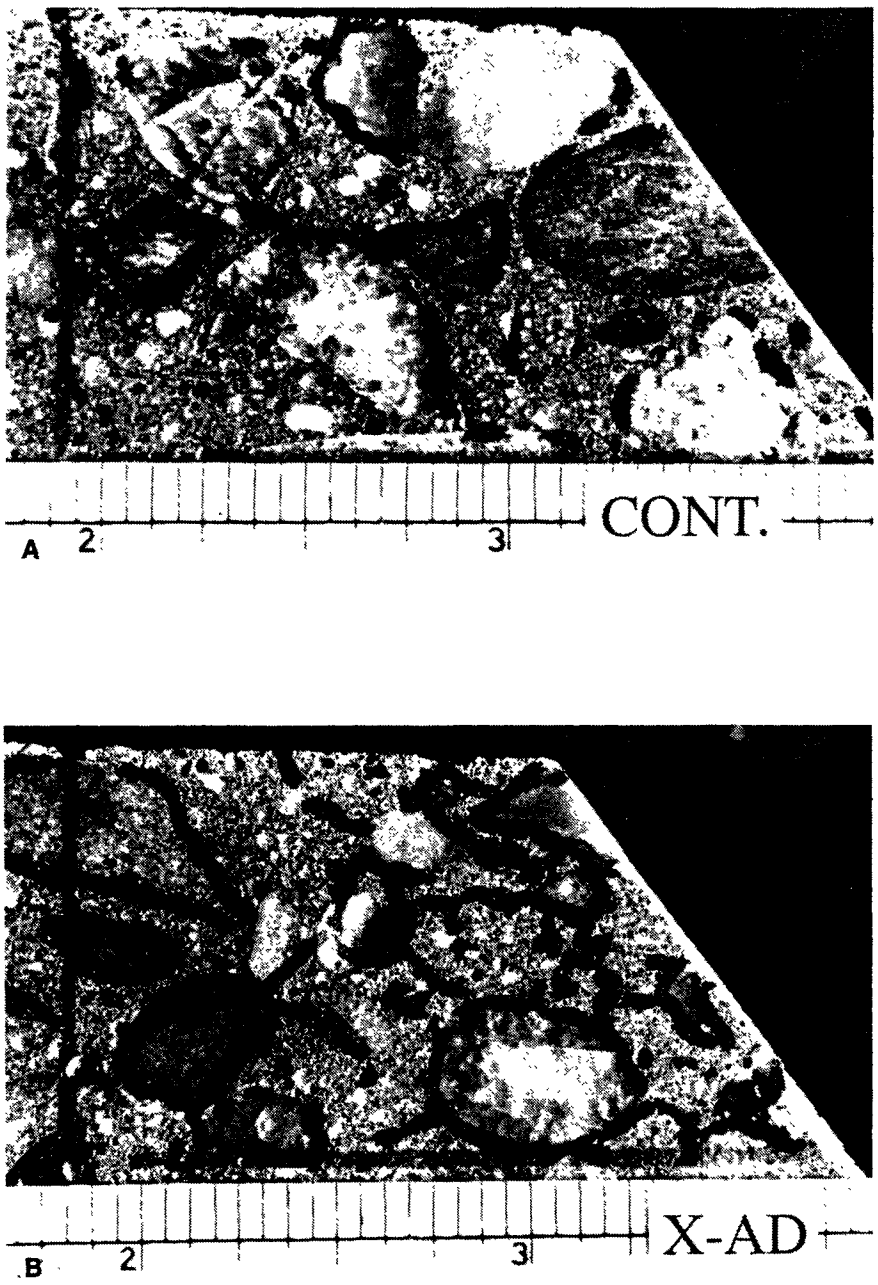


Figure 8-9 FINELY LAPPED SURFACES OF BEAMS TESTED FOR RESISTANCE TO FREEZING AND THAWING. The mixtures were identical except that the mixture shown in B contained an experimental admixture. The portions examined are of a similar size. The scale is in inches.

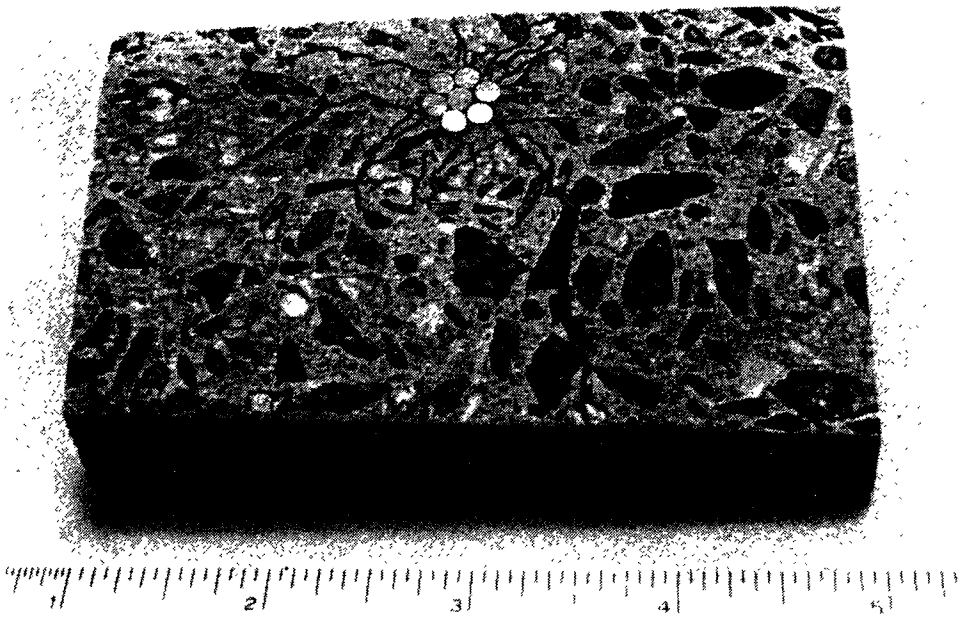


Figure 8-10 LAPPED SURFACE OF SLICE OF CONCRETE CONTAINING REINFORCING CABLE. With the stereomicroscope, the specimen could be seen to contain many fine cracks, but the relationship of all of the cracks to the reinforcing cable could not be noted until the cracks had been marked with ink and the crack pattern examined without magnification. The field of view (the portion of the slice) seen at the magnification necessary for observation of the fine crack system is about the size of one wire of the cable. The scale is in inches.

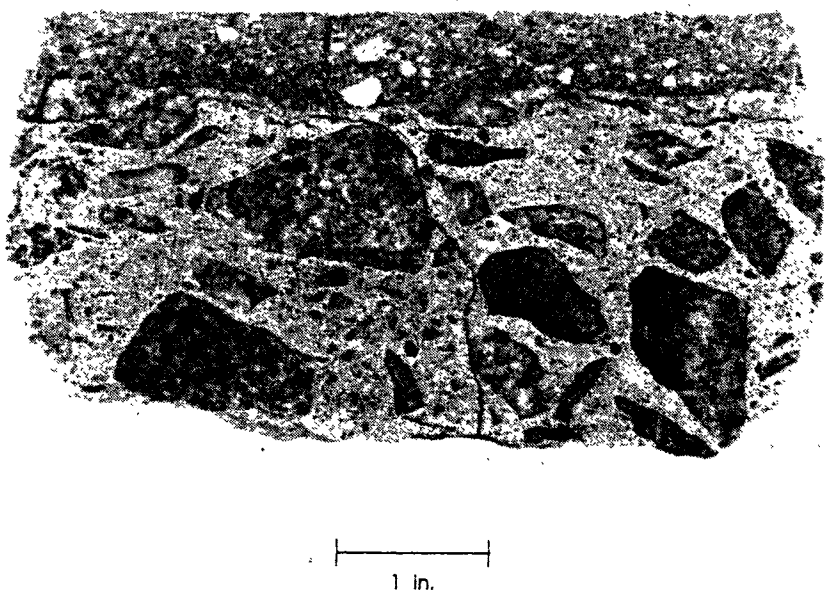


Figure 8-11 CRACKING JUST BELOW BOND IN CONCRETE WITH LATEX CONCRETE OVERLAY. Notice that the vertical crack in the substrate continues through the latex concrete and is expressed at the wearing surface.

8.5 ENHANCEMENT OF MARKED FEATURES

All the notes produced during the examination of the slice should be reviewed, and all the marks that are intended to be visible in photographs should be made permanent and clear enough to be recorded in a photograph. If they are not, they should be enhanced.

8.6 PHOTOGRAPHING OF SLICES AND MAKING OF PHOTOMICROGRAPHS

CAUTION: Most inks begin to fade after they have been in contact with HCC paste for a few days. Therefore, obtain any photographs promptly and make any necessary assessments of the condition of the concrete within a day or two. It is not known whether this fading is more rapid on young or old concrete or if it is due to ionic movement of the ink (sinking into and being distributed in dilute condition throughout the HCC) or by a chemical reaction.

Each exposure should be recorded in a notebook or file expressly designed for such data. Data that will aid in improving future photographs should be included: (1) record the illumination and the adjustments on the light meter or shutter control, and (2) record the ground cloth and background when appropriate. A system of identifying all negatives should be devised and this identification recorded with the negatives, on any archive prints, and in all notes concerning the photographs or specimens. Figure 8-12 is a sample of a sheet used in the VTRC stereomicroscopy photograph notebook.

8.6.1 Photographs of Marked Slice

The photographs may include the entire slice or small areas of the slice may be recorded as enlarged by a camera lens or enlarger. Such photographs should be included in any important final report; they will be invaluable in any legal controversy. A permanent record should be made in the file of roll and frame numbers or photographic file numbers.

8.6.2 Photomicrographs

Important features that are visible only with magnification should be recorded in photomicrographs so they can be easily discussed with the client. A record of their appearance should be included in the data file on these specimens. Such pictures can be taken with a camera on the upright port of the stereomicroscope (see Fig. 2-16).

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PHOTOMICROGRAPHIC DATA SHEET #_____

DATE_____/_____/_____ ROLL#_____FR#_____
DAY MONTH YEAR

FILM_____ASA_____INCR?_____

CAM. OC.

ZOOM

OBJ.

SPECIMEN IDENTIFICATION:

SKETCH

SET: ASA_____ASA_____CTR . .T . . . .D . .

CHECK: 0 | | | | | |

CT METER— 0 R B

UP-PATH W G Y LO-PATH W G R out

RELEASE EST. EX._____TIME OFF, USED ON PRESET.

REMARKS:

Figure 8-12 SHEET USED IN VTRC STEREOMICROSCOPY PHOTOGRAPH NOTEBOOK

Chapter 9
WATER-CEMENT RATIO

9.1 OVERVIEW

The water-cement ratio is an exceedingly important parameter of the quality of HCC. It is the main control of the compressive strength, abrasion resistance, and permeability. The water-cement ratio can be estimated by petrographic means, or the cement content can be determined by chemical analysis and compared with the cement content specified. The petrographer may be requested to estimate the water-cement ratio whenever concrete does not meet compressive strength specifications. Whether requested or not, an estimate of this ratio should be a regularly scheduled portion of any general examination of HCC.

9.2 PROCEDURES

9.2.1 Estimation

The water-cement ratio can be estimated by following a five-step procedure, as listed in Table 9-1.

1. Consider the air-void parameters. Determine if there is any indication of an excess amount of air voids or excess size of air voids (see Figs. 6-2 and 6-3). Usually, the air-void system is the first-noticed indication of a high water-cement ratio. The changes in the air-void parameters are because (1) more air is generated and stabilized in mixtures where there is more water, and (2) air-entraining admixtures are diluted (the surface tension inside the air voids is less effective). The dilution causes irregular voids and excessively large voids to be more prevalent. In concrete with a low-to-medium water-cement ratio, the air-void system appears normal and the specific surface is high. There are few (usually less than 1.5% by volume) large and irregular voids.

2. Consider the quantity of the paste. In HCCs with a high water-cement ratio that has occurred because excess water was added to the mixture, the paste content appears high. That is, the aggregate content may appear low. This is due to the fact that water is a component of the paste and excess water expands the paste volume relative to the aggregate volume (see Fig. 7-2A).

3. Consider the appearance of the paste. Study a finely lapped slice of the specimen with the stereomicroscope at 100X. Compare the appearance of the finely

Table 9-1
PROCEDURE—ESTIMATION OF WATER-CEMENT RATIO

1. Consider the air-void parameters.
2. Consider the quantity of the paste.
3. Consider the appearance of the paste.
4. Study the reaction to needles and picks.
5. Estimate the absorption and permeability.

lapped surface with that of specimens in the reference collection that have a known water-cement ratio.

Examine the tiny dark particles that are the remnants of the ferrite (iron-bearing interstitial) portions of the cement. Decide if these particles are more or less prevalent in the specimen under study than in the specimens in the reference collection of normal HCC fabricated with the same type of cement. If the cement type is not known, study the concretes of all the cement types in the collection.

Study the texture of the paste, and compare it with that of specimens in the reference collection. If the paste has a very smooth uniform surface resembling a plastic or ceramic, the water-cement ratio is low. Careful observation of the texture of the paste at 80X to 100X magnification will indicate that the matrix of HCCs with a high water-cement ratio is different; it is almost sugary. That is, it appears to be composed of a mass of individual particles, as is a sugar cube. This is why it fragments so readily. In extreme cases, these particles may seem to be equant individuals just barely stuck together. When the water-cement ratio is only moderately high, this feature is beyond the resolving power of the stereomicroscope but an experienced observer will be able to see that the paste texture is more open than desired and there is not a compact tight structure as in HCC with a low water-cement ratio.

HCC paste with a high water-cement ratio looks and is more fragile than paste in normal HCC. It is typically lighter in color than would normally be expected.

The average HCC with a medium-to-low water-cement ratio has a dense, solid appearance. In HCCs with a very low water-cement ratio or when certain particulate admixtures are used, the paste appears very smooth and dense, almost like a plastic.

4. Study the reaction to needles and picks. Scratch and pick at the paste while observing the reactions of the paste with the stereomicroscope. If the water-cement ratio is high, edges will seem to come off by fragmentation rather than by the cracking or bending that is common in HCCs with a low water-cement ratio.

5. Estimate the absorption and permeability. Place a drop of water on the finely lapped (oil free) slice, and observe the drop. If the water beads up from surface tension (looks as if it could roll around) before it sinks in, the water-cement ratio is normal or low. If the drop spreads out with a feathery edge and sinks into the concrete quickly, the water-cement ratio is high. The more slowly the water drop disappears into the concrete, the lower the water-cement ratio and the permeability.

9.2.2 Chemical Determination

If the compressive strength of the concrete is low, the chloride ion permeability is high, the microstructure of the paste appears to be sugary, an aggregate with a known high water demand was used, or more quantitative data are required, a chemically determined cement content of the hardened concrete may be indicated. The costs of this analysis and the arrangements with the chemist or testing laboratory are usually the responsibility of the client. The methods used over the years for this chemical determination are discussed by Hime (1978) and in ASTM C 1084. Other modern methods are discussed by Clemena (1972) and Pistilli (1976). If the amount of water used is known (rare in field concrete), the water-cement ratio can be calculated from this determination.

The proper chemical determination of the cement content requires the use of a method appropriate for the type of aggregate present. A chemical determination of the cement content gives a result that is an average of the cement content of the specimen and provides no information on the extreme conditions that may exist in local zones in the specimen and whose extent and continuity may be critical to the strength and durability of the subject concrete. The selection of the particular portion of a specimen for analysis will affect the results. The chemical method cannot distinguish between cement that has been tied up in only partially hydrated rims and balls and cement that has dispersed and hydrated and thus contributes to the strength of the HCC. If the portion selected has a large proportion of knots of cement or cement rims on aggregates or both, the results will indicate sufficient cement content. If the portion selected is a light-colored portion containing excess air voids, less than a normal amount of unhydrated cement, and paste with a sugary texture, the results will indicate a low cement content. The petrographer must use good judgment and a sense of justice in selecting the portion of the specimen for chemical analysis. There may be no representative portion of the specimen that is of sufficient size for chemical analysis. In such a case, the petrographer might inform the client of the data already obtained and recommend procuring additional specimens.

The result of the cement analysis is reported (usually to the client) in pounds per cubic yard. If the reported amount of cement is significantly less than the amount of cement intended to have been used in the mixture, the water-cement ratio is high and either the volume of the concrete increased (usually because of excess water) or less than the prescribed amount of cement was used. Because the analysis is not performed on specimens of HCC suspected of having a normal or low water-cement ratio, we have never had to report a case where the cement content indicated that a significant excess of cement was added or that a significant amount of water was omitted.

Chapter 10
ALKALI-AGGREGATE REACTIONS

10.1 OVERVIEW

The concrete petrographer is often called upon to explain the causes of deterioration of an HCC placement. The determination of the severity and extent of the distress must include an examination of the placement. The laboratory work of the petrographer includes identification of the aggregates involved in the reaction, examination of cracks and crack surfaces, identification and study of any reaction products, and performance of various tests of the nature of the aggregates and their reactions.

Concrete is fabricated by placing aggregate consisting of gravel, sand, or rock fragments or other mineral material in a very active chemical environment. The fluid of fresh cement paste is a saturated solution of calcium hydroxide. This solution is very alkaline; a pH of 13.5 is not uncommon. These alkaline solutions are so strong that they will chemically burn skin, etch glass, cause dissolution of many siliceous rocks, and cause chemical and volume changes in certain carbonate rocks. They are much stronger than any alkalies that are to be found in any of the solutions to which rock might be subjected during the natural weathering and degradation cycles in normal geological environments.

Because of the different chemical reactivities of the ions and their different sizes, the alkalies (in cement) are reported as the weight percentage of the sodium oxide (Na_2O) plus 0.658% of the weight percentage of the potassium oxide (K_2O). At the present time, the alkali content of most cements in Virginia is reported to range from about 0.55% to 0.70%. In eastern Canada (where deleterious expansive alkali-silica reactions are a severe problem), the alkali content is above 1.5% for cement in many of their cement manufacturing plants. The lower the alkali content, the less severe the alkali-aggregate reaction will be. Cement is not always the only source of the alkalies involved in the deleterious reactions. Deleterious alkalies may come from ground water, sea water, deicing chemicals, fertilizers, or other sources.

All ordinary hardened cement paste contains water of crystallization, crystals of calcium hydroxide, and, depending on the rainfall, humidity, and water table level, a certain amount of interstitial water. In all hardened concrete, there is an interstitial saturated solution of calcium and other hydroxides.

The role of aggregates in concrete was originally perceived to be entirely mechanical. Aggregate particles were thought to be unaffected by the concrete paste and were selected on the basis of their physical properties, such as shape, density ("specific gravity"), surface texture, and amount and interconnection of internal surfaces. However, there are chemical reactions that take place between the aggregates and the paste. Within a concrete mass that contains aggregate particles with a variety of lithologies, there are differences in the interfacial zones that are dependent on differences in the chemical and microstructural nature of the aggregate. The differences in the reactions at the aggregate-paste bond are most easily detected and identified by examination of thin sections of the concrete with the petrographic or P/EF microscope. Such an examination will show that the microstructure and

chemical constituents of the bond area vary with the lithology of the aggregate particles. It can be shown that, on occasion, this zone of chemical reaction can increase the strength of the concrete. This increase in strength is usually due to the filling of bond-area cracks with cementitious reaction products that have not caused any deleterious expansion.

There are two major types of deleterious alkali-aggregate chemical reactions that can occur in HCC when the aggregate type and chemical constituents of the paste are such that these reactions are possible: the alkali-silica and the alkali-carbonate reactions. Both of these reactions are deleterious when they cause sufficient expansive force within concrete to rupture the concrete despite the restraint of the mass of the placement and the powerful bonding within the cement paste. If the expansive force is so minor that it cannot overcome the restraint imposed by the concrete, then no deterioration occurs (Hilton, 1974; Houston, 1969).

10.2 ALKALI-SILICA REACTION

10.2.1 Overview

The first known of the alkali-aggregate reactions was the alkali-silica reaction. It was first reported by T. E. Stanton in 1940 as a reaction of the alkalies of the cement with aggregates in California that contained opal (Stanton, 1940). In the earlier literature, it was called the alkali-aggregate reaction (Diamond, 1978). This is a reaction between the hydroxide ions associated with the dissolved salts of sodium and potassium and the silica molecules of certain imperfectly crystallized siliceous rocks and minerals, such as opal, chert, and cristobalite, or highly siliceous volcanic glasses or highly strained or granulated siliceous rocks, such as the metaquartzites, other stressed silicates, and exceptionally fine-grained siliceous rocks (such as siltstones and phyllites). The reaction produces a silica gel that will expand in the presence of moisture. The expanding gel causes cracks in the aggregate and paste. The cracks allow more moisture to enter and expansive gel to fill the cracks and cause more expansion.

It is the hydroxide ions associated with the sodium and potassium ions in solution that cause the deleterious reactions. These alkalies are not always present in sufficient amounts to cause a noticeable reaction. When the percentage of Na_2O plus 0.658% of the K_2O is less than 0.60% of the cement, the cement is generally deemed to be too low in alkali content to contribute significant expansion due to the alkali-silica reaction. At the present time, we do not know if this is a sufficiently low limit for the alkali content. According to Bryant Mather, it is not sufficiently low for the conditions in certain western states (B. Mather, personal communication, October 1991).

Low-alkali cement was thought to be generally available in Virginia. Changes in cement production methods in order to use fuel more efficiently and comply with environmental regulations have placed controls on the emission of stack gases into the atmosphere. Therefore, the alkali content of cement has increased (occasionally as high as 1.0%). In any case, we now know that some of the cement obtained years ago was not, continuously, as low in alkalies as it was thought to be. At the present time, we are finding that, in some cement plants, the cement produced in the winter is more apt to be high in alkalies than the cement produced in the warmer months, presumably because of the temperature differentials in the stack.

With the exception of the pure limestones and dolomites, almost all rocks contain some silica. Silica is the most common oxide in the earth's solid crust. The more soluble the form of the silica, the faster and more intense is the reaction. Varieties of naturally occurring silica minerals are listed in order of decreasing reactivity in Table 10-1. This list is very general because, of course, the order is dependent on the degree of disorder to be found within the particular variety and mode of occurrence of the species.

Table 10-2 shows other rocks listed in order of decreasing reactivity. This list is also very general because the order in the list is dependent on the degree of disorder to be found in the particular variety and amount of the deleterious component present.

The expansion in these reacting concretes is caused by the most poorly crystallized portion of the silica-bearing reactive material dissolving in the highly alkaline sodium/potassium-alkali pore solution of the paste and forming an expansive gel within the siliceous particle. With expansion, the gel migrates out of the particle and can permeate the paste and collect in entrapped voids, pores, and microcracks or both. The gel formed at the aggregate surface and that formed before hardening (usually distributed interstitially throughout the paste) are high in lime. These

Table 10-1
SILICA MINERALS IN ORDER OF DECREASING REACTIVITY

- Amorphous silica: sedimentary or volcanic glass (a volcanic glass that is devitrified and/or mostly recrystallized may still be reactive)
- Opal
- Unstable crystalline silica (tridymite and cristobalite)
- Chert
- Chalcedony
- Other cryptocrystalline forms of silica
- Metamorphically granulated and distorted quartz
- Stressed quartz
- Imperfectly crystallized quartz
- Pure quartz occurring in perfect crystals

Table 10-2
ROCKS IN ORDER OF DECREASING REACTIVITY

- Volcanic glasses, including tuffs (especially highly siliceous ones)
- Metaquartzites (metamorphosed sandstones)
- Highly granulated granite gneisses
- Highly stressed granite gneisses
- Other silica-bearing metamorphic rocks
- Siliceous and micaceous schists and phyllites
- Well-crystallized igneous rock
- Pegmatitic (coarsely crystallized) igneous rock
- Nonsiliceous rock

high-lime gels are thought to be innocuous and unable to expand. Pozzolanic materials cause rapid reactions with the alkalies and cause the alkalies to be contained in the innocuous (nonswelling) gels. Gel masses formed later, especially within the aggregate, are able to absorb water and expand. This gel ruptures the aggregate particles and collects in pockets, cracks, and crevices. The gel in such pockets may expand, open any microcracks, and create new cracks. Sometimes, the entire center of a highly reactive aggregate particle, such as chert, may be converted to an expansive gel that can create, ooze out into, and enlarge microcracks. When the concrete is broken or cut open, many large cracks may be seen radiating from reactive particles. Destructive cracks may also be found radiating from gel-filled pores and voids.

Once the reaction has started in a concrete, it will continue to complete deterioration as long as deleterious alkali ions, reactive aggregate, and water are available. There is no known way to neutralize the reaction without damage to the concrete.

The speed of the reaction depends on the solubility of the silica-bearing minerals involved, type of gel formed, amount of water-soluble alkali present, and availability of water. Good drainage will slow down the reaction. A good air-void system may slow down the expansion. A low water-cement ratio or the low permeability contributed by GGBFS or certain mineral admixtures, such as most pozzolans, will slow the movement of water through the concrete or prevent and thus slow the reaction and the subsequent expansion. Conversely, a high water-cement ratio with high-alkali cement will speed the process (see Appendix F).

10.2.2 Field Examination

The petrographer should go to the placement and inspect the distressed concrete if the client is concerned about the possibility of an expansive alkali-aggregate reaction or the petrographer suspects an alkali-aggregate reaction because the photographs of the placement furnished by the client or the specimens of distressed concrete show evidence of such a reaction (see ACI 201.1R). The evidence may be any of the following:

- cracking that is more open in the upper, drier, more exposed portions of the HCC than it is in the deeper wetter portions
- a crack pattern that seems to indicate that the surface shrunk relative to the underlying concrete (in reality, the underlying concrete has expanded relative to the surface) but the cracking cannot be attributed to plastic shrinkage cracking (see Chapter 4) because a true fracture is seen on the crack surfaces (as contrasted with the "pull apart" structure of the plastic shrinkage crack), the path of the crack goes through the aggregate and paste indiscriminately, or the shape of the crack does not conform to that of plastic shrinkage cracking
- rims on or in the aggregate particles
- open or gel-filled cracks in the aggregate particles
- deposits of gel or carbonated dehydrated gel
- expansion of the placement
- aggregate that is siliceous or a carbonate with a reactive microtexture (in my view, all siliceous rocks should be considered potentially reactive; I have yet to

Table 10-3
FACTORS TO BE CONSIDERED IN FIELD EXAMINATION FOR ALKALI-AGGREGATE REACTIONS

1. Crack pattern
2. Structural evidence of expansion
3. Rocks and minerals in the aggregate of the distressed placement
4. Exudations, coatings, and pore fillings
5. Sufficient sampling of the HCC

see a siliceous rock available in sufficient quantities to be used as an aggregate that has such a perfect crystal structure that I would consider it nonreactive)

Five items should be considered, as listed in Table 10-3: (1) the crack pattern; (2) structural evidence of expansion; (3) rocks and minerals in the aggregate of the distressed placement; (4) exudations, coatings, and pore fillings; and (5) sufficient sampling of the HCC.

10.2.2.1 Crack Pattern

The pattern of cracking indicating expansion of the concrete can be very similar to the cracks generated in masses of mud when the top layer dries and shrinks, plastic shrinkage cracks (see Chapter 4), and the cracks in certain lava flows that occur when the surface cools rapidly and shrinks. Each portion of the surface pulls away from every other portion, generating an irregular honeycomb pattern. The size and regularity of the pattern depend on the cohesiveness, uniformity, and isotropy of the material and the speed of shrinkage (see Fig. 10-1).

This pattern is generated because there is a differential volume change between the exposed surface material and the attached massive substrate. In plastic shrinkage cracking, mud cracking, or cooling lavas, the surface has shrunk relative to the substrate. In expansive alkali-aggregate reactions, the pattern is generated by an increase in volume of the substrate relative to the volume of the overlying surface material. The surface becomes cracked because it is attached to the underlying material. The lower portion of the concrete is damper than the surface portion due to contact with ground water or water vapors rising from the water table and lack of drying by sun and wind. Chemical reactions can take place more readily in the presence of water than in dry zones; therefore, more expansion takes place in the lower portions of the concrete that is reacting by these mechanisms and the surface does not expand. In the early stages of this reaction, the concrete in the lower expanding portions of the placement is squeezed together by the reaction and may not show much cracking. The surface crack pattern has been called pattern cracking, map cracking, Isle-of-Man cracking (see Fig. 10-2), and crows-foot cracking.

In ordinary concrete that is free to expand equally in all directions parallel to the plane of the surface, the classical cracking pattern is usually very evident on the surface in all the stages of the deterioration until the concrete is reduced to a rubble during the last stage. In continuously reinforced HCC or in concrete units that are much longer than they are wide, the concrete is not free to expand equally in all directions. In the early stages of the deterioration, it can expand only at right angles to the length and the cracks will of necessity be at right angles to the direction of the expansion and therefore parallel to the long dimension (and the reinforcing steel when present). In the early stages, longitudinal cracking caused by the

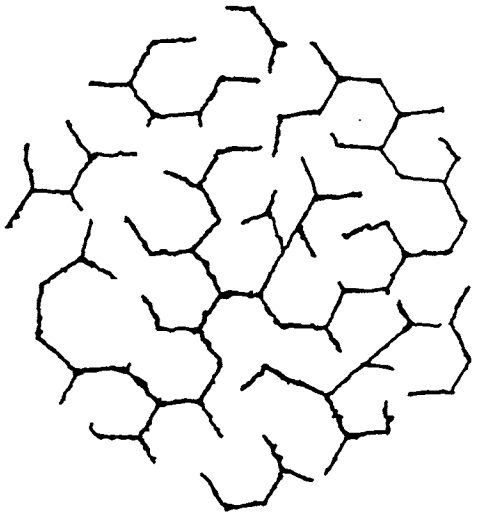


Figure 10-1 IDEALIZED SKETCH OF BROKEN HONEYCOMB PATTERN

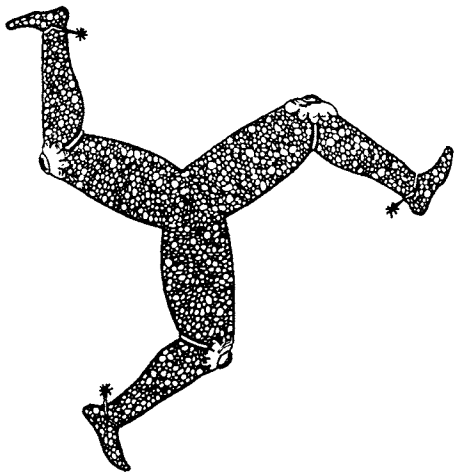


Figure 10-2 SYMBOL FROM COAT OF ARMS OF RULERS OF ISLE OF MAN. This triskelion shows the typical 120° angles of pattern cracking and illustrates why this kind of cracking is sometimes called Isle-of-Man cracking.

alkali-aggregate reactions has often been mistaken for cracking due to expansion of the corrosion products of the reinforcing steel. If a nonreinforced, relatively equant concrete member fabricated with similar materials (such as a bridge approach slab) and placed at the same time is available, it may show the classical pattern cracking long before the longitudinal or reinforced unit exhibits anything other than longitudinal cracking. If a reinforced concrete pavement has deteriorated to the stage of extensive longitudinal cracking, any companion nonreinforced concrete may have deteriorated to a rubble and been replaced long ago. Figures 10-3 and 10-4 show examples of concrete distressed by an alkali-silica reaction.

10.2.2.2 Structural Evidence of Expansion

The expansion of the placement may be very evident. Expansion joints may have closed, with the joint compound having been squeezed out. Guardrail sections that had been planned with a space between may be abutting and grinding together and destroying each other. Occasionally, the expansion may cause blowups, with slabs of the concrete appearing to jump upward because they no longer fit the space. The expansion can cause shearing of bolts and, occasionally, humping of nearby flexible paving on the shoulders (see Figs. 10-5 through 10-7).

If elements of the placement are being forced together or the crack pattern indicates that the lower or damper portions of the concrete may have expanded relative to the upper drier portions or both, the concrete is probably deteriorating by internal expansion.

The expansion is not always clearly shown and may be difficult to understand and document. The expansion is greatest in the areas that have the least optimum combination of factors, that is, the areas that combine the reactive aggregate in sufficient amounts; a sufficient amount of alkali in the cement; and, most variable of all, sufficient amounts of moisture and a sufficiently high permeability to permit the reaction and expansion to occur. Because the reaction cannot occur without moisture, the reaction and expansion are greatest at the depths in the concrete that very seldom become dry. The surface zone of the concrete is the driest portion, does not react as much, and does not expand as much. The surface portion is bonded to the concrete beneath and must move with it. Therefore, it cracks (see Fig. 10-8). An internal RH of 80% is all that is needed for any and all chemical reactions using water to proceed (alkali-silica reaction, cement hydration, etc.). Nearly all concrete will have an internal RH of more than 80% if one side is on the ground and the RH is measured at a depth of 2 in. from the exposed surface—even in the desert (B. Mather, personal communication, October 1991).

10.2.2.3 Aggregate Lithology

By inspection, hardness, and reaction to dilute HCl, a preliminary identification of the major portion of the aggregate as either siliceous or carbonate rock should be made. Certain aggregates may be mixtures of various rock types, and some rocks may be mainly carbonate materials but may contain sufficient highly reactive silica to cause alkali-silica deterioration.

10.2.2.4 Exudations, Coatings, and Pore Fillings

The alkali-silica reaction produces an expansive silica gel that will be visible on the surface of the concrete in most advanced cases of deterioration. The gel may be

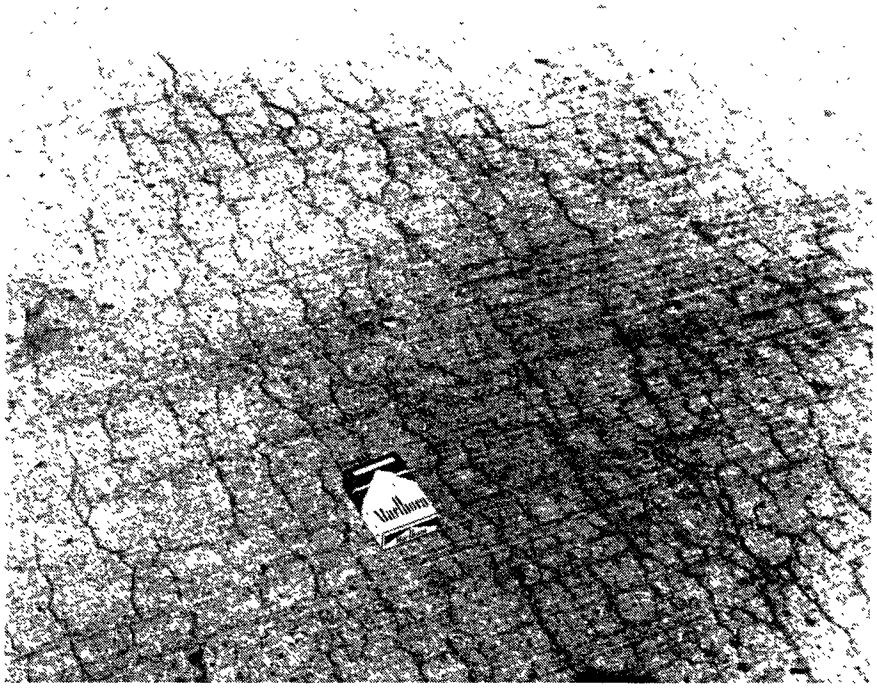


Figure 10-3 TYPICAL DESTRUCTIVE ALKALI-SILICA REACTION IN PAVEMENT

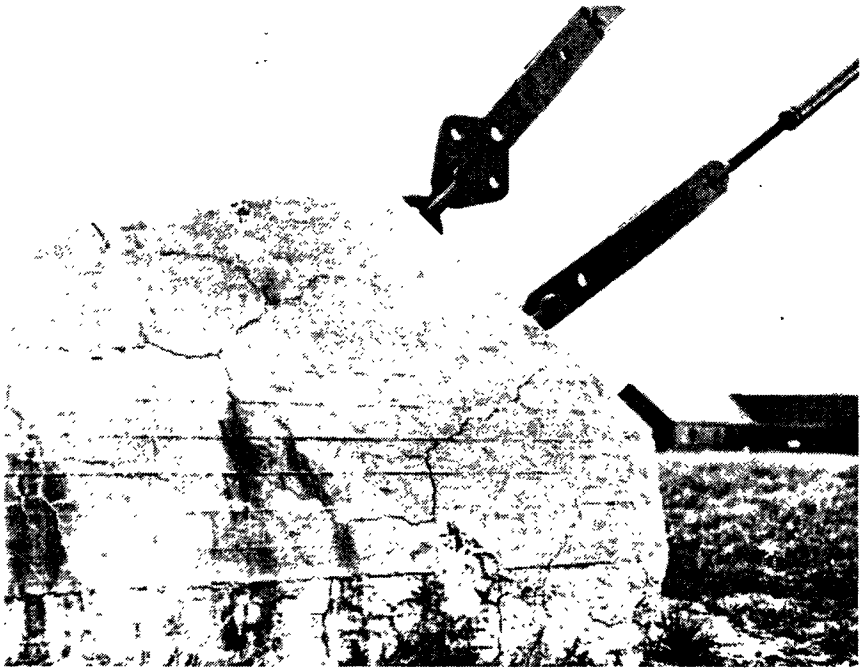


Figure 10-4 DESTRUCTIVE ALKALI-SILICA REACTION IN ANCHOR BLOCK



Figure 10-5 WHEEL GUARD SECTIONS DESTROYING EACH OTHER

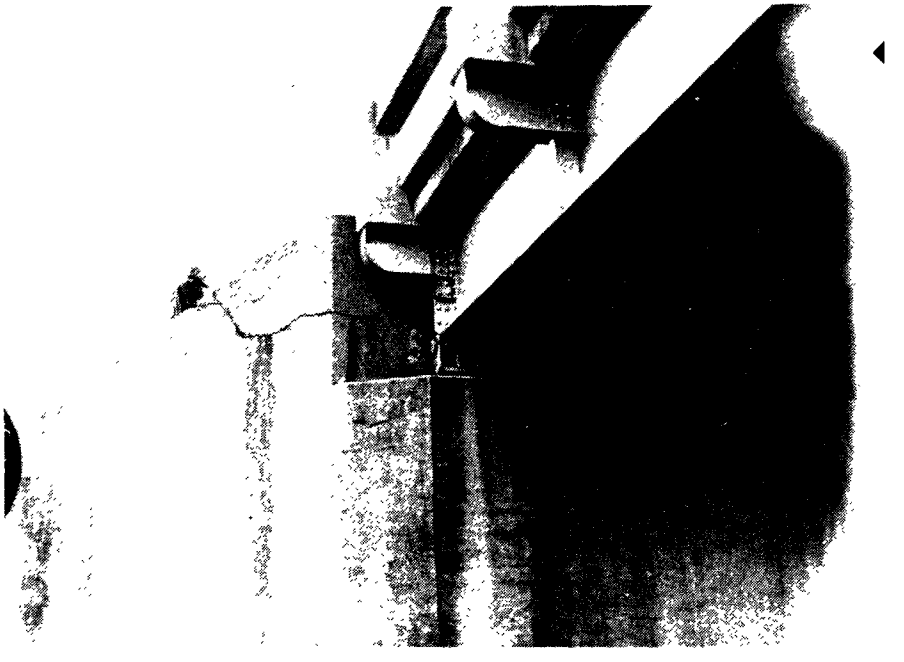


Figure 10-6 UPPER PORTION OF BACK WALL SHEARED BY EXPANSION OF BRIDGE DECK

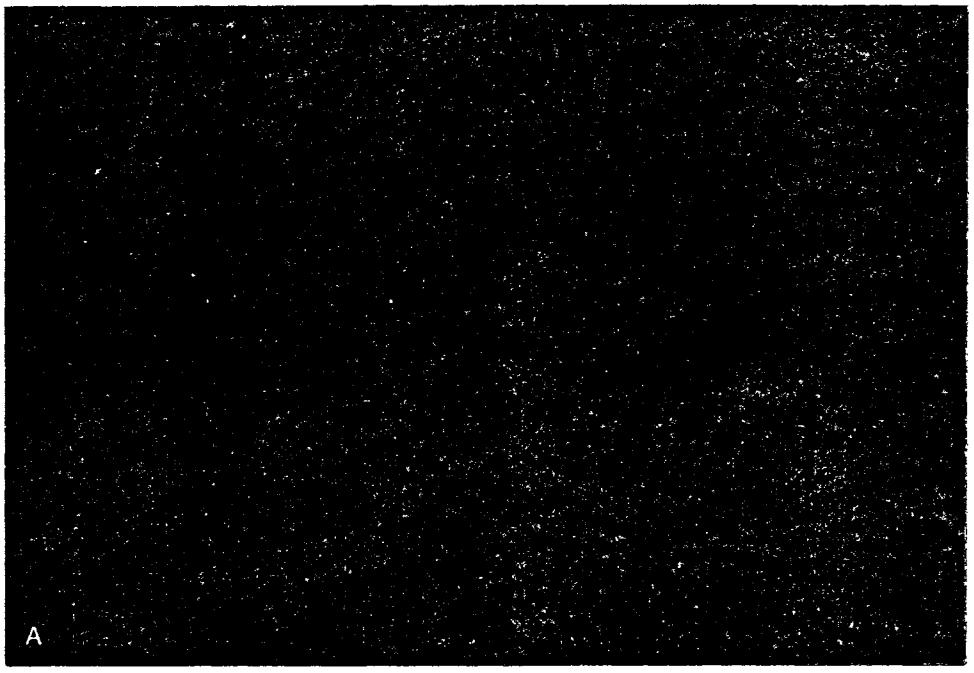




Figure 10-7 DESTRUCTIVE ALKALI-SILICA REACTION IN PAVEMENT. A. Pavement. **B.** Alkali-silica gel in a 2-mm void in a lapped slice from the pavement in **A.** The dark aggregate is siltstone. (Photographs by M. C. Thomson, PennDot.)

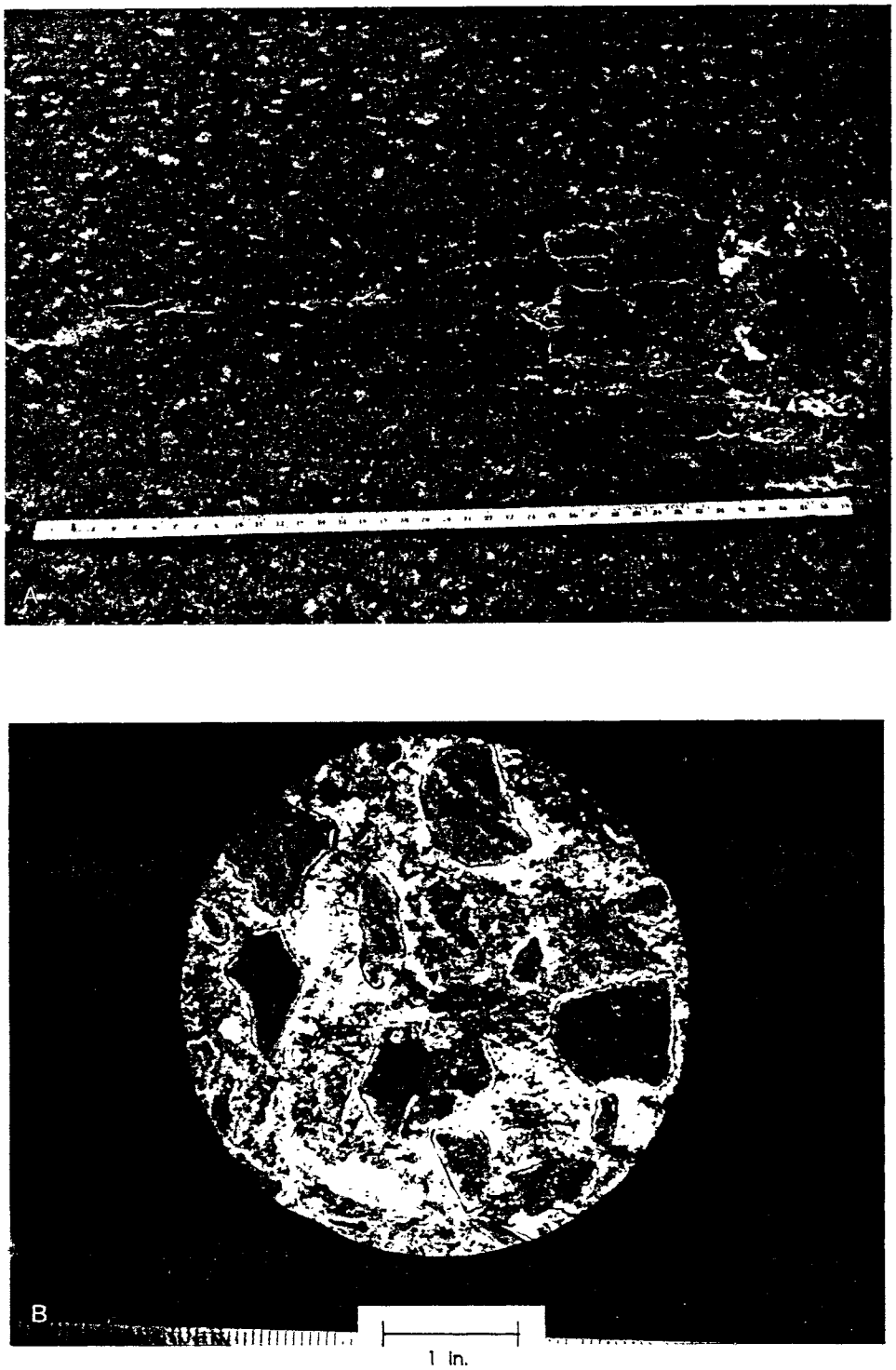


Figure 10-8 ALKALI-SILICA REACTION IN LONGITUDINALLY REINFORCED PAVEMENT CONSTRUCTED WITH DARK METABASALT AGGREGATE. A. Pattern of cracks on surface. B. 4-in. core that fractured on this surface on removal from the pavement. Notice the dried, white, carbonated gel around each piece of aggregate.

translucent and moist or sticky but often becomes dehydrated and carbonated to an opaque white coating or an efflorescence (see Figs. 10-9 and 10-10).

Occasionally, as the gel dehydrates on a pavement surface, it becomes polished to a translucent glaze. In a severely deteriorated bridge, the silica gel and leached-out calcareous reaction products may form stalactites hanging under the bridge. In cases of an alkali-silica reaction with little or no deterioration due to expansion, the silica gels may exist within the concrete as pore fillings or as fillings in cracks produced by structural strains or deterioration due to freezing and thawing.

The exudations may be tested for the presence of silica gel by means of the uranyl-acetate test (Stark, 1990). This test is detailed in 10.2.3. It will require a source of electric power and a light-tight viewing box (portable darkroom) for use in an outdoor environment.

If the deterioration is determined to be due to expansion of the concrete of the placement and there is no evidence of alkali-silica gels (even when the HCC is examined in thin section with the petrographic or P/EF microscope), the distress must be due to a cause other than the alkali-silica reaction. If the aggregate is fine-grained limey dolomite, the alkali-carbonate reaction is likely. Discoveries of new reactions will occur only if the petrographer and the client or engineer consider all the facts with an open mind.

If the alkali-silica gels exist and the major portion of the aggregate is carbonate rock, then a sufficient quantity of chert, opal, or other siliceous substance of low crystallinity must be present within the carbonate rock as vein fillings, siliceous fossils, or other inclusions.

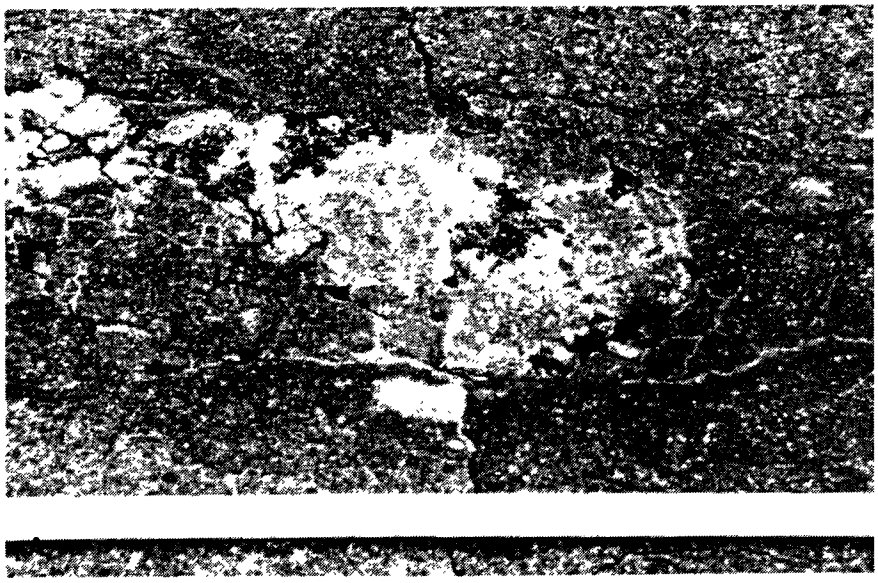
10.2.2.5 Sufficient Sampling

Sampling of HCC in the early stages of alkali-aggregate deterioration should be much more complete than the sampling suggested in Appendix B. The surface evidences of alkali-aggregate reactions are not usually evenly distributed on the placement and are not often indicative of the condition of the HCC beneath the surface. Whereas it is important to obtain specimens of the badly deteriorated areas, it is equally important to study specimens of concrete from areas that have no surface deterioration so that an assessment of the extent of the underlying expansion can be made. Full-depth cores should be taken of several areas showing surface distress, of apparently undamaged HCC halfway between such areas, and at regular distances between the halfway cores and the most distressed areas.

10.2.3 Laboratory Examination

The laboratory examination can be conducted in three steps as follows:

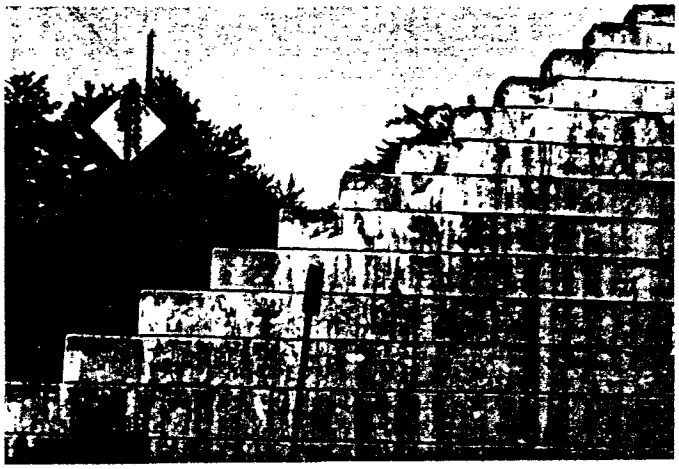
1. Study cut or cored specimen surfaces and crack surfaces produced by the reaction (see Chapter 8). By studying the crack structure and surface of the cracks, provide the client with the reasons this has been determined to be an expansive reaction rather than plastic shrinkage cracking (see Chapter 4). Examine the surfaces for the presence of any reaction products and for the relationship of the cracks in the paste to any deterioration of the aggregate particles. Place particular emphasis on the specimens of the portion of the placement that has expanded. The material of the severely cracked drier surface portion has not expanded so much and may not exhibit so abundantly the reaction products that cause the expansion





B

Figure 10-9 ALKALI-SILICA GEL. **A.** Gel exuding at deteriorating portion of pavement. **B.** Gel on surface of core removed from deteriorating pavement. Notice the 120° cracking.



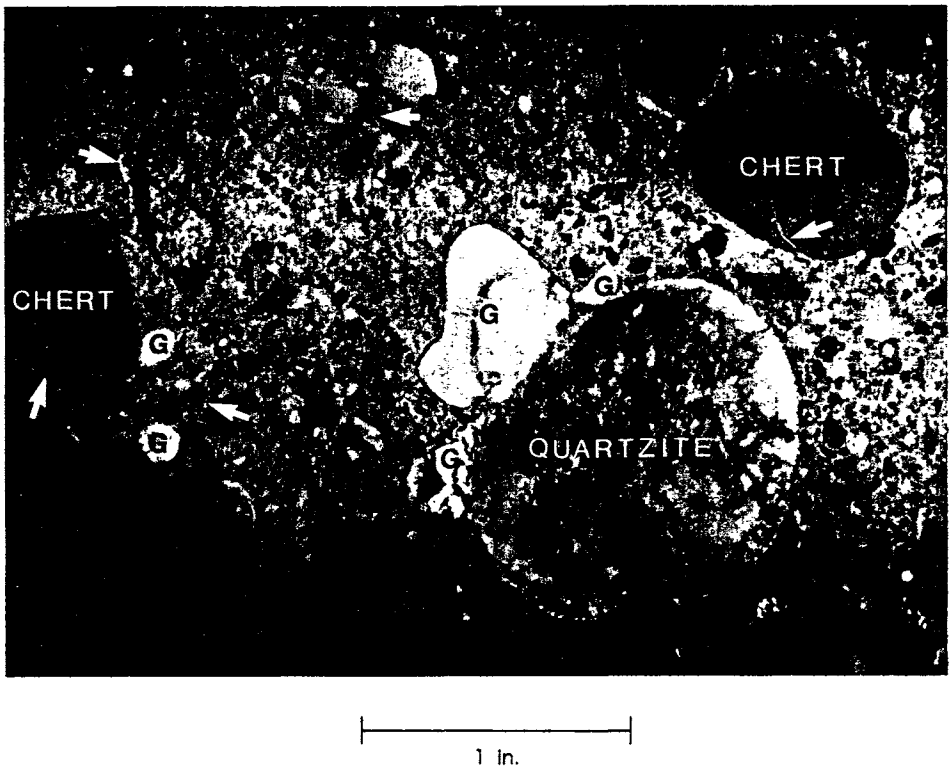


Figure 10-10 ALKALI-SILICA GEL ON BACK WALL. **A**. Gel on deteriorating back wall. **B**. An interior surface showing type of aggregates and reaction products of this reaction. *G* = pocket of alkali-silica gel; *arrows* indicate gel-filled cracks.

and the deterioration. Study the aggregate altered by the alkali-silica reaction. Many aggregate particles may be seriously deteriorated. Such deterioration may be (1) rims on the aggregate that seem to make the exterior of the aggregate less permeable, (2) gel-filled cracks extending out into the paste, (3) hollowed-out centers or centers of sedimentary particles (e.g., sandstone) that have had the original cement between the sand grains removed by the reaction so that the centers are more friable than the rims, and (4) aggregate particles that have been cracked by deposits of gel and dehydrated gel that occur as rims within and around the particles, often on bedding planes or other partings intrinsic to the aggregate. Report the various types of deterioration observed.

When liquid gels exude onto the surface, saturating the paste, filling the cracks, and oozing from the surfaces or dehydrated, carbonated gels can be seen as a white deposit, the alkali-silica reaction is in its most easily identifiable phase (see Figs. 10-8 through 10-10). These white deposits are common as rims around coarse aggregate in the paste and the cracks and shaley partings in the aggregate and as fillings in voids. These deposits are more common with depth. In the near-surface portions of the concrete, the rims may be difficult to see. The rims that occur inside the aggregate are most easily seen on surfaces that have been forced apart by the reaction, that is, in cracks caused by the reaction (see Fig. 10-10). On the surfaces of cores and on sawed and lapped surfaces, there may be no evidence of rims whatsoever and the only hint of an alkali-silica reaction may be scattered white void fillings of dehydrated silica gel.

The interior of the reacted concrete often has large cracks that may be empty because they have been forced apart by expansion elsewhere in the system. The cracks may be filled with alkali-silica gel and other reaction products, such as calcium carbonate and calcium hydroxide.

2. Identify alkali-silica gels in exudations, aggregate rims and cracks, and pore fillings by means of the method using a uranyl-acetate solution and fluorescent light that was well described and illustrated by Stark (1990). The use of this definitive test will prevent any confusion between silica gels and deposits of other secondary products, such as calcium carbonate, that might be leached from the concrete. Silica gel is common in all HCC fabricated with a siliceous aggregate. It is not necessarily deleterious. It must cause expansion to cause distress.

If this test is used, care must be taken to follow all the safety precautions described. A fresh concrete surface free of laitance and carbonation is required. In the laboratory, a fresh surface can be sawed, produced with an electric rotary hammer fitted with a bushing head that looks like a meat tenderizer (see Fig. 10-11), or produced by breaking the specimen with a sledgehammer or in a trimmer (Fig. 2-10). Outdoors on the surface of a placement, the fresh surface can be produced by using a rotary hammer or by sawing or hand hammering off a section of concrete. In the laboratory, the uranyl-acetate–treated specimen is viewed in ultraviolet light in a dark place (closet or darkroom). Outdoors, a light-excluding viewing box (portable darkroom) containing the ultraviolet light is required because the fluorescence of the silica gel that has absorbed the uranium ions is too faint to be seen in daylight.

Any powdered concrete is rinsed from the surface. The uranyl-acetate solution is sprayed on the surface (Fig. 10-12), allowed to react for 5 min, and then rinsed away. The concrete is examined for fluorescent gel with the ultraviolet light (see Fig. 10-13).



Figure10-11 ELECTRIC HAMMER REMOVING CARBONATED SURFACE OF HCC.
A. Operation of hammer. B. Close-up of bushing head for electric hammer.



Figure 10-12 TESTING FOR ALKALI-SILICA GEL. A. Spray bottle for applying rinsing water and uranyl-acetate solution. Notice the gloves used to protect the hands from the uranium-bearing chemical. **B**. Dark viewing box. The ultraviolet light is mounted inside the box. The bottom edge of the box is soft, easily conforms to the concrete surface, and excludes light. The viewing port is rimmed with a soft dark sponge shaped to conform to the observer's face.

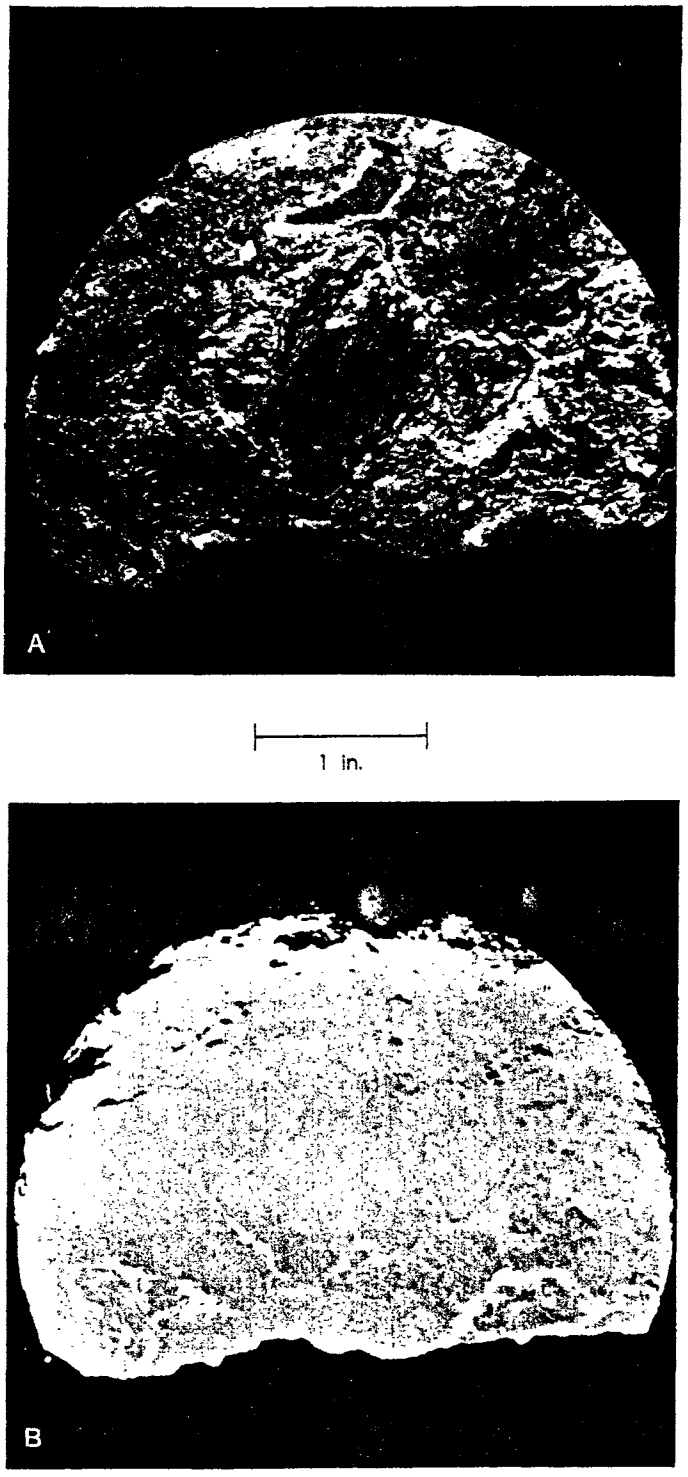


Figure 10-13 SPECIMEN TREATED WITH URANYL-ACETATE FROM PAVEMENT WITH DESTRUCTIVE ALKALI-SILICA REACTION. A. Ordinary light. B. Ultraviolet illumination causes silica get to fluoresce. (Darkroom photograph; exposure 1.5 min.)

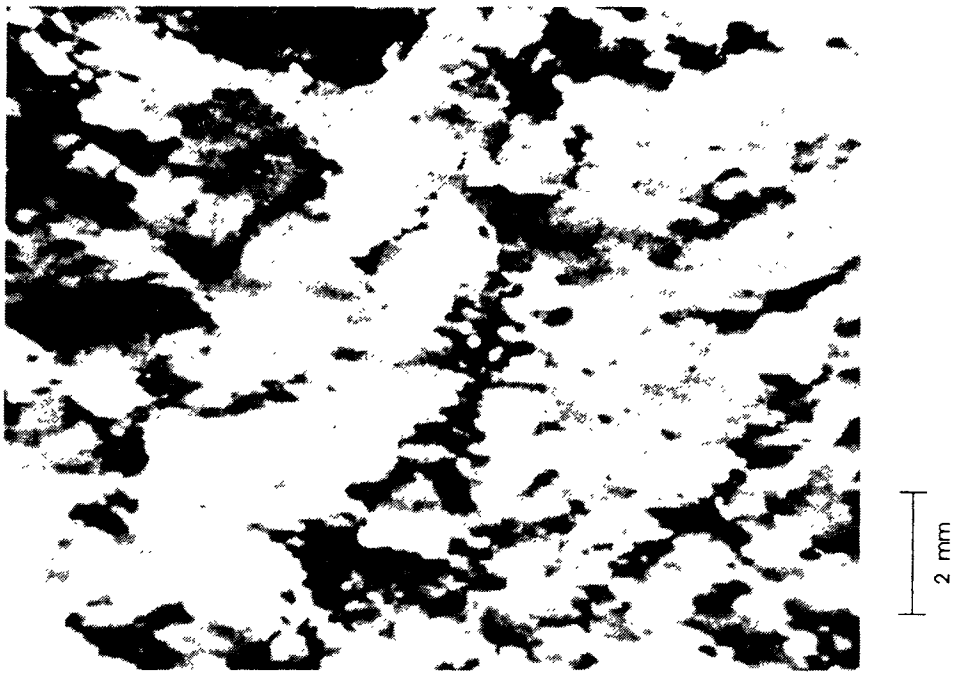


Figure 10-14 THIN SECTION OF HIGHLY STRAINED QUARTZITE. Crossed nicols.

3. Examine a number of finely lapped slices. Select a variety of aggregate particles for study with the petrographic or P/EF microscope, and mark the surface of the specimen, usually a lapped surface, to indicate the location of the desired thin sections. Prepare thin sections (about 15 μm in thickness) of these aggregates and adjacent areas of paste. Identify the constituents, and determine the structure of the reactive components by use of the petrographic or P/EF microscope. Report the structure and mineralogic and lithologic identity of reactive particles to the client.

One of the several kinds of rock that are often subject to this reaction is shown in thin section in Figure 10-14.

10.2.4 Testing of Siliceous Aggregates

Specimens of aggregate are often submitted to the petrographic laboratory to be tested for suitability for use in HCC. Siliceous aggregates may be tested for potential alkali-silica reactivity. Unfortunately, the test results may not be conclusive.

The probability of specific aggregate types taking part in this reaction and causing deterioration in the first few years can generally be ascertained in accordance with ASTM C 227 (mortar-bar method). The ability of the procedures specified in ASTM C 227 to detect the siliceous rocks that react slowly is generally limited by the willingness of the testing technologists to extend the time limits of the test. The time limits prescribed by the test may allow the detection of only aggregates that will show silicate-aggregate reactive symptoms early in the life of the placement, probably within the first year or two. An aggregate that does not cause any deleterious reaction in a placement for the first 6 or 7 years might not expand sufficiently to exhibit its deleterious nature until it has been tested for 3 years or more, if ever.

In recent years, there has been a controversy concerning the proper method of conducting this test. The containers in which the mortar bars are presumably maintained at 100% humidity may or may not have the moisture evenly distributed throughout their height. The method presently requires lining the cans with blotting paper to distribute the humidity; some researchers have stated that the blotting paper causes dripping on the specimens and leaching of the alkalies from the paste (Rogers & Hooton, 1991). If the alkalies are thus leached out, they are not available for reaction in the mortar bars and the expansion is less. Alkali ions have been found in the fluid at the bottom of the containers. For these reasons, this test has been conducted with many different arrangements of mortar bars and blotting paper, but no one way has, over time, remained the accepted standard method.

Regardless of the leaching difficulties, the test specified in ASTM C 227 is the type of test for alkali-silica reactivity favored by many concrete technologists because the test (or modified versions of the test) can be used to predict the possibility of the expansive alkali reactions occurring in specific cement-aggregate combinations. Thus, if the test performs as it should (it does not with some aggregates), an aggregate can be tested with cements of several different alkali contents and decisions can be made concerning the most cost-effective solution to the problem of deciding which materials to use for a proposed placement.

Because of the problems encountered in the leaching of the alkalies out of the specimen and because of the long time involved in obtaining results in the case of the slowly reacting aggregates, certain other test procedures have been devised. The test that is thought to show the most promise is ASTM C-9, Proposal P 214. This test has not been formally accepted by ASTM; it is undergoing extensive experimental use. The results to date, when compared to the recommended criteria, indicate that this test can (with certain aggregates) generate data that seem to be in conflict with field performance and commonly accepted knowledge concerning the alkali-silica reaction (D. S. Lane & H. C. Ozyildirim, personal communication, October 1991). The test is a rapidly reacting test, and even slowly reacting aggregates can demonstrate their deleterious nature in a few months. Because this test requires that the mortar-bar specimens be placed in a 1 normal solution of NaOH during the reactive period, it has the disadvantage that specific cement aggregate combinations cannot be tested. It is a test for only the reactivity of the aggregate.

Placements fabricated with high-alkali cements and aggregates that have shown reactivity under testing do not always show deterioration. The reaction requires that the alkalies go into solution to produce the associated hydroxide ions that can attack the reactive aggregate, the presence of water, and that the concrete is sufficiently permeable to permit the movement of ions.

10.3 ALKALI-CARBONATE REACTION

10.3.1 Overview

The alkali-carbonate reaction was first observed by Swenson (1957) in Kingston, Ontario, Canada, and found by Newlon and Sherwood (1962) to occur in Virginia. This reaction occurs between certain impure dolomitic limestones of a specific petrographic type (Newlon, Sherwood, & Ozol, 1972; Swenson, 1957; Swenson & Gillot, 1960; H. N. Walker, 1978) and the hydroxide ions associated with the dissolved salts of sodium and potassium alkalies that may be found in the cement paste.

The alkali-carbonate reaction is not so well understood as is the alkali-silica reaction. The expansion produced occurs within the aggregate particle. The expansion causes cracks within the aggregate and the paste and therefore deleterious expansion of the mass. The dolomite crystals in the aggregate are chemically altered by the alkali solutions in what is apparently a multistep process. Which step causes the expansion has not been determined.

All known reactive carbonate rocks are from the geologic time period known as the Ordovician period. Fortunately, the particular proportion of minerals and microstructure of the reactive carbonate rock involved are not very common. This rock has been suspected of not being a geologically stable combination of minerals (Steidtmann, 1917). Theoretically, one might say that it is reactive because it is not stable.

The carbonate aggregate that is associated with this reaction is an impure dolomitic limestone. The two major carbonate minerals, calcite and dolomite, are present in nearly equal amounts. Noncarbonate minerals (insoluble in weak HCl), usually of submicroscopic size, make up 10% to 25% of the mass and generally consist of clay, various iron sulfides, and quartz. Small quantities of other minerals may also be present. The aggregate is often (not always) dark gray or nearly black because of the finely divided iron sulfides. The texture in a hand specimen is subconchoidal because the individual crystals are so small and interwoven that their cleavage and parting do not affect the surface.

When the weight percentage of Na_2O plus 0.658% of the weight percentage K_2O is less than 0.45% of the weight of the cement, the cement has been considered to be too low in alkali content to contribute to a significant chemical reaction and concomitant expansion due to the alkali-carbonate reaction (Swenson & Gillott, 1960). At the present time, we do not know if this low limit for the alkali content will prevent the alkali-carbonate reaction in every case.

The alkali-carbonate reaction does not appear to be related to the reaction found in the Midwest that has been termed *D-cracking*. D-cracking has not been found in Virginia, and therefore VTRC has not had any experience with it. Refer to Appendix E for more details on D-cracking.

10.3.2 Field Examination

The field procedures listed in 10.2.2 should be followed. Figures 10-15 through 10-17 are photographs of deterioration caused by the alkali-carbonate reaction.

The petrographer should ascertain if the coarse aggregate is a carbonate rock. If any silica-gel exudations, coatings, or pore fillings are observed, the expansive distress may be partially or completely due to an alkali-silica reaction despite the fact that the aggregate rock is predominantly a carbonate. If exudations, coatings, or pore fillings exist in an alkali reaction with a dolomitic limestone, the dolomitic limestone will be found to contain a large quantity of poorly crystalized siliceous material and should be tested as a siliceous aggregate.



Figure 10-15 DESTRUCTIVE ALKALI-CARBONATE REACTION IN WALKWAY PAVEMENT. In Kingston, Ontario, Canada.

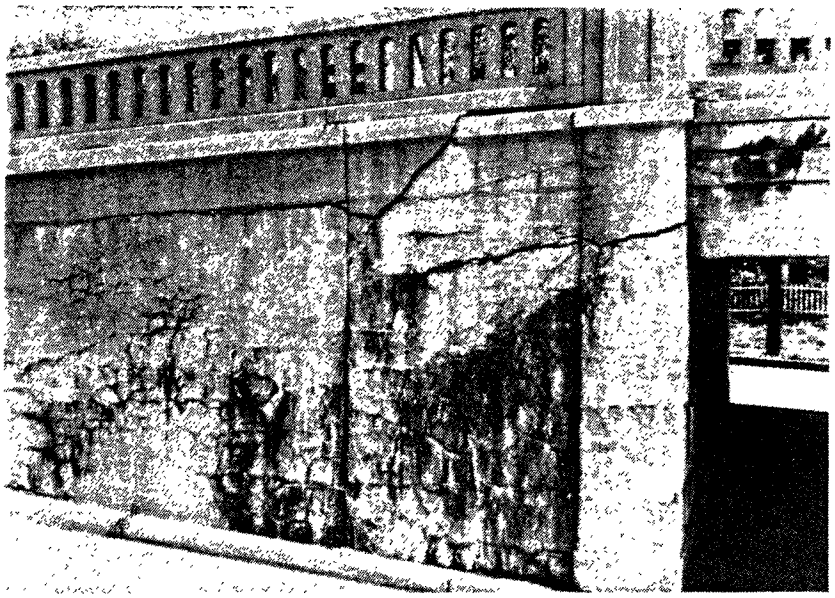


Figure 10-16 DETERIORATION DUE TO A COMBINATION ALKALI-SILICA REACTION AND ALKALI-CARBONATE REACTION. In New Jersey.

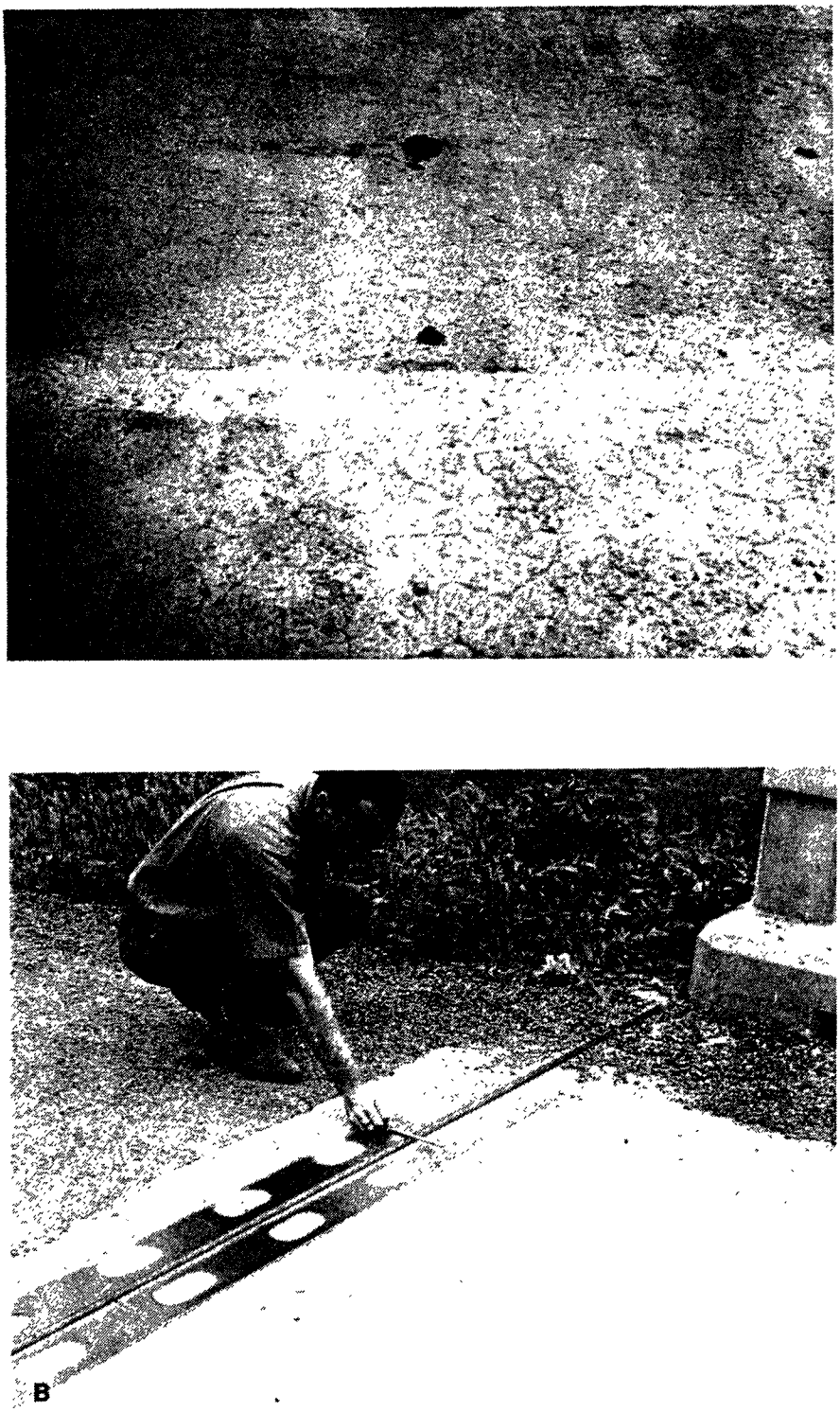


Figure 10-17 DESTRUCTIVE ALKALI-CARBONATE REACTION IN BRIDGE DECK. Near Harrisonburg, Virginia. **A.** Expansive cracking of bridge deck. **B.** Complete closure of joint.

10.3.3 Laboratory Examination

The laboratory examination can be conducted in three steps, as follows:

1. Study cut or cored specimen surfaces and crack surfaces produced by the reaction (see Chapter 8). By studying the crack structure and surface of the cracks, provide the client with the reasons this has been determined to be an expansive reaction rather than plastic shrinkage cracking (see Chapter 4). Examine the surfaces for the presence of any reaction products and the relationship of the cracks in the paste to any deterioration of the aggregate particles. If any silica-gel deposits are found, the expansion may be due to the alkali-silica reaction and the procedures and tests used should include those prescribed for siliceous aggregates. Place particular emphasis on the specimens of the portion of the placement that has expanded. The severely cracked surface portion has not expanded as much and may not exhibit any deterioration of the aggregate. The deeper portions will have more deteriorated aggregate even though it is not necessarily cracked as much. The signs indicating alkali-carbonate reactivity can be very subtle because the reaction does not cause the growth or exudation of any characteristic reaction products, such as the gel associated with alkali-silica reactions.

Very fine cracks in the aggregate are often found in the portion of the placement affected by the expansion of the alkali-carbonate reaction. These cracks may extend into the paste. They may be randomly oriented but may sometimes occur just under the surface of the aggregate as if a thin layer of the aggregate was about to pop off (see Fig. 10-18). Cross sections of elongated or flat particles affected by the reaction may show one or more lengthwise fine cracks nearly centered in the particle. It

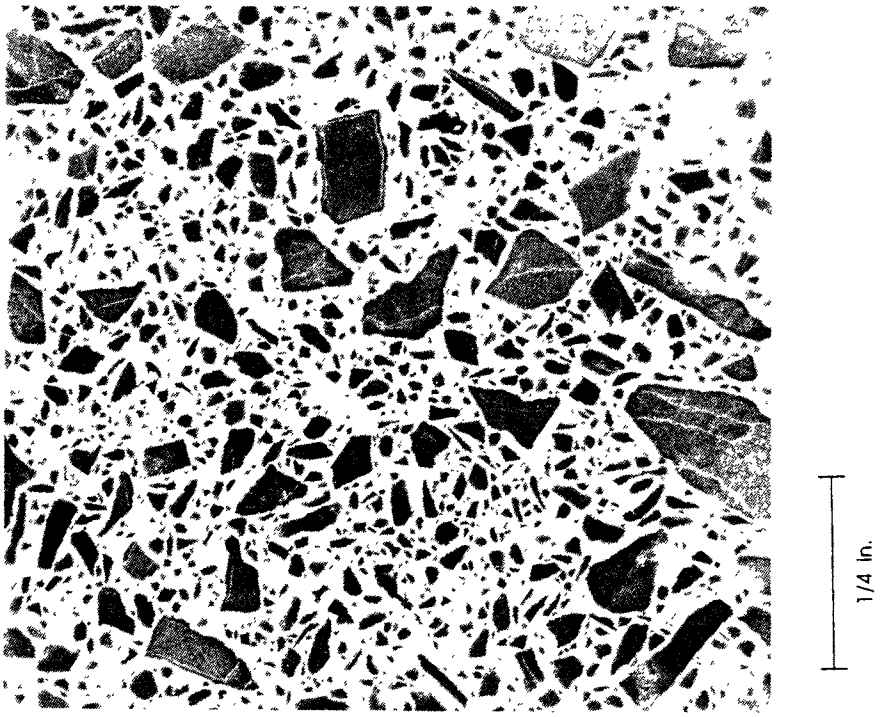


Figure 10-18 FINE CRACKS IN REACTIVE CARBONATE AGGREGATE IN MORTAR BAR WITH HIGH-ALKALI CEMENT. Although the expansion of the mortar bar was negligible, these cracks are typical of those that may be found in specimens that reacted in service.

may be impossible to find any signs of deterioration in the aggregate particles in the less reacted, drier, but more heavily cracked portion of the placement.

2. Examine several finely lapped slices. Study the slices with the stereomicroscope, and select several paste areas and numerous aggregate particles for the production of thin sections and detailed study with the petrographic microscope. Prepare thin sections of the aggregate as detailed in 5.3.2.

The most easily detected identifying characteristic of the alkali-reactive carbonate rocks is the microtexture of the aggregate. Invariably, this texture is that of small rhombic crystals of dolomite suspended in a calcite, micrite matrix that contains the fine, particulate, insoluble constituents. This texture may occur evenly throughout the aggregate or may be confined to certain seams and crack fillings. All occurrences of carbonate rock that have been found to be reactive have portions in which this particular texture exists. However, not all dolomitic limestones with this texture expand with sufficient force to cause distress.

If the dolomite rhombs are large enough, they may in some cases be observed with a hand lens or stereomicroscope on a lightly etched, finely lapped surface of the aggregate. This surface may be fabricated on a large fragment (e.g., 3 by 4 in.) of the rock or may be a lapped surface produced on a slice of the HCC.

3. Examine thin sections with the petrographic microscope. The dolomite crystals in these reactive rocks are often no more than 10 μm in any direction, and observation of them often requires that the thin section be, at least in part, much thinner than an ordinary thin section for ordinary geologic procedures (see 5.3.2). The texture of the micrite itself is often hidden by the dark murky pall caused by the finely divided insoluble constituents. When the individual grains of the micrite can be discerned, they can be seen to be completely anhedral and tightly locked together, sometimes interlocked in the fashion of the curved seams of the leather cover on a baseball. Sometimes, the dark, greasy looking murk collects into thin, darker brown or black stringers that wind along the contacts of the other constituents. Figure 10-19 shows examples of the microtexture of alkali-reactive carbonate rocks. Figure 10-20 shows similar sections of nonreactive carbonate rocks.

When the alkali-expansive carbonate rocks are used with high-alkali cement, thin sections of the reacted material show definite changes in the microstructure of the aggregate. Many of the euhedral dolomite crystals have been altered. Sometimes, the dolomite has been altered to calcite. Sometimes, the dolomite crystals are partially or completely gone and only a hole the shape of the crystal remains (see Fig. 10-21).

Reactive carbonate rock has been tested in other chemical solutions that occur in HCC (Walker, 1979b). The most startling results were obtained with a solution of sodium and calcium carbonates (see Fig. 10-22).

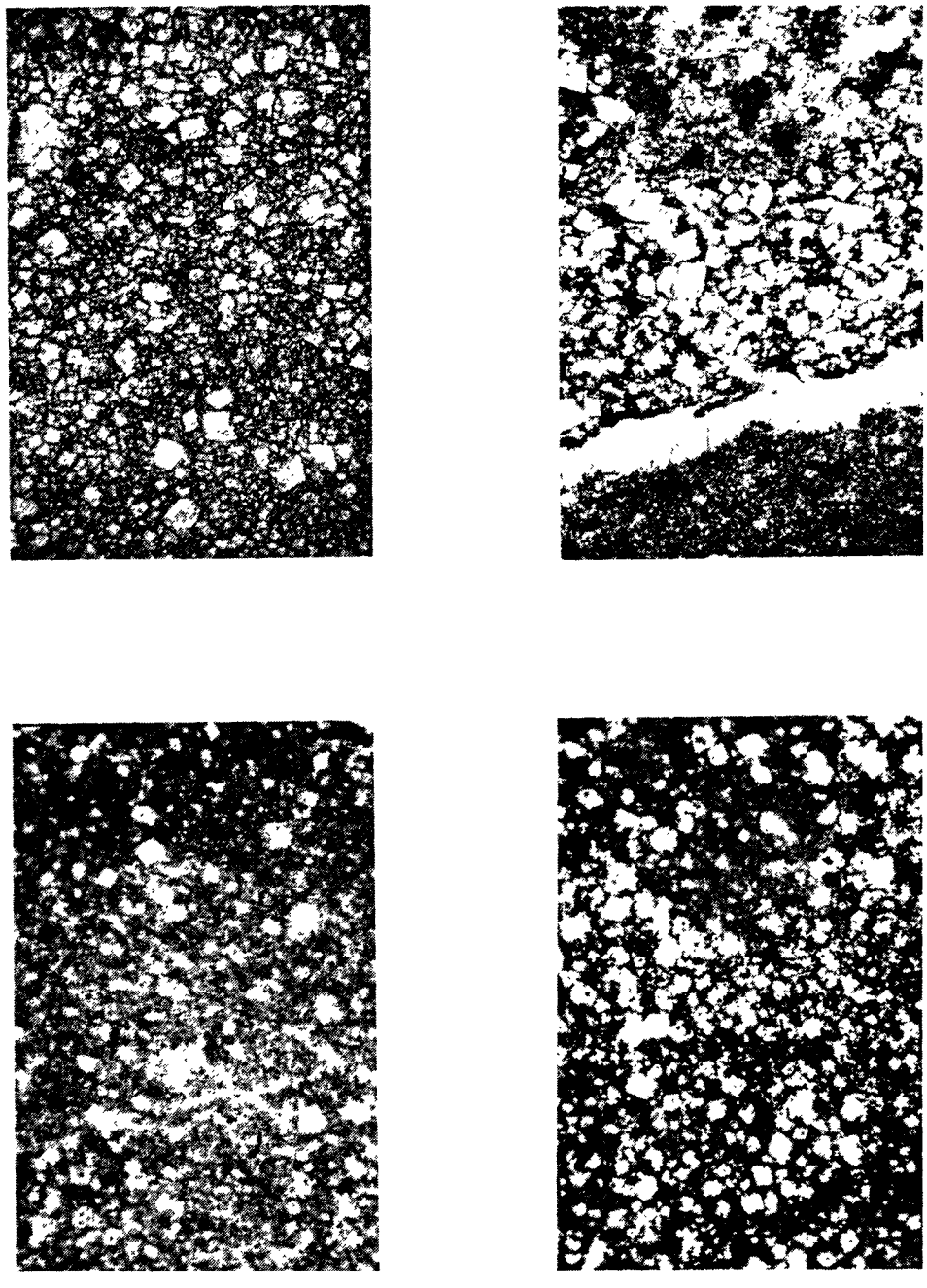


Figure 10-19 ALKALI-REACTIVE MICROTEXTURE IN FOUR CARBONATE ROCKS

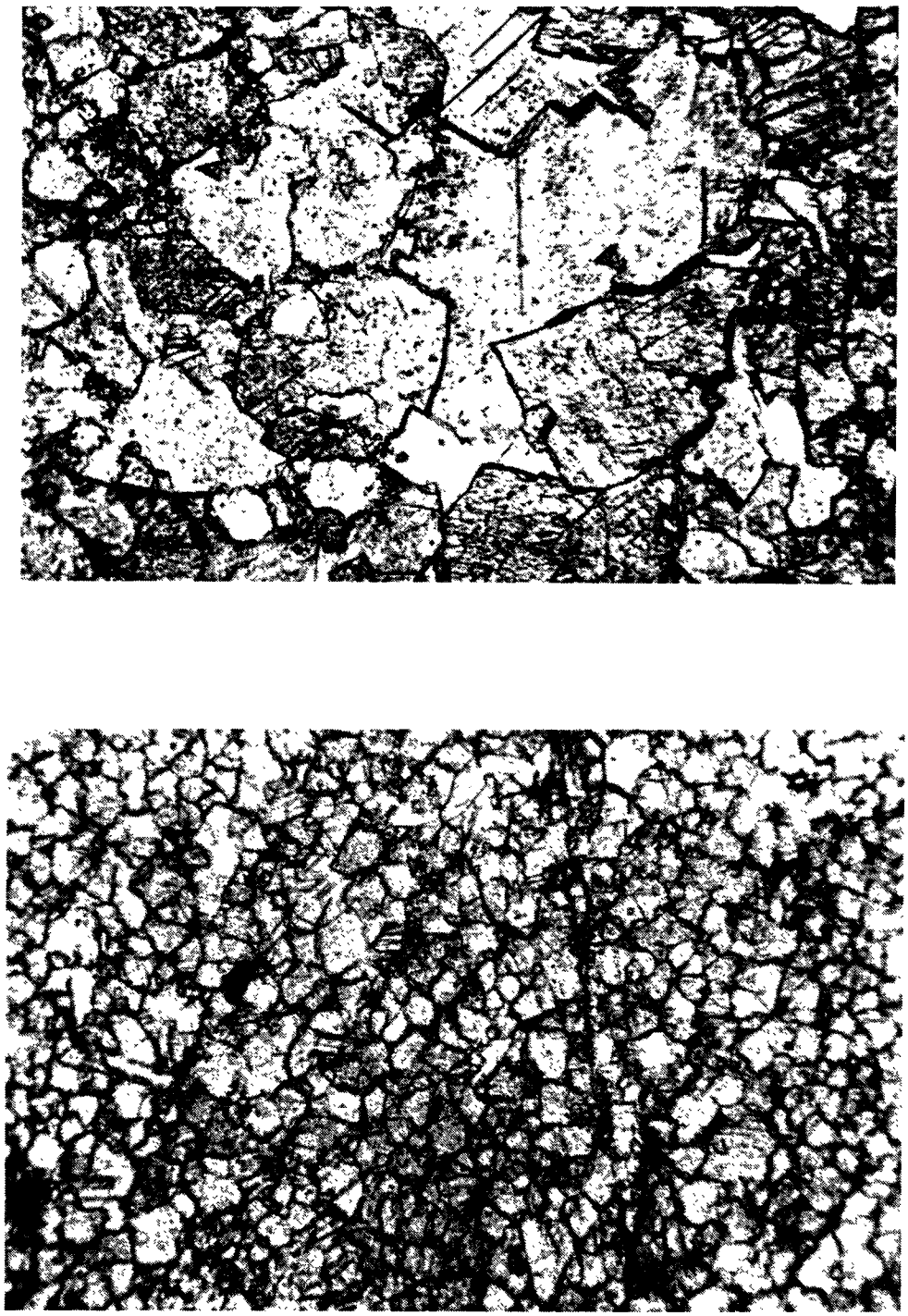


Figure 10-20 NONREACTIVE MICROTEXTURES OF CARBONATE ROCKS. Examples are shown to illustrate the difference between these crystalline textures and the partially crystalline reactive textures shown in Figure 10-19.

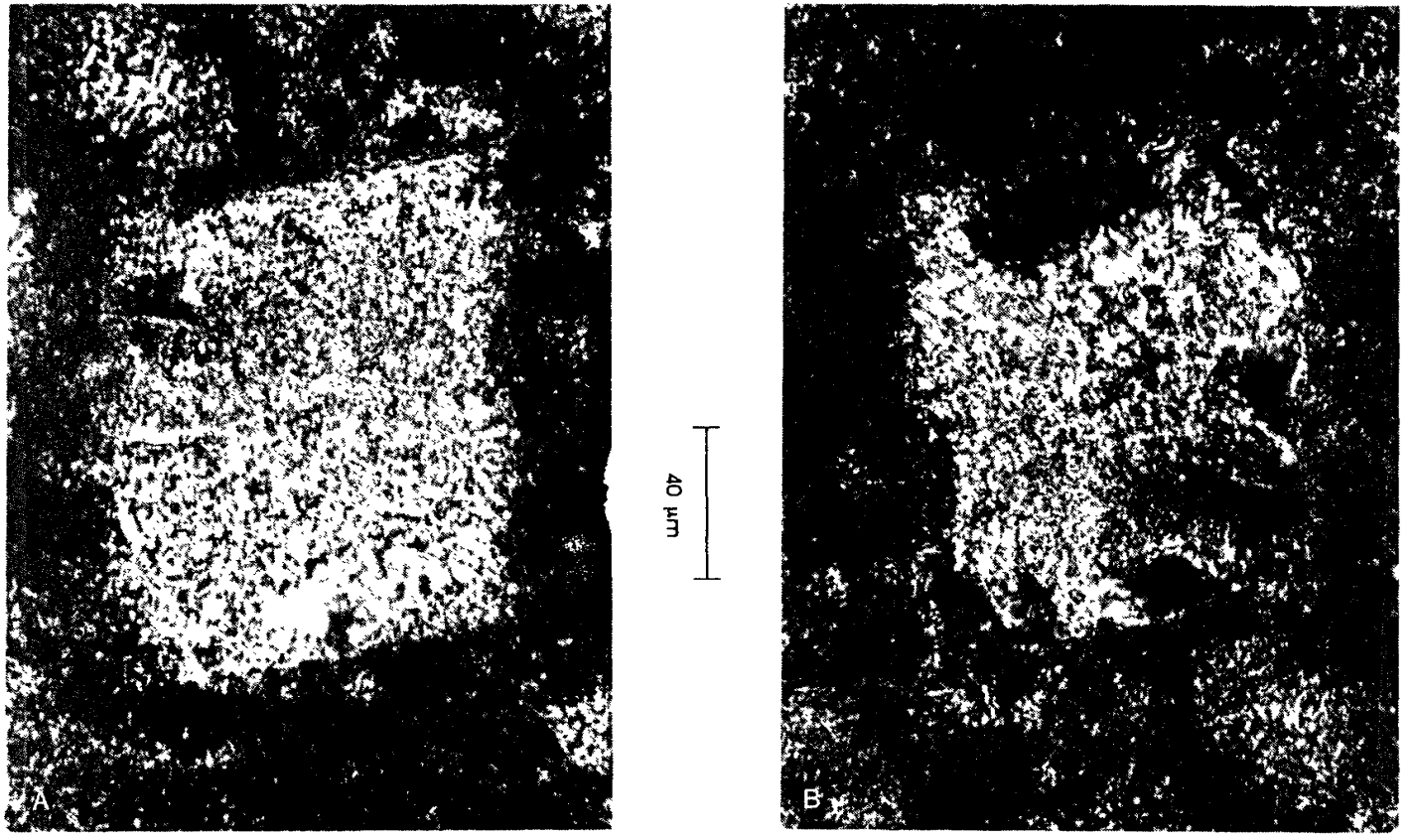


Figure 10-21 REACTED DOLOMITE CRYSTAL. A. Rhombic shape of an unusually large dolomite crystal in very dark micrite viewed with plane polarized light (from very reactive carbonate aggregate tested in accordance with ASTM C 586). **B.** Same view as **A** with crossed nicols. Notice that a portion of the crystal has been dissolved away. The intense birefringence of calcite and dolomite would ensure the detection of these minerals.

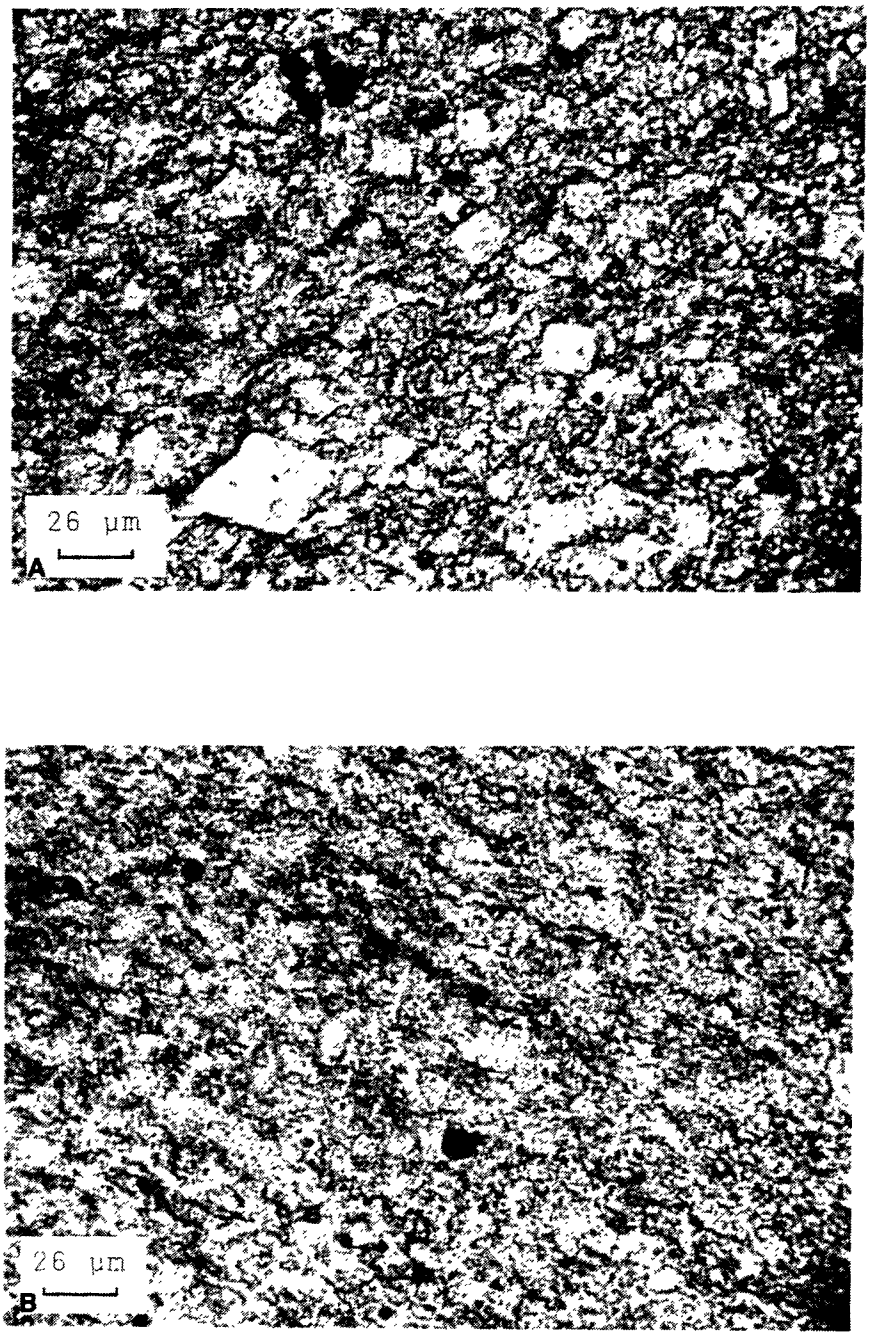


Figure 10-22 REACTION OF DOLOMITIC ROCK. A. Microstructure of a reactive dolomitic rock before soaking in a solution of sodium and calcium carbonates. B. Microstructure after test. Notice the destruction of the rhombic structures. The rock has been altered to a fine-grained mixture of calcite and dolomite.

10.3.4 Testing of Carbonate Aggregate

Specimens of aggregate are often submitted to the petrographic laboratory to be tested for suitability for use in HCC. Carbonate aggregates may be tested for potential alkali-carbonate reactivity. This testing is performed in the following three steps:

1. Examine numerous thin sections of the aggregate with the petrographic microscope. Because all reactive carbonate rocks have the distinctive microtexture described, the most efficient procedure is to examine for the texture. Such an examination requires no reaction time or special storage space. If the texture is found, the aggregate is to be considered potentially reactive. If the texture is not present, the aggregate is not considered alkali-carbonate reactive. If placements fabricated with this aggregate exhibit expansion, the aggregate may not have been properly sampled or the cause of the expansion may be an alkali-silica reaction.

2. Test aggregates considered to be potentially reactive in accordance with ASTM C 586 (rock cylinder method). This is the most rapid method of testing carbonate aggregates for alkali reactivity. It requires less storage space, less fabrication time, and less reaction time than any other test. If the aggregate shows no significant expansion, it is considered nonreactive. Only the potential for deleterious reactivity is determined because the expansion in the aggregate does not necessarily indicate that the expansion is powerful enough to disrupt concrete (Hilton, 1974). The method in ASTM C 227 cannot be used to test carbonate aggregate for reactivity because it requires that the aggregate be crushed to a small size (for use in mortar bars) and the larger size alkali-carbonate reactive aggregate is often more expansively deleterious than the smaller size (Newlon, Sherwood, & Ozol, 1972).

3. Test aggregates considered to be potentially reactive by reason of their expansion in accordance with ASTM C 1105 (concrete beam length change due to alkali-carbonate reaction). This test will detect most expansion caused by carbonate-reactive aggregate. Much instructive information concerning the testing of carbonate aggregate may be found in Newlon, Sherwood, and Ozol (1972). Recognize that HCC placements that either have been allowed to dry and kept dry or have such an impermeable structure that water cannot circulate within the concrete will probably show no reaction or distress regardless of the results of any testing regime.

Chapter 11
PARTICULATE MATERIALS OTHER THAN PORTLAND CEMENT

11.1 OVERVIEW

The concrete petrographer is often called upon to ascertain the presence or absence of particulate materials other than portland cement in a specimen of HCC. These materials include all the finely comminuted solid substances that may be added to HCC to improve its properties or reduce its cost. These substances are of relatively small grain size, usually at least as small as that of portland cement. Included are pulverized limestone (Kleiger & Hooton, 1988), hydrated lime, natural pozzolans, fly ash, GGBFS (Hwang & Shen, 1991), and silica fume (see Tuthill, 1978b; Transportation Research Board, 1990).

In general, when these materials have been used in a concrete mixture as prescribed, the resulting concrete is tougher and less permeable than a similar concrete that does not include these materials. Historically, some of these substances were considered to be adulterants and the use of them was considered an effort to dilute the cement and produce a less costly, less energy-intensive product of unknown quality. However, in general, these materials improve HCC and the major drawback to their use is delayed strength gain (except with silica fume). In cold weather, this delay can be sufficient to mandate against their use. When HCC containing these materials has not attained the expected maturity and is used to support further placements, equipment, or personnel, the results can be disastrous. The petrographer should be aware of these problems. Where young HCC failed to bear the expected load, the cause could be the use of materials whose slow strength gain was not properly allowed for.

These materials should not be considered as adulterants when they have been specified for use and necessary allowances made. The particulate materials that are rich in amorphous silica (GGBFS, fly ash, silica fume, and natural pozzolan) are pozzolanic and will combine with the calcium hydroxide of HCC to form silicate hydrates that are cementitious and indistinguishable from the hydrated products of portland cement. According to B. Mather (personal communication, October 1991), GGBFS is a slowly hydrating hydraulic cement and is, therefore, not a pozzolan, although its action is, in many ways, similar to that of a pozzolan (ACI 226.1R). Such a reaction can, during the early stages of the setting of the cement paste, cause the sequestration of deleterious alkalies in nonswelling silica gels (see Appendix F). These gels may be thinly distributed throughout the paste. Thus, these pozzolanic materials can control the deterioration that might be caused by the alkali-aggregate reactions.

The fine grain size, slow hydration, and, in some cases, particular particle shape add desirable properties to HCC. The heat of hydration is generated more slowly, and, therefore, the temperature of the HCC does not rise so high. One of the most important desirable properties conferred by these materials is the decreased permeability of the resulting HCC. The decreased permeability is not only desirable in and of itself, it also decreases the movement of solutions and diffusion of ions and

thus any chemical activity, such as that of alkali reactivity or the activity of invasive deleterious substances. Pozzolanic materials are reported to be effective in increasing the acid resistance of concrete (see Tuthill, 1978a). Natural pozzolans and fly ash should comply with the requirements of ASTM C 618, and GGBFS with ASTM C 989; a separate specification for silica fume is in preparation.

The appearance of a mature concrete in which these materials have been used is generally that of concrete with a low water-cement ratio (see Chapter 9). Pulverized limestone and hydrated lime cannot be positively identified in thin sections of mature HCC. Their presence may be suspected in HCC that appears to have the texture of an HCC with a low water-cement ratio, but they cannot be positively identified. It is impossible to be certain with mature concrete, and without any magnification, if GGBFS or silica fume is present or if the appearances of impermeability are due solely to a low water-cement ratio.

11.2 PROCEDURES

11.2.1 Ground Granulated Blast-Furnace Slag

1. Examine exterior surfaces, broken surfaces, and lapped slices of the HCC. When very young, the pastes containing GGBFS are a characteristic dark bluish green. The chemical reasons for this color in portland blast-furnace clay cements were investigated by Mather (1957), and it was concluded that the development of the color varies from cement to cement and that the coloring agent is in a reduced state, the color being lost on oxidation. Regardless of maturity, if these pastes are kept underwater and not allowed to dry, they will retain this color. As the concrete begins to dry, the surface color fades. If the HCC is sawed or broken while it is only partially dry, it can be seen to be mottled with dark and light places. When it is dry throughout, it will be a uniform light color (see Fig. 11-1). This color is not the same color as ordinary HCC. It is not a gray but rather a very light shade of slightly greenish tan or cream. The mottled appearance of an interior surface of GGBFS-bearing concrete of intermediate age is often of concern to field personnel. It has often been thought that the mixing of the HCC was incomplete. In thin sections examined with the petrographic microscope, the dark-colored areas appear no different from the faded areas and both types of areas seem to have about the same concentration of the GGBFS. If GGBFS was used in the mixture, this mottling is inconsequential and will fade. GGBFS cannot be detected in mature, fully dried concrete with the stereomicroscope.

2. Examine several thin sections of the paste of the specimen with the petrographic microscope. GGBFS can be seen in thin section and is quite distinctive in appearance. It is primarily glass and, therefore, almost completely lacking in birefringence.

3. Compare the view observed with the views in Figure 11-2. If GGBFS cannot be found by examination of the paste in a thin section, report that it was not detected in the specimen. Figure 11-2 illustrates the appearance of GGBFS at three stages of hydration.

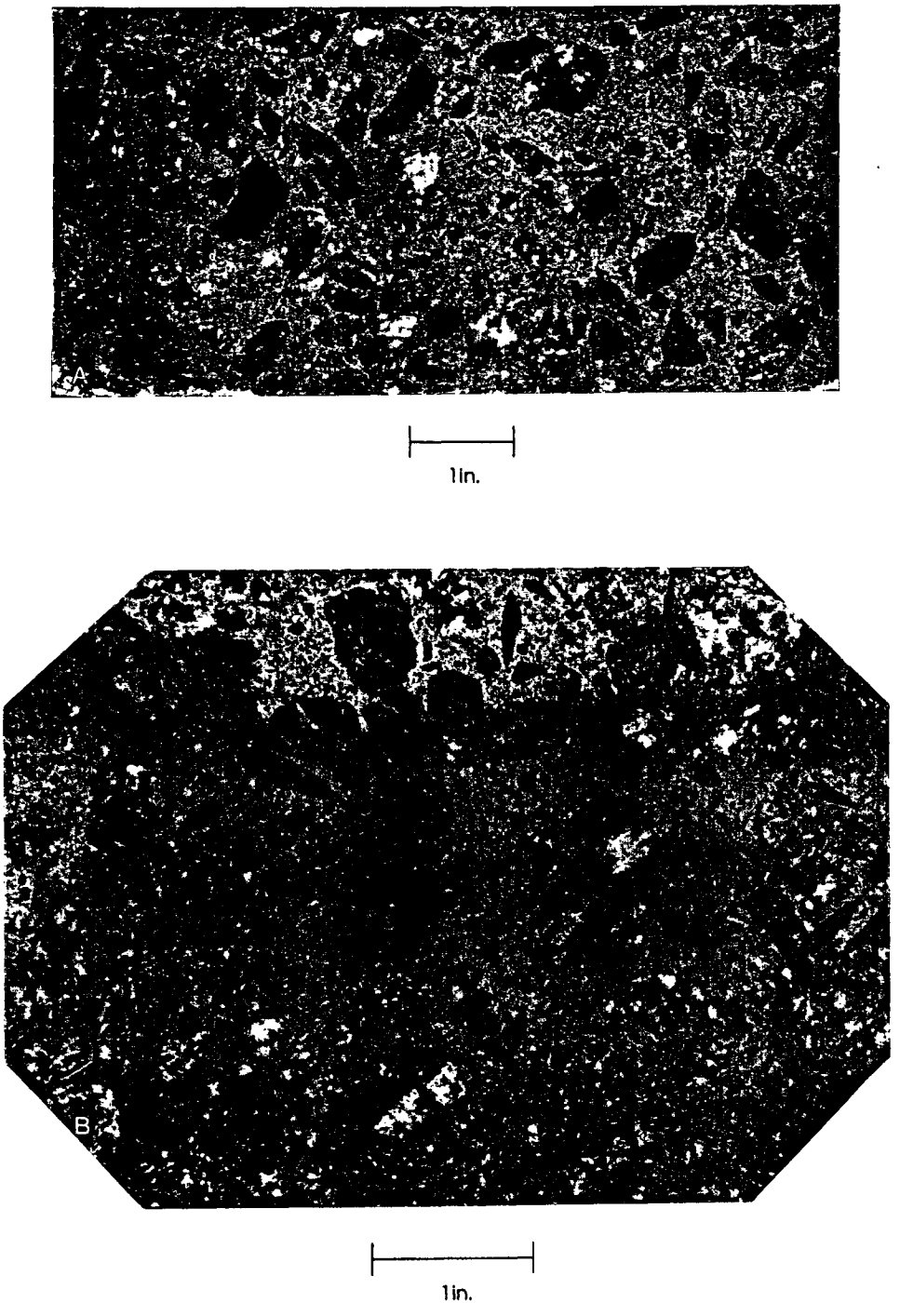


Figure 11-1 HCC CONTAINING GGBFS. A. Cut surface on HCC containing GGBFS exposed to air 6 months. **B.** Interior of beam of HCC containing GGBFS, illustrating the partially dry two-color stage.

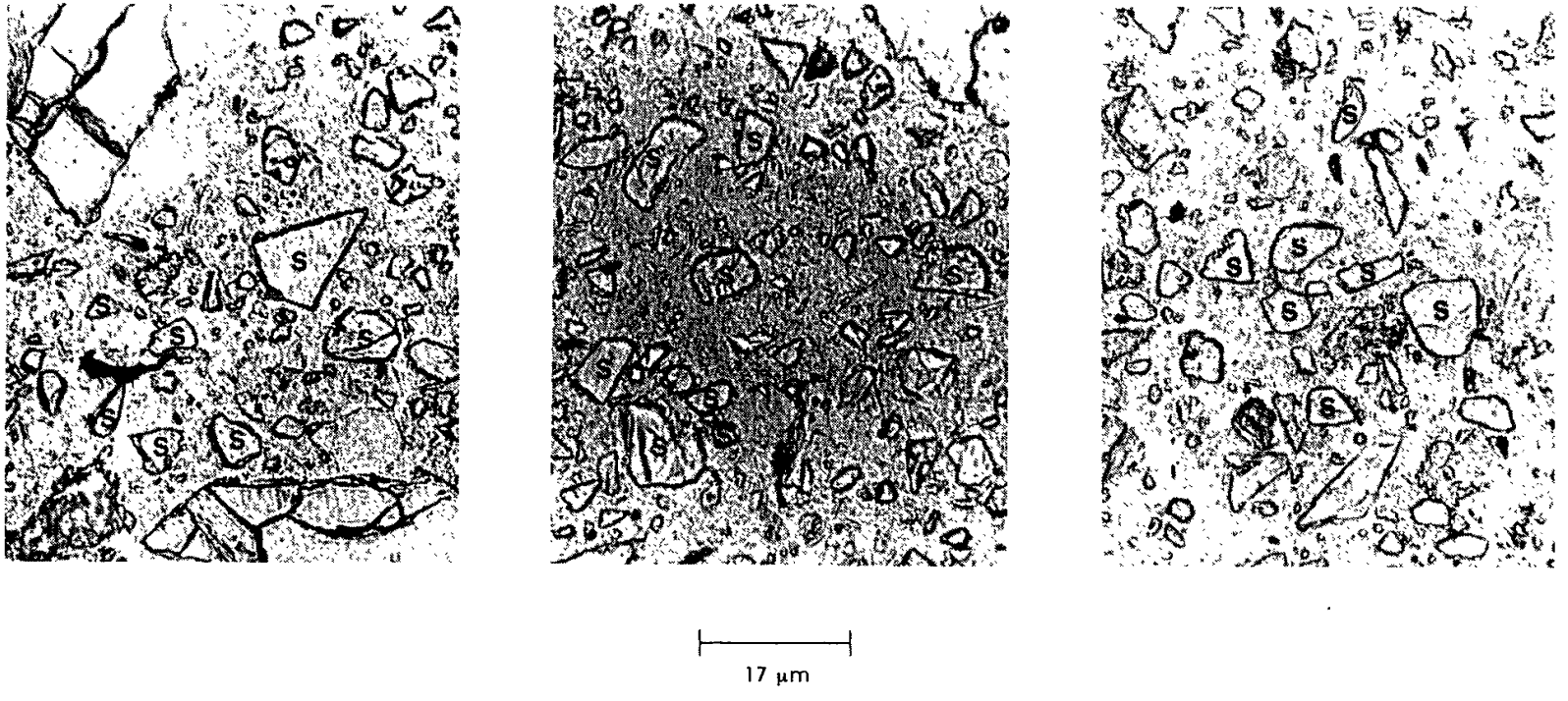


Figure 11-2 THIN SECTIONS OF CONCRETE CONTAINING GGBFS. Concentration is 65% of cementitious material. Slag particles are marked with an *S*. **A.** At 28 days hydration. Note the angularity of the slag fragments. **B.** At 56 days hydration. Note the slight rounding of the slag fragments. **C.** At 6 months hydration. Note the further rounding of the slag fragments.



Figure 11-3 ETCHED AREA OF LAPPED SLICE CONTAINING FLY ASH. The partial fly ash particles are indicated by *arrows*. The particle at the *starred arrow* is a frothy fly-ash agglomeration. Nearly complete white cenospheres probably filled with froth or a multitude of smaller cenospheres are marked with a *W*. These whole cenospheres have been almost completely exposed by the etch procedure; some small whole cenospheres may have been lost during the etch procedure. Cup-shaped portions of cenospheres are marked with a *C*.

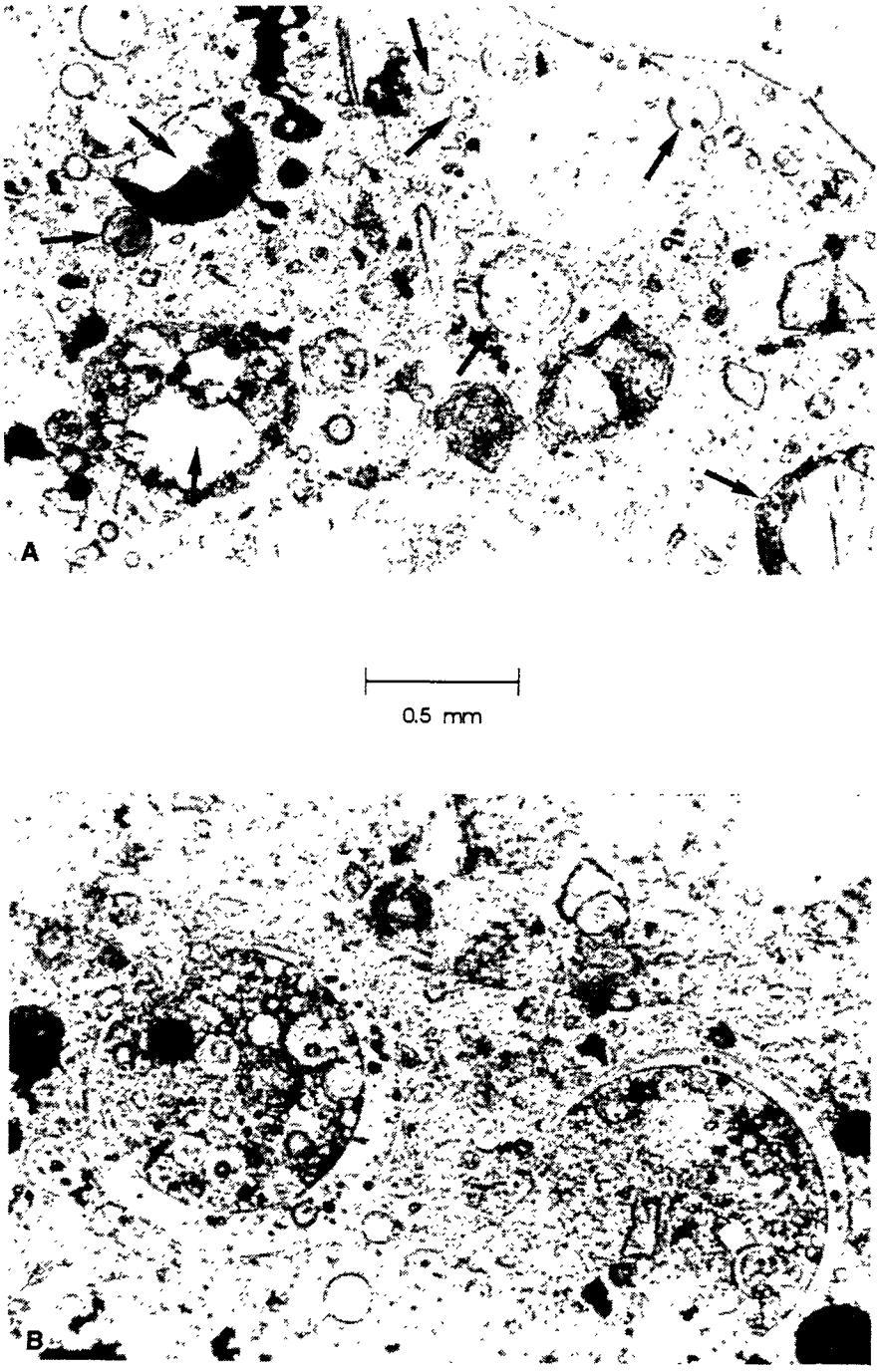


Figure 11-4 FLY ASH IN THIN SECTION OF HCC. As seen with petrographic microscope. **A.** Fly ash particles are indicated by *arrows*. Note the broken fly ash particle that is composed of dark and light glass. **B.** Cenospheres of fly ash filled with smaller cenospheres.

11.2.2 Fly Ash

1. Examine a lapped slice of the HCC with a stereomicroscope. Fly ash is usually of quite varied particle sizes, and the larger particles can be seen with the stereomicroscope. Most of the individual particles of fly ashes approved for use in HCC are colorless to white. Some of the larger particles are hollow spheres: cenospheres. The walls of the cenospheres are frequently thin enough and the cenospheres large enough to be mistaken for entrained air voids (see Walker, 1983). Sometimes, little dark (black or brown) balls of fly ash (sometimes hollow) can be seen; sometimes, agglomerations of fine fly-ash froth are present (see Fig. 8-6).

2. To detect all the particles of fly ash and distinguish them from air voids, etch the slice or a portion of the slice, rinse, blot, allow to dry, and examine with the stereomicroscope (see 5.2.3). Observe the acid-resistant glass walls of any fly ash particles (see Fig. 11-3). These glass walls are very thin but can be seen with low-angle illumination projecting above the paste that has been etched away. Air voids will not have such projecting walls.

3. Examine thin sections with the petrographic microscope (see Fig. 11-4).

4. If fly ash cannot be detected on an etched lapped slice and cannot be found in the thin section, report that it could not be found in the specimen.

11.2.3 Silica Fume

Silica fume cannot be detected by any of the methods commonly in use in the average concrete petrography laboratory. Silica fume has such small particles and is so lacking in birefringence that its presence cannot be detected even in thin section at 400X. Report that the presence of silica fume cannot be detected by the laboratory. (Laboratories that have the capability of examining specimens with a scanning electron microscope can detect and photograph this material.)

Chapter 12
EXAMINATION WITH THE PETROGRAPHIC MICROSCOPE

12.1 OVERVIEW

A petrographic microscope, also called a polarizing microscope, is best described as a compound transmitted-light microscope to which components have been added to enable the determination of the optical properties of translucent substances. The designation of the microscope as a compound microscope indicates that it has an ocular that focuses on a virtual image of the subject produced in the tube of the microscope by the objective lens.

The professional petrographic microscope has a substage condenser that can be centered and focused. The substage has field and aperture diaphragms. The polarizing components are the upper and lower polarizing devices, the Bertrand lens and its mounting (between the upper polarizing device and the ocular), an accessory flip-in lens for convergent light mounted as the top element of the condenser, and a graduated rotating stage with a removable click stop that can be activated to indicate a 45° rotation from any selected direction. The focus knob(s) and the stand are graduated to permit the determination of thickness by differential focusing. Figure 12-1 shows a petrographic microscope.

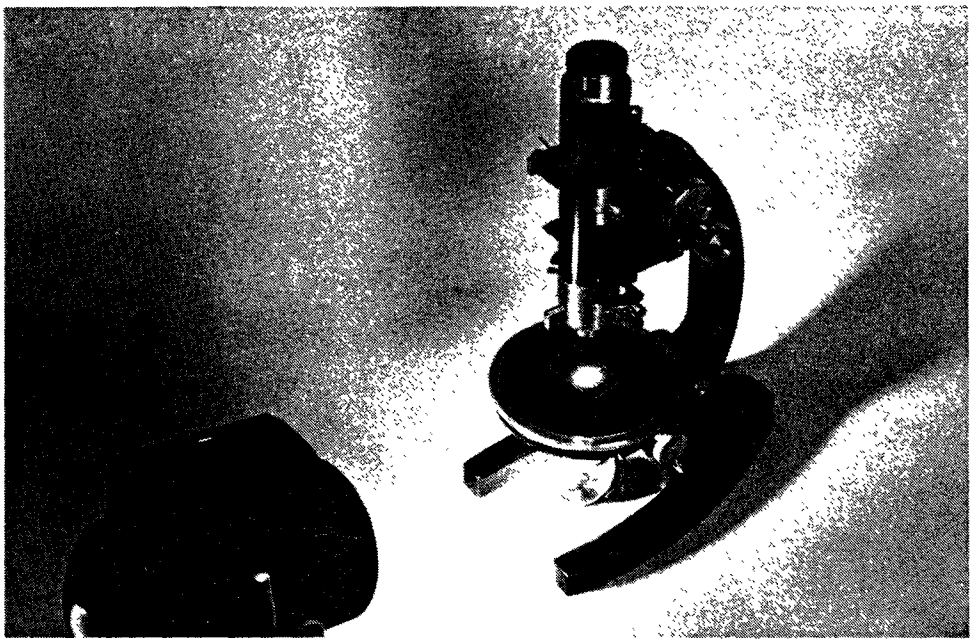


Figure12-1 PETROGRAPHIC MICROSCOPE

The first polarizing devices were prisms fabricated from crystals of the mineral calcite specially cut and cemented back together according to the plan of Nicol (Nicol, 1828, as cited in Johannsen, 1968). Such devices were originally called nicol prisms (Johannsen, 1968). Now they are called nicols. Crystals of calcite, $CaCO_3$, were chosen because light that goes through this substance in certain directions is split into two distinctly different rays strongly polarized at 90° to each other that travel at widely different indices of refraction; thus, calcite has a high birefringence. Nicol used the plane on which he cemented the crystals back together as a plane of total reflection for the ray with the index of refraction most different from that of the cement.

In modern petrographic microscopes, the nicols are polarizing plates fabricated in much the same way as are the lenses in polarized sunglasses. If two polarizing plates of sufficient thickness and quality are superimposed with their polarization directions at right angles to each other (thus, crossed nicols), no light can penetrate the pair. This is because the first polarizing plate excludes all light that is polarized perpendicular to the direction of the plate polarization, concomitantly polarizing the remaining light parallel to the plate polarization, and then the second plate does likewise. Together, all light is excluded. An indication of this effect can be observed by looking through two polarized sunglasses lenses superimposed at 90° to each other. Polarized sunglasses work because all reflected light is (at least partially) polarized parallel to the substance from which it is reflected and the polarizing material prevents the passage of rays polarized in a horizontal direction, such as that reflected from puddles, snow, automobile surfaces, and pavement. The effect may be observed if you look through a polarized lens at a patch of reflected glare while turning the lens to various orientations relative to the polarization of the glare. (The sky is polarized by reflection from the molecules of the atmosphere.)

A properly adjusted petrographic microscope with nicols of sufficient quality allows no light discernable to the eye to penetrate the upper and lower polarizing devices when their polarization directions are at 90° to each other and there is no birefringent substance between them. However, if a birefringent substance (such as a crumpled piece of cellophane) is placed between two polarizing plates that are positioned with their polarization directions at 90° to each other, the birefringence of the inner substance polarizes the light that travels through the first polarizing plate in directions parallel to the optical directions of the substance and the optical system will transmit light. The intensity and color of this transmitted light are controlled by the birefringence, optical orientation, and thickness of the interior substance. The lower and upper nicols, or polarizing plates (called the polarizer and analyzer, respectively), of the petrographic microscope act as do the plates of polarized material. If both nicols are in the optical path and are oriented at right angles to each other and a birefringent specimen material is on the stage, the amount of the birefringence and many other optical properties may be determined.

In the petrographic microscope, the light is collimated by the condenser into a bundle of beams all parallel to the optic axis of the microscope. The specimens examined are transparent-to-translucent thin sections or grain mounts of the material under study (see 5.3 and 5.4). The light beams are polarized in one direction (by the polarizer) before the light reaches the specimen. This light is called *plane polarized light.*

The direction of polarization produced by the polarizer varies from microscope to microscope (usually north-south) and is often adjustable. The analyzer may be

placed in the path of the light after it leaves the specimen whenever birefringence or optical directions are being determined. The analyzer is identical in nature with the polarizer, and for most work, the polarization of the analyzer is perpendicular to that of the polarizer (i.e., the analyzer polarization is east-west). In the standard orientation, the analyzer allows the transmission of light that is not polarized at right angles to the light from the polarizer.

When both nicols are in use in this standard orientation, the object on the stage is said to be viewed with crossed nicols. In addition, most petrographic microscopes are equipped with a slot at 45° to the main polarization directions and various retardation plates that can be used in this slot in the determination of a number of optical properties. The most common and most useful of these plates is the 1/4 wave plate, or gypsum plate.

12.2 USES

The uses of the petrographic microscope include identifying translucent substances by means of their optical properties and by reference to the various charts and tables in the literature (Bloss, 1961; Kerr, 1959; Larsen & Berman, 1964; Rogers & Kerr, 1942). Thus, the composition and identity of these substances and the relationships between various phases of the material under study may be discovered. From these data, facts concerning the history and method of formation of the subject material can be deduced. The optical properties of substances given in texts, charts, and graphs have generally been accurately determined by the use of a universal stage on which the substance can be oriented at any desired angle to the optic axis of the microscope and the plane of rotation. In this manner, the optical properties have been determined for various substances. The concrete petrographer does not usually try to attain this degree of accuracy.

12.3 PROCEDURES

Courses on the use of the petrographic microscope are available at most colleges and universities that have departments of mineralogy or geology, in most departments of materials engineering, and in some departments of chemistry. The textbooks available on this subject vary widely in their emphasis. Certain texts concentrate on the theories of the behavior of light in various types of crystal structures, use of the Bertrand lens, and various optic axis figures. Some are oriented toward identifying and naming the minerals; others concentrate on teaching recognition of individual types of rock.

The recommended procedures vary from one author to another. For example, Bloss (1961) taught that birefringence is the most important property by which to identify a mineral substance. His book has charts and graphs that start out with the determination of this property and then branch to include other properties. The charts and lists of Larson and Berman (1964) have their first subdivision of lists of minerals on the number of optic axes possessed by the substance, the second subdivision on the optic sign, and the third on the index of refraction. Deer, Howie, and Zussman (1962) and Palache, Berman, and Frondel (1951-62) wrote important mineralogic reference books that list the optical properties of minerals in the part of the text that describes the mineral under discussion.

The standard texts written for geologists and mineralogists assume that all thin sections are 25 to 30 μm thick. With thin sections of HCC and similar materials, the grain size is so small that much thinner sections are often desirable (see 5.3.1). Unless highly specialized equipment is used, it is impossible to produce ultrathin sections that are the same thickness across their areal extent. Often, a thin section will vary in thickness from 20 μm in one area to nothing in another. This lack of flatness will, at first, seem objectionable to the average classically trained petrographer, but once having become accustomed to, the microscopist will realize that the lack of flatness can allow a mineral substance to be viewed in a greater variety of ways than if the section were the same thickness everywhere.

The identification of aggregate minerals and rocks and concrete reaction products may usually be most efficiently accomplished by knowing which mineral substances are likely; noting the outward physical properties, color, cleavage, and hardness either in a hand specimen or with the stereomicroscope; and using the petrographic microscope to observe the general appearance in polarized light to determine the approximate birefringence, indices of refraction, and some of the other optical properties. The procedures involved include determining of some of the following optical properties, listed in order of most common usage:

- The distinctive cleavage, growth lines, inclusions, and parting patterns.
- Positions of extinction (directions in the substance where crossed nicols permit the passage of least light).
- The alignment of the positions of extinction with specific directions (crystallographic axes, cleavage planes, growth lines, etc.) within the subject substance.
- The birefringence as estimated from the maximum double refraction (highest order of color seen when the positions of extinction are at 45° to the nicols) and the thickness of the crystal being examined. The thickness of the subject substance may be determined by differential focusing or by the double refraction exhibited by adjacent substances of known birefringence. The birefringence of the mineral substances is listed in the various charts and tables of mineral properties (Bloss, 1961; Kerr, 1959; Larsen & Berman, 1964; Rogers & Kerr, 1942) and is the difference between the highest and lowest index of refraction of the substance. The maximum diffraction is used to determine this property. For this purpose, grain mounts are much more suitable than thin sections. If the substance has a pronounced cleavage or crystal shape that influences the orientation of the particles on the glass slide of a grain mount, some particles should be induced to roll to a new orientation by the nudging of the cover glass with a needle. Birefringence is one of the least variable of the easily determined optical properties. It is relatively constant throughout a family of minerals (such as the feldspar family); the indices of refraction and color may vary from one family member to another.
- The pleochroic properties (if any) as determined by the change in color and intensity of color observed in plane polarized light as translucent substances are turned to the various positions of extinction; the orientation of these color changes with respect to cleavage and crystallographic axes is an important part of the data on pleochroism.
- The indices of refraction of a substance determined by comparison in plane polarized light with the index of refraction of the medium (other minerals, mounting epoxy, index of refraction oil, etc.) with which it is surrounded. This is usual-

ly done by noting the motion of the Becke line (the bright line that develops as the objective lens is moved out of focus). This determination is aided by the contrast (relief) with which the substance is seen. The greater the difference in the index of refraction between the subject substance and its surroundings, the greater the difference in relief between the substances. This contrast will change as a highly birefringent mineral is rotated from one position of extinction to another and a light ray with a different index of refraction is made parallel to the polarizer. For more exact work, the substance is isolated and fragments are mounted in various standard index of refraction oils.

- The alignment of the individual indices of refraction of a substance with specific directions (crystal axes, cleavage planes, growth lines, etc.) within the subject substance.
- The number of optic axes, optical sign, and angle between the optic axes. These are determined by means of the conoscopic lens arrangement (i.e., using convergent light and the Bertrand lens). If the Bertrand lens cannot be focused or is otherwise imperfect, smaller but often sharper optic axis figures may be observed without it by removing the eye lens and looking down the microscope tube at the back lens of the objective. To center the microscopist's eye, a pinhole eyepiece is often used. The data concerning the optic axes and optical sign are not often required in the petrographic study of HCC.

Many identifications can be made from the general appearance, parting, color, cleavage, estimated index of refraction, and approximate birefringence. The experienced petrographer can accurately recognize a large number of minerals by observing the general appearance in plane polarized light and the birefringence as viewed with crossed nicols. Of course, unfamiliar substances will require the determination of a number of various properties before identification can be made.

Chapter 13
EXAMINATION WITH THE POLARIZING/EPIFLUORESCENCE MICROSCOPE

13.1 OVERVIEW

The use of fluorescence microscopy in the study of HCC was initiated by Wilk, Dobrolubov, and Romer (1974) in Switzerland and was then used mainly as a tool to determine the quality of the air-void system in HCC. Wilk and associates used ultraviolet light transmitted through a thin section. This work prompted my development of a multimode microscope (Walker & Marshall, 1979). The object of the design of the P/EF microscope was to make it possible to view any spot on a thin section with all of the illumination modes of a petrographic microscope and with incident ultraviolet illumination on a single microscope stand so that the specimen did not have to be physically moved from one instrument to another and various forms of illumination did not have to be removed and replaced as the mode of illumination was changed. All the modes of viewing that are possible with a petrographic microscope were combined with the ability to view the fluorescence (when illuminated by ultraviolet light) within a thin section impregnated with specially dyed epoxy (see 5.3.4). The design is such that changing the exciter filters by adjusting a turret housing and the dichroic mirrors (DM) by moving a slide across a slot, exchanging barrier filters (BF), and flipping shutters are all that are required to switch from one mode to another. Soeder (1990) used epifluorescence microscopy with a different dye, DM, and BFs to study the pore structure of rocks of low permeability.

It was found that when ultraviolet illumination was transmitted through the thin section it caused fluorescence of all the fluorescent dye throughout the thickness of the section. The dye was distributed through all the capillary pores, in all the cracks no matter how small, and in all the small voids remaining in the paste as the cement hydrated. The fluorescence existed throughout the thickness of the section and produced a haze of uncollimated light that confused all viewing with a cloud in which any opaque particles (such as cement ferrites) seemed to float. If the section was more than 25 μm in thickness, even some of the air voids would be difficult to distinguish from the background haze. The work of Beauchamp and Williford (1974) and Beauchamp, Williford, and Gafford (1972) indicated that thinner sections would provide more definition. It was determined that the ultraviolet illumination must be incident (come from the direction of the objective, i.e., epi) upon the thin section so that the portion of the specimen being viewed was the first part of the specimen illuminated; thus, the ultraviolet light exciting the most clearly viewed fluorescence was not shaded by other portions of the specimen (Walker & Marshall, 1979).

The question has been asked: "Why use a thin section? Why not just use a highly polished slice for this incident light microscopy? After all, that is how one studies ore specimens." The answer is two-fold: (1) Ore specimens are opaque, and only the features on the surface are visible in incident illumination. Most of the components of HCC are translucent, and light penetrates the specimen. Ultraviolet light causes

fluorescence throughout the entire thickness of the specimen and creates a pervasive yellow glow caused by the fluorescent-impregnated porous areas of the paste throughout. This glow of uncollimated fluorescence looks like a pool of glowing liquid in which any opaque particles seem to float. Other details are masked. It is necessary to make the specimen being examined thin so that the unwanted ultraviolet light can go on through and not bounce around and make noisy, meaningless fluorescence. (2) We need to be able to look at any spot of interest with the standard petrographic methods (that require transmitted collimated light) as well as with the fluorescence-causing methods, and we need to do it without switching microscopes and losing our place on the section.

The incident arrangement of the ultraviolet illumination necessitated using uncovered thin sections and the type of objective lenses required by uncovered thin sections. The light created within the specimen by the fluorescence of the dye is uncollimated and radiates from every fluorescent point. Such uncollimated light strikes a cover glass at random angles. Only light that strikes a cover slip at 90° to the surface will travel straight on through. The light produced at other angles bounces around under the cover slip and from the highly finished surface of the thin section and creates a yellow haze that obscures the viewing at all but the lowest magnifications.

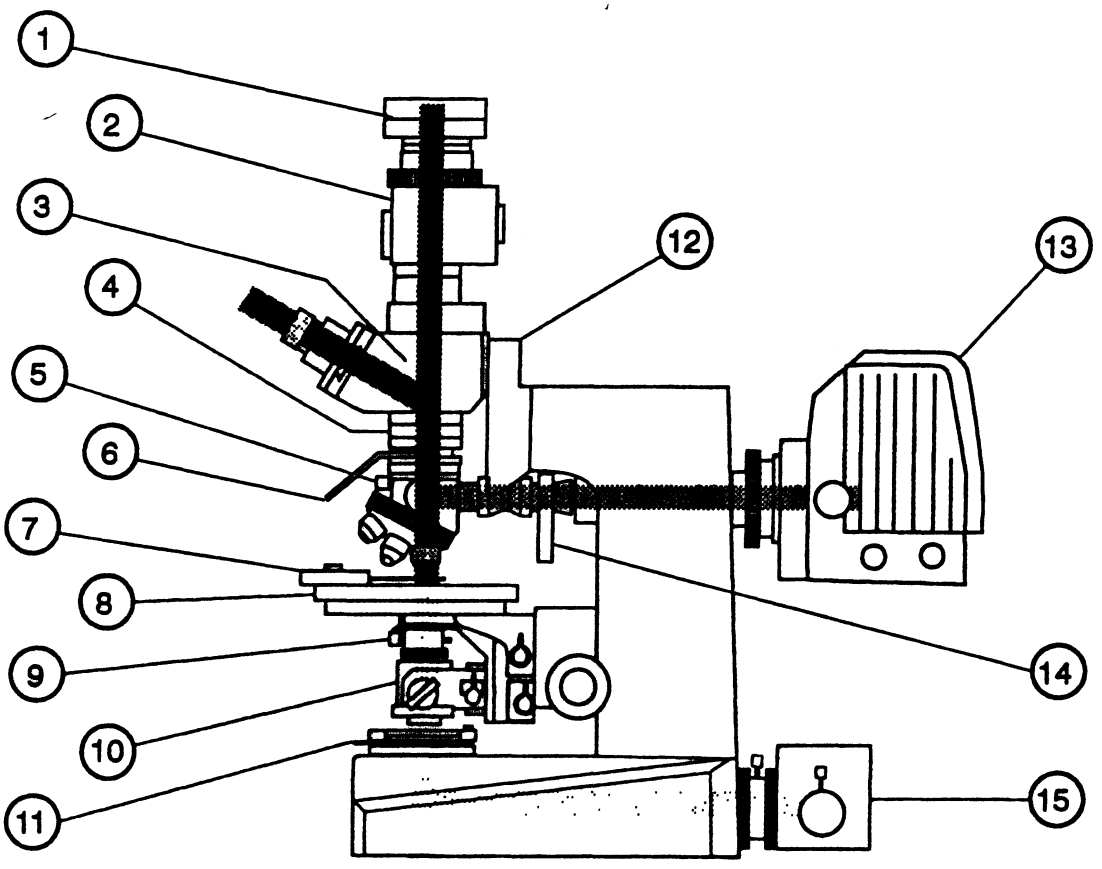
Because the incident ultraviolet light is collimated by the objective lens, there is a greater concentration of this light at the point of focus with the higher power lenses; i.e., more ultraviolet light reaches each fluorescent molecule of the dye when the higher power objectives are used. Therefore, the fluorescence is brighter when the higher power objectives are used. At very high magnifications (60X objective), the lens and lens mounting are so close to the specimen that a yellow haze is produced by light bounced from the lens surface to the specimen surface and back, just as when a cover slip is used. This effect precludes the use of oil immersion lenses and lenses of greater power than 40X.

This microscope is diagrammatically shown in Figure 13-1, and a photograph of the equipment is shown in Figure 13-2 (see Walker, 1988). The P/EF microscope described in this chapter was obtained in 1977 under strict budget requirements. A more modern P/EF microscope that was specifically designed for this use would be more convenient than the model described here. Such a microscope might have different exciter filters, DMs, and BFs built into the instrument and would probably have different engravings for the various filters.

The ultraviolet light is incident on the specimen and is produced by a 200-watt mercury arc lamp. Incident microscope illumination requires a vertical illuminator to direct the light to a special mirror (DM) above the objective lens that will direct the light down through the objective without hindering light traveling from the objective to the ocular. For ultraviolet illumination, the wavelength of the light is controlled by the exciter filters, DMs, and BFs.

The vertical illuminator has a built-in turret for exciter filters:

- UG-5, ultraviolet light (U engraving)
- BG-3, violet light (V engraving)
- BG-12, blue-violet light (B engraving)
- IF-545 and BG-36, green light (G engraving)



1. CAMERA BACK, 35 MM
2. AUTOMATIC EXPOSURE METER
3. BINOCULAR TUBE
4. ANALYZER AND BERTRAND LENS
5. DICHROIC MIRROR AND BARRIER FILTER SELECTOR
6. LIGHT SHIELD
7. POINT COUNT STAGE
8. ROTATING STAGE THAT CAN BE CENTERED
9. POLARIZING CONDENSER
10. AUXILIARY LENS SYSTEM
11. SWING OUT MOUNT FOR BG-12
12. STAND MODIFICATION
13. MERCURY BURNER, 200 W, FOR INCIDENT ILLUMINATION
14. EXCITER FILTER TURRET
15. HALOGEN LIGHT FOR TRANSMITTED ILLUMINATION

COMBINED ILLUMINATION

INCIDENT ILLUMINATION

TRANSMITTED ILLUMINATION

Figure 13-1 LIGHT PATHS IN P/EF MICROSCOPE

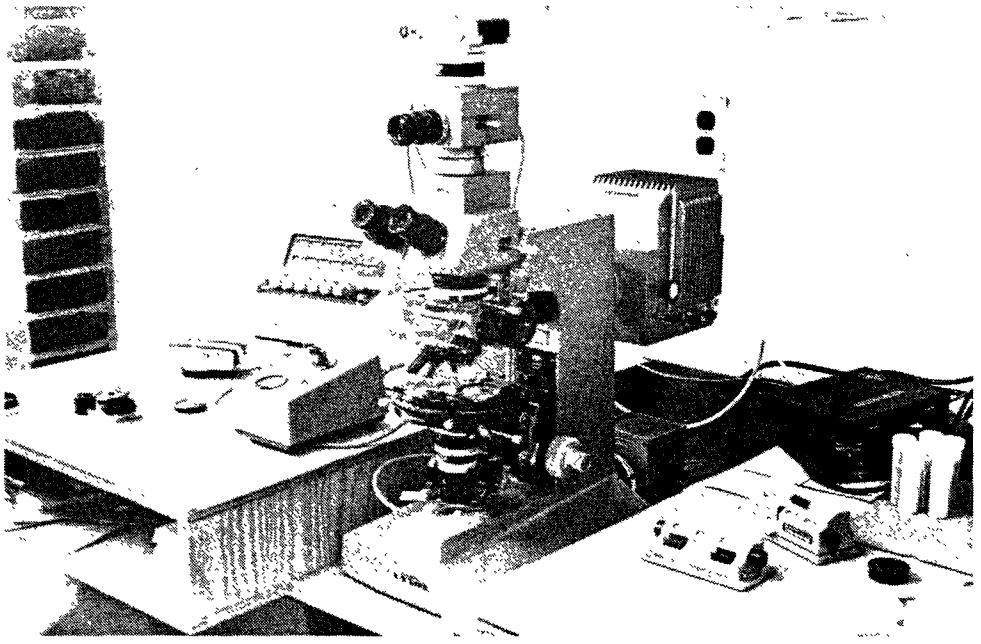


Figure 13-2 P/EF MICROSCOPE. In the right foreground are two pushbutton counters. The camera and exposure meter are on top of the microscope. The control for the exposure meter is between the microscope and the Swift automatic point-counter keyboard in the left background.

There is a slide containing the complementary DMs and BFs controlled by a lever in a four-position slot:

- DM 400 with BF L-410 (U engraving)
- DM 455 with BF Y-455 (V engraving)
- DM 500 with BF O-515 (B engraving)
- the fourth position (G engraving), a straight-through path for ordinary polarized light microscopy (factory altered)

For the particular fluorescent dye in use, the exciter filters built into the turret in the vertical illuminator are usually sufficient. The only exciter filters used are the BG-3 (V engraving) and the BG-12 (B engraving). The illuminator has an accessory slot for the insertion of extra filters. Occasionally, it has been convenient to have an extra BG-12 filter for use in the optional exciter filter slot. The only additional BFs used have been a Y-485, a Y-495, and an O-515. Figure 13-3 illustrates the relationship of the filters to the dye emittance spectrum. A different dye would have necessitated different filters. To lessen the eye fatigue due to the high contrast, a small amount of transmitted light filtered by a BG-12 filter 3.0 mm in thickness is used. A special swing-out mount for this filter was fabricated in the VTRC shop, as illustrated in Figure 13-4.

The transmitted light is from a low-voltage halogen source. The microscope accessories include a Swift automatic point counter, several other pushbutton counters (for counting voids, cracks, and traverses), and a filar micrometer.

The fluorescence elements are used in the study of HCC mainly because much of the paste (especially the hydration products of the cement) is colorless and has a

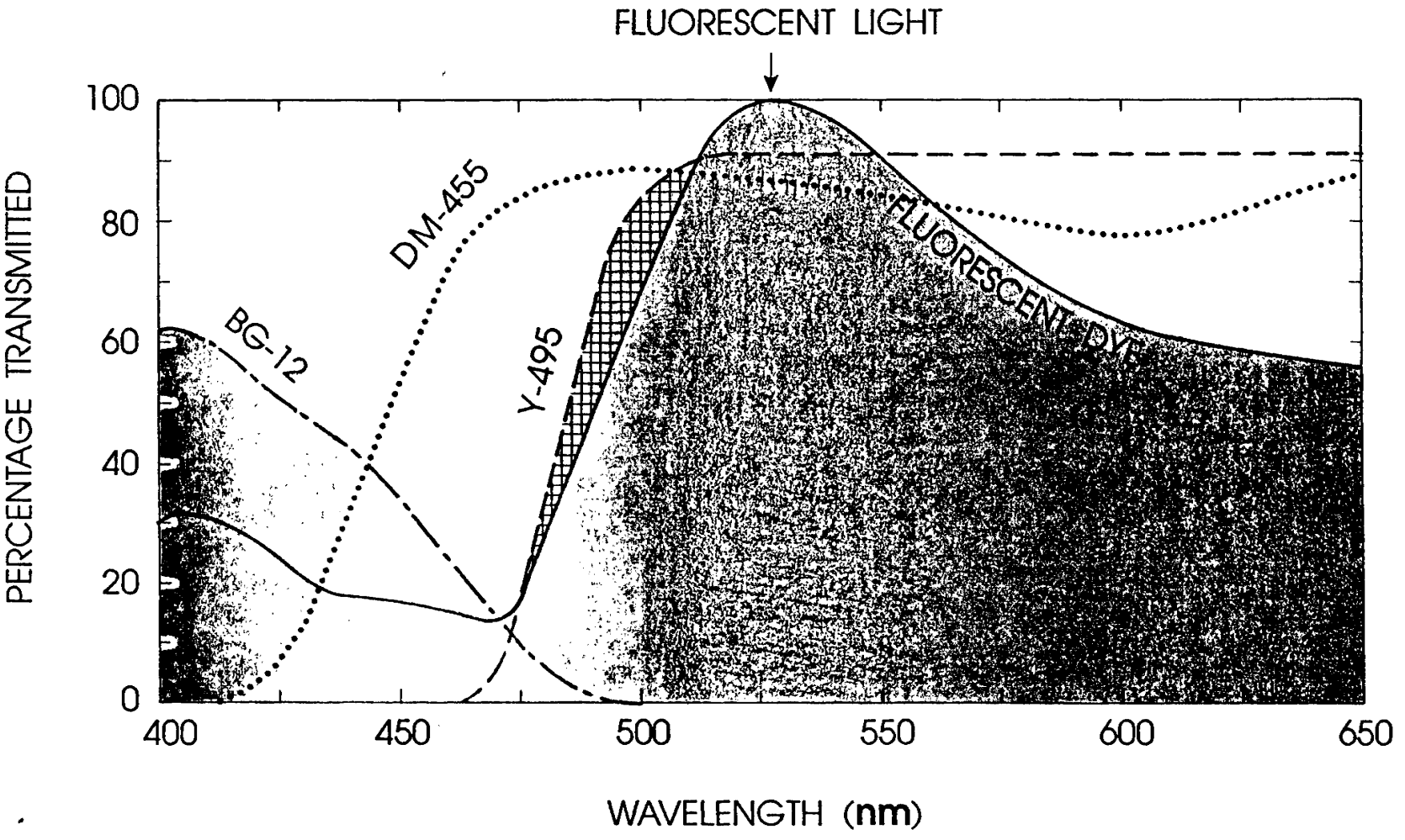


Figure 13-3 RELATIONSHIP OF FILTERS TO DYE EMITTANCE SPECTRUM. Percentage transmittance of exciter filters, dichroic mirrors, and barrier filters of a microscope compared with the excitation spectrum of fluorescent dye.

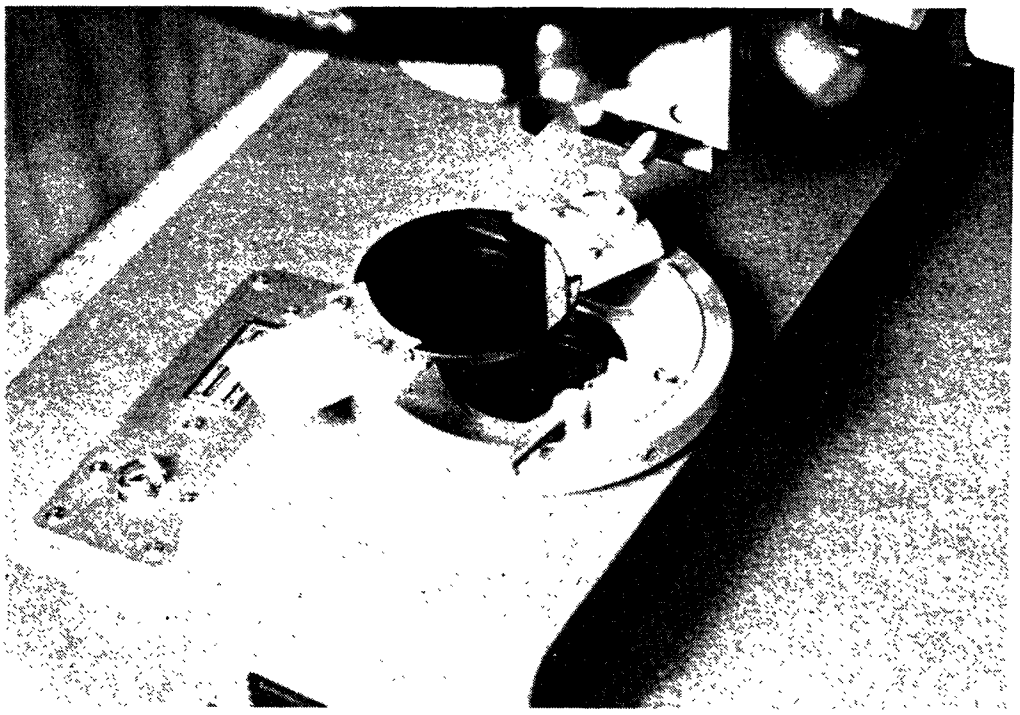


Figure 13-4 SWING-OUT FILTER OVER LIGHT PORT ON BASE OF MICROSCOPE. The filter holder was fabricated in the VTRC shop.

low index of refraction and essentially no birefringence. These properties of the paste are so much like empty space that it is nearly impossible to ascertain, in thin section, whether the microscopist is viewing a paste section or an empty space. The ability to distinguish empty space from colorless substances with zero birefringence is the major reason the fluorescence is such a useful complement to the microscopist's repertory of determinative methods. Included in the apparent empty space are those areas that contained water or air before the drying and vacuum impregnation of the concrete specimen with the dyed epoxy. Such features include cracks in both the aggregate and paste, the porosity of the aggregate, and the capillary system of the paste.

The microscopist can, with this one microscope, while examining one particular area, switch back and forth between the fluorescence features and the polarizing features (plane polarized illumination and crossed nicols); thus, it is possible to detect and assess areas of empty space and define their relationship to the reaction products, minerals, rock types, coatings and shapes of the aggregate particles, and other components of the concrete. A feature not readily recognizable in one mode can be easily examined in another. The aggregates, secondary mineralization, and reaction products can be identified and studied with the polarizing capabilities, whereas the areas of empty space and their distinction from some of the hydration products can be determined best by use of the fluorescence of the spaces impregnated by the dyed epoxy.

13.2 USES

13.2.1 Cracks

13.2.1.1 In Aggregate

Because the impregnation with the fluorescent dye precedes the thinning of the section to the point that will create new cracks in the aggregate, it is possible to know whether a particular crack (such as a cleavage crack in aggregate) was indigenous to the specimen or was caused by the processes used in the thinning of the section. If the crack is filled with the fluorescent dye, the crack preceded thinning and can be assumed to be a feature of the specimen that was present before it came to the laboratory.

13.2.1.2 In Paste of Concrete

In concrete, the cracks may go in any direction and often skirt the edges of aggregate particles. Even if the thin section is very thin, there may be no unobscured path along a crack for transmitted light to come through (see Fig. 13-5). The fluorescence of the impregnating epoxy mixture creates light within all the cracks. Even those cracks whose width is below the resolving power of the lenses used may be detected and their location identified. Because of the uncollimated nature of this fluorescent light, the apparent width of these very fine cracks is somewhat increased.

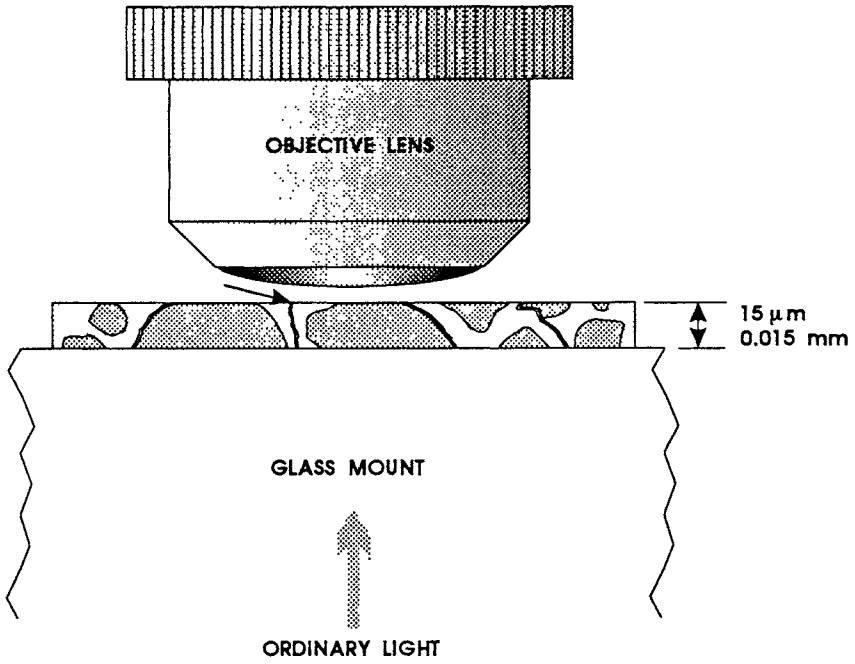


Figure 13-5 CRACKS IN THIN SECTION OF CONCRETE. The *arrow* indicates the only crack that would be visible with transmitted illumination. Impregnation with a fluorescent dye and illumination with ultraviolet light would make possible the observation of the fluorescence of the dye in the other cracks. (The supporting glass slide would be about 100 times the thickness of the thin section of concrete.)

13.2.2 Air-Void Parameters

For laboratory-produced concrete examined at a moderate magnification of about 200X, it was found that air content determinations made on two (more were considered too costly) thin sections of HCC by the fluorescence method had no correlation with the air content determinations made on a finely lapped slice of the concrete by the linear traverse method of ASTM C 457 even though great care was taken to focus on the edges of the voids (see Walker, 1979a). Although there was no correlation in the air-void determinations, a good correlation was found with the determinations of the specific surface and spacing factor. In the original work done by Wilk, Dobrolubov, and Romer (1974), the spacing factor was considered to be the most important parameter to be determined. Because the Virginia specifications for air content are in terms of percentage of air voids, it was decided that this technique did not fill the needs of VDOT.

13.2.3 Hydration

The optical properties of the cement and the empty space in and near a cement grain when compared with similar properties of cement grains of known age and similar size and history will indicate the approximate age and degree of hydration of the cement.

13.2.4 Effect of Fine Aggregate

A smooth-surfaced, sound, fine aggregate (few re-entrant angles, low porosity, and cracks) will produce a concrete mixture that is easy to finish without additional water. An irregularly shaped or porous fine aggregate will create a harsh mixture with a high water demand and prompt the contractor to add more water (see Appendix D; Walker, 1988). By reason of the poor distribution of the cement and the additional water, the concrete may have low strength. The irregular shape and porosity of the poor sand, pockets of excess water, and concomitant clumped distribution of the cement particles may be observed with the P/EF microscope.

13.2.5 Photomicrographs

Photographing the views seen is very useful for demonstrating to a client the characteristics of the specimen concrete. The photographs taken have also been useful in explaining the problems that can occur with certain materials, such as a sand with a high void content or high internal porosity.

13.3 PROCEDURES

13.3.1 General Techniques

1. Examine fluorescent-impregnated ultrathin sections of HCC with the illuminator set for violet light (V settings). Use the accessory BF Y-485. Under these conditions of illumination, the areas of bright fluorescence (empty areas, air voids, and very porous paste) are bright yellow; areas of lower fluorescence (ordinary paste) are greener; and areas of no fluorescence, such as nonporous aggregate particles, are dark blue.

2. Continually check to ensure that the focus of the microscope is in the proper plane. Even sections as thin as 10 μm can be focused on in more than one plane. Keep the focus on the surface of the section that is closest to the objective lens. This plane is the clearest and most free of the circles of confusion caused by nearby out-of-focus objects. Be alert to changes in the focal plane as different thicknesses of the thin section are moved under the microscope. Be aware when objects outside the plane are viewed in an out-of-focus manner. The light from the fluorescence can be so intense that the microscopist may become tempted to register all objects that fluoresce as empty space without checking the plane of focus and ascertaining that the fluorescence is truly from the plane being examined.

3. Make all quantitative determinations (point counting, chord accumulation, and size measurements) on the surface of the section nearest the objective lens (see Fig. 13-6). An object may be seen differently at two different planes of focus (see Fig. 13-7).

13.3.2 Cracks

1. Check all cracks (in aggregate as well as in paste) seen with the polarizing features of the microscope with the fluorescent features to make sure that they contain fluorescent dye.

2. If fluorescence is lacking, change to the polarization mode and check for very faint birefringence and any relief within the crack that differs from that of the mounting medium. Faint birefringence and unexpected relief indicate the presence of a substance filling the crack such as silica gel or another

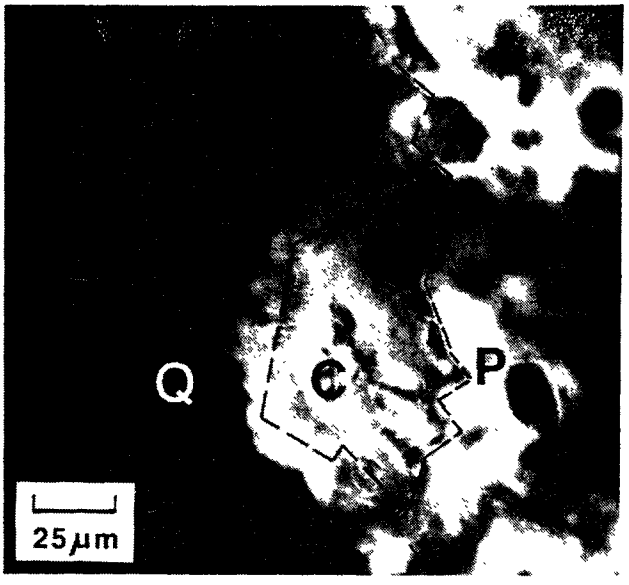


Figure 13-6 FLUORESCENCE FROM POROUS CLAY POCKET SHINING THROUGH EDGE OF QUARTZ PARTICLE. *P* is the paste. *C* is the clay pocket. *Q* is the quartz aggregate. The focus plane is within the section (Walker, 1981).

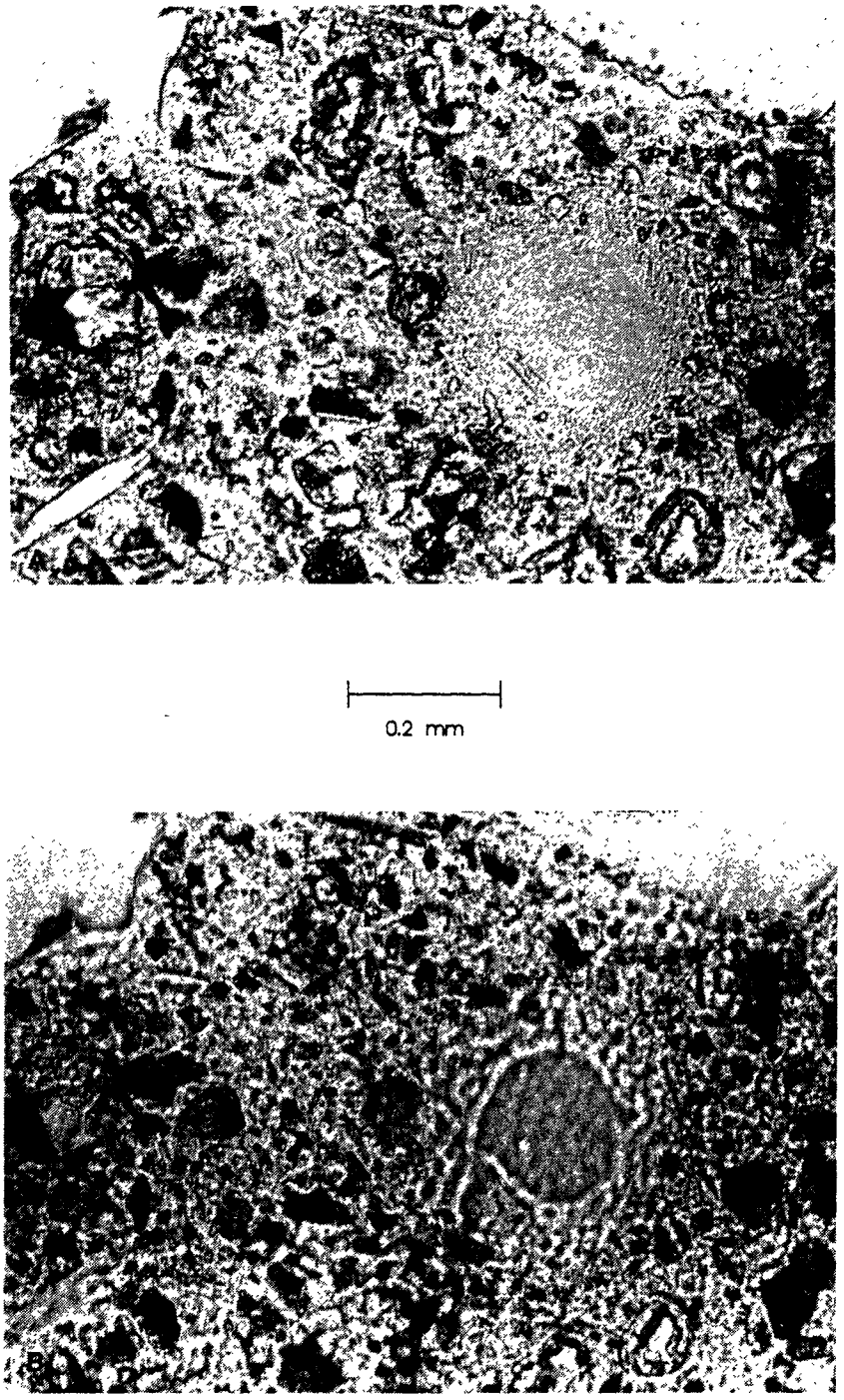


Figure 13-7 VOID IN THIN SECTION. A. Void in thin section with focus plane on top surface of section. B. Same as A with focus plane at bottom of section.

secondary reaction product. If the nonfluorescent crack is really empty, it occurred during thinning of the sample and is not indigenous to the specimen.

3. Check the extent of cracking by examining the specimen with ultraviolet illumination so that the very fine cracks may be observed (see Fig. 13-5). Fluorescence will indicate the fine continuation of the wider, more easily seen cracks and the fine cracks occurring at the boundaries of the aggregate.

13.3.3 Air-Void Parameters

1. When the specific surface, spacing factor, or both are the required data and the specimen is too small to allow a sufficient slice to be prepared or equipment necessary to perform an examination in accordance with ASTM C 457 is lacking, examine a minimum of two fluorescent-impregnated ultrathin sections with ultraviolet illumination and an automatic stage, such as the Swift point counter or similar device.

2. With the automatic motion of the point counter, make traverses across the sections at intervals of 2 mm or greater. The traverses must cover the entire usable area of the thin sections.

3. Record all aggregate, paste, and voids that occur at the points defined by the automatic stage.

4. Continually check to ascertain that the focus is on the near surface of the section and that the edge of the void defining the boundary of the void is at the surface. If the void boundary is beneath the surface, ignore the void and record for the point the substance (aggregate or paste) that occurs on the surface plane of examination.

5. Record the number of voids traversed by use of an accessory push-button counter. Unless the distance between points is so small that the entire section is scanned during step 3, this must be performed during a second pass over each traverse.

6. Calculate the data obtained by the method prescribed in the modified point-count method of ASTM C 457.

7. Unless the sum of the areas of the thin sections, total length of traverse, and number of points counted are at least the minimum prescribed by Tables 1 and 2 of ASTM C 457, do not report the percentage of air voids.

13.3.4 Porosity Related to Carbonation

Study the degree of carbonation and the consequences to the integrity of the HCC by switching back and forth between the polarizing and fluorescence features of the microscope as shown in Figures 13-8 and 13-9. Figure 13-8 shows that carbonation on the exterior of HCC can provide a tighter, less permeable structure than exists below the carbonation. Figure 13-9 shows the high porosity that often accompanies and may be the cause of carbonation in the interior of HCC.

13.3.5 Water-Cement Ratio and Permeability

1. Examine the permeability and water-cement ratio of the HCC by estimating the amount of open area shown by the fluorescence of the dye in

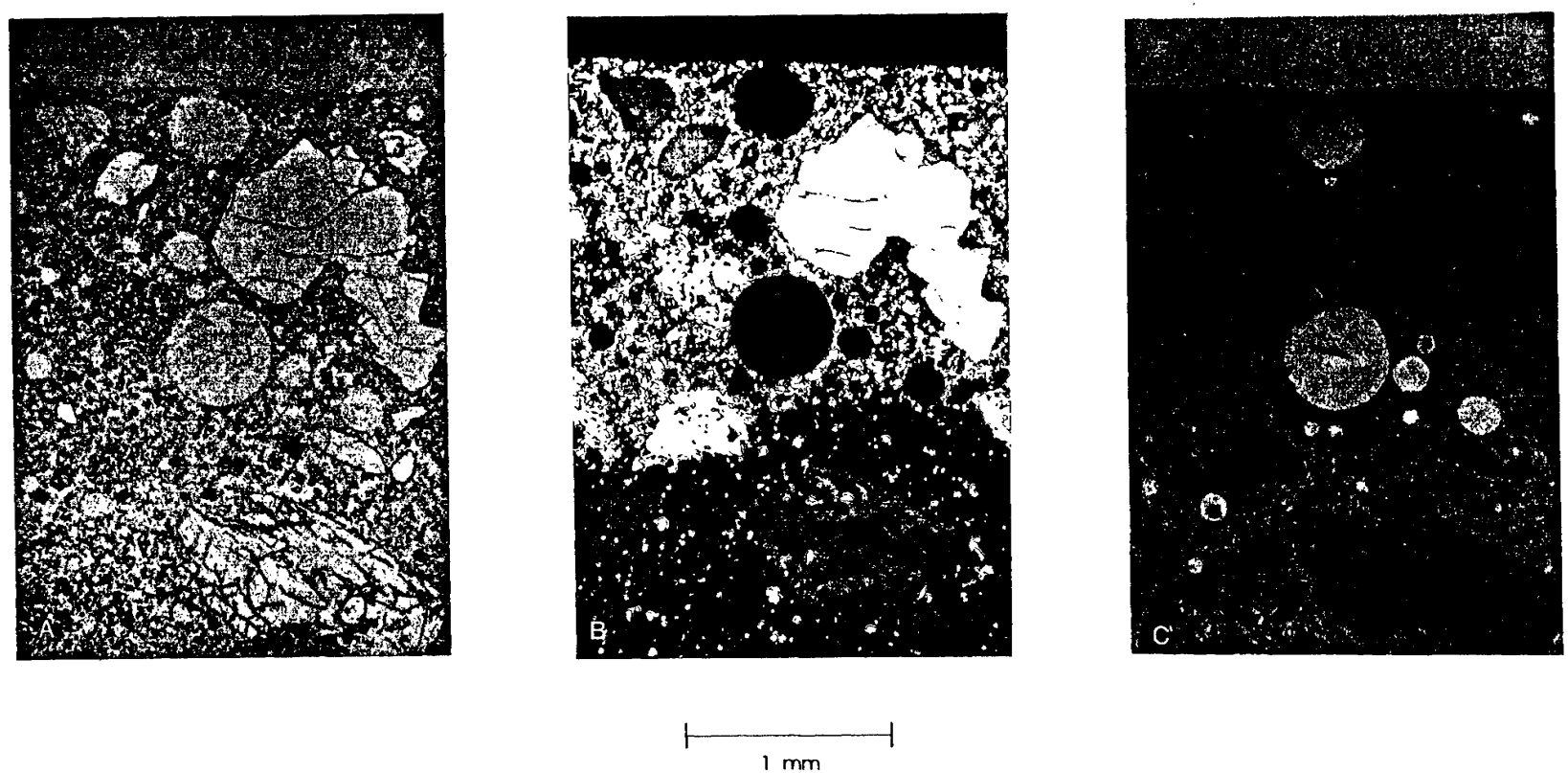
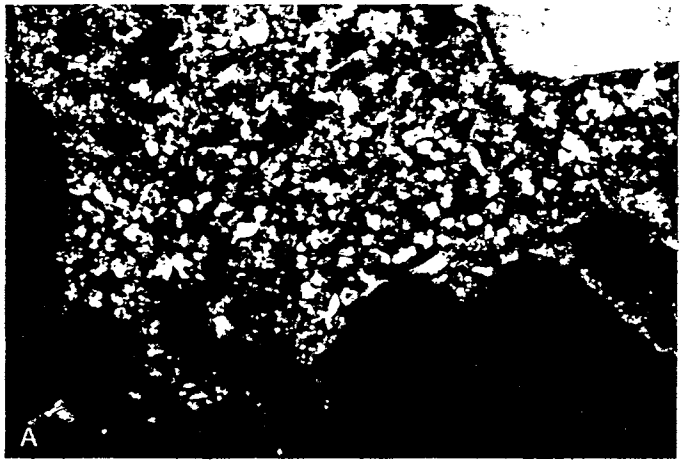
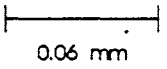


Figure 13-8 THIN SECTION OF EXTERIOR PORTION OF HCC. A. The surface exposed to the air is at the top. **B.** Same view as **A** but viewed with crossed nicols. The bright area shows the high birefringence of the calcite of the carbonated area. **C.** Same view as **A** and **B** viewed with ultraviolet light, causing fluorescence of the pore structure of the HCC. Notice that there is more porosity indicated by fluorescence in the portion of the HCC furthest from the surface than there is in the carbonated zone.





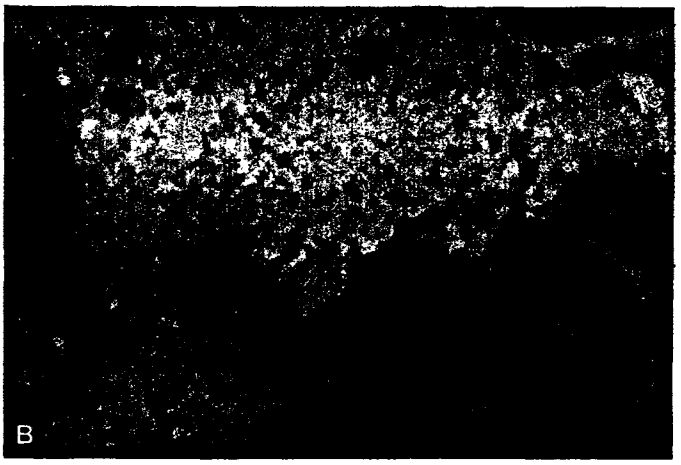


Figure 13-9 THIN SECTION OF INTERIOR PORTION OF HCC. A. Viewed with crossed nicols. The bright area shows the high birefringence of the calcite of the carbonated area. **B.** Same area as **A** viewed with ultraviolet light causing fluorescence of the pore structure within the carbonated area.

the cracks, pores, and capillary system. If the cement particles are congregated in bunches and there are zones of high capillarity around the fine aggregate particles, assume that the fine aggregate had a high water demand and the water that was attracted to the aggregate is now in the paste causing zones of weakness. These methods are best used when there are companion thin-section specimens of high-quality HCC.

2. To compare the permeability of an HCC containing a specific admixture or an aggregate with the permeability of an HCC of known quality, examine fluorescent-impregnated ultrathin sections of both materials. The material with the fewer fluorescent cracks and capillaries is the one that is less permeable and probably has the lower water-cement ratio.

13.3.6 Hydration

1. Examine cement grains with the polarizing and the fluorescence features of the microscope. The center of an only partially hydrated cement particle will still have the birefringence of the unhydrated cement (see Fig. 13-10).

2. Compare the birefringence of the center of the cement grain with that of cement grains of similar size and history. When a cement particle is completely hydrated, the center may become empty or filled with reaction products (see Fig. 13-11). The size of the compared grains is important because the outer hydrated portion of a very large cement grain can protect the inner portion from hydration.

13.3.7 Quality of Fine Aggregate

1. By means of the polarizing features, examine numerous sand particles in fluorescent-impregnated ultrathin sections of the concrete under study. Identify the minerals, and describe the shape of the particles and any coatings on them.

2. With fluorescence-causing illumination, study the sand particles and the paste in which they are imbedded.

3. Examine the effect of the quality of fine aggregate on the surrounding paste. Notice the bond between the paste and the aggregate, any cracking or porosity within the aggregate, and the distribution of the cement particles.

4. Compare with thin sections of an HCC made with a fine aggregate with a known low-void content that has performed well in concrete mixtures. The void content of sand is explained in Appendix E. If lacking good comparative thin sections, draw your conclusions by a study of Figures 13-12 through 13-14, which illustrate the appearance of various qualities of fine aggregate in different forms of illumination.

13.3.8 Photography

1. The best black and white photomicrography is obtained when the BG-12 exciter (B engraving) and the DM 500 with the BF O-515 (B slot position) are employed. When viewed by the eye, the fluorescence is orange-yellow and the aggregates are black; in photographs, the contrast is good but some paste details are lost.

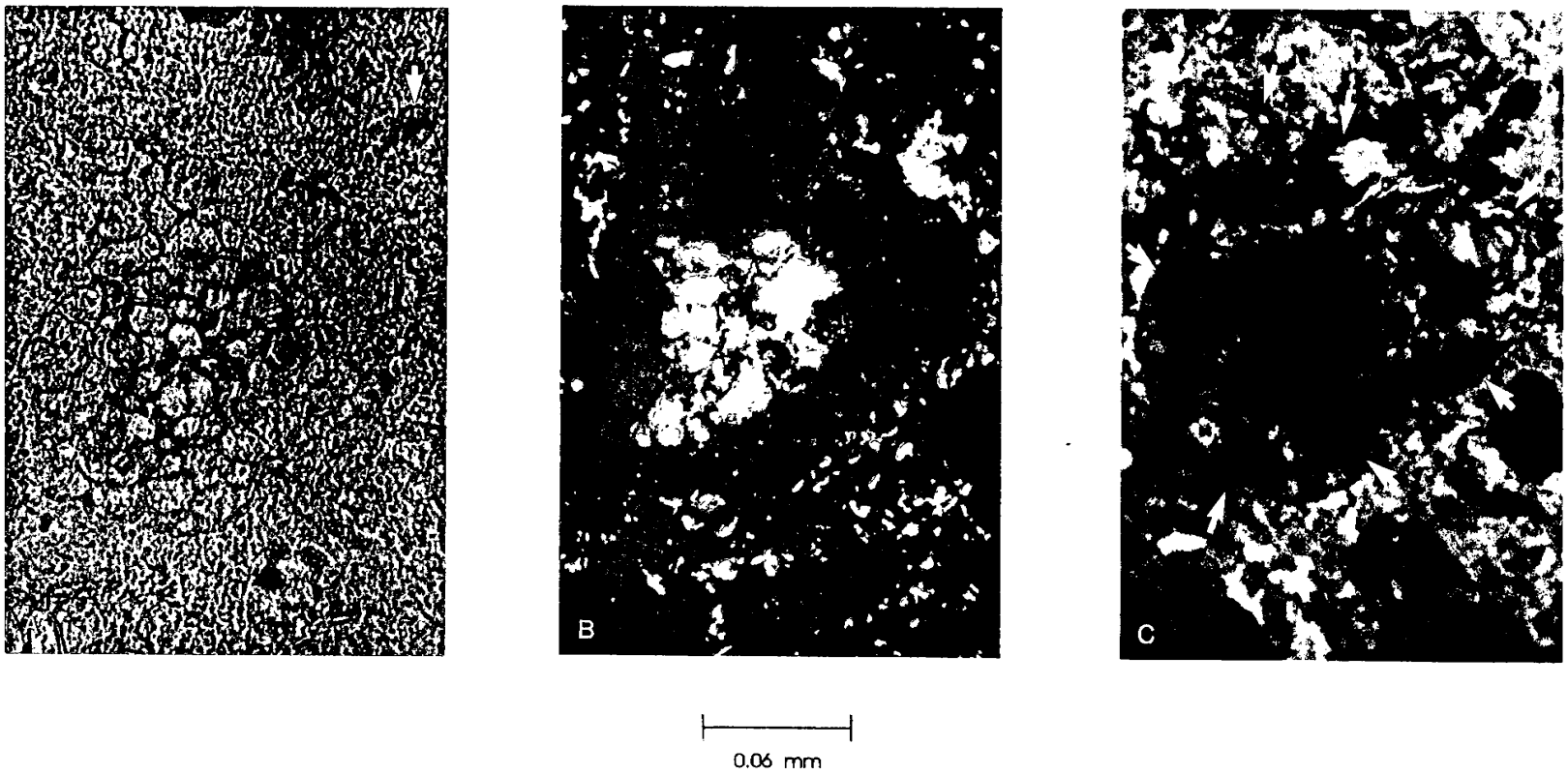
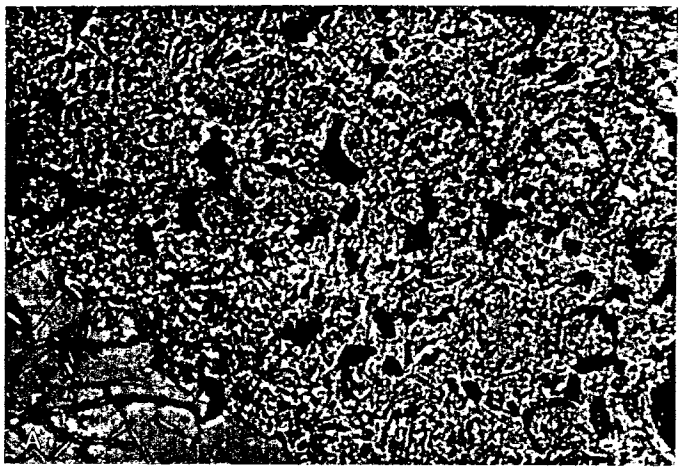
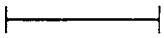


Figure 13-10 THIN SECTION OF 50-YEAR-OLD CONCRETE. At the center is the remnant of a very large cement grain (cement was more coarsely ground then). Modern cement is usually about the size of the completely hydrated and filled cement grain indicated by the *arrow.* **A.** Viewed with plane polarized illumination. **B.** Same location as **A** viewed with crossed nicols. Notice the original birefringence still present in the unhydrated central portion of the grain. **C.** Same location as **A** viewed with ultraviolet light causing fluorescence of the dye in the pore structure. The structure indicates that the original external boundary of the cement grain was the thin line, here indicated by *arrows.*





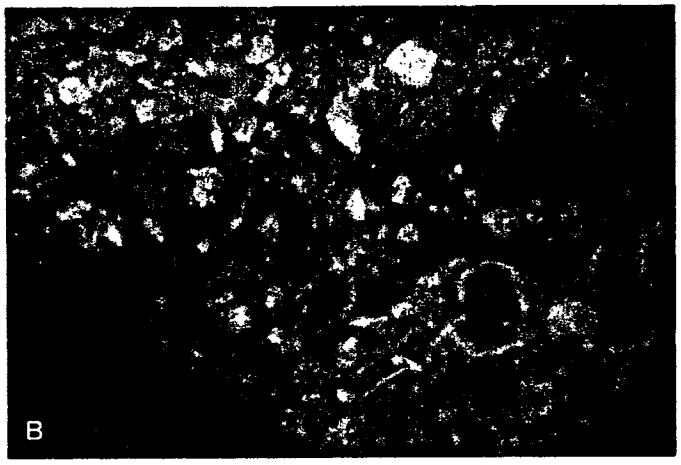


Figure 13-11 THIN SECTION OF 25-YEAR-OLD CONCRETE. The cement grains are completely hydrated. Some cement grain centers are empty; others contain secondary mineralization. **A.** Viewed with plane polarized light. **B.** Viewed with incident ultraviolet illumination causing fluorescence of the dye in empty spaces.

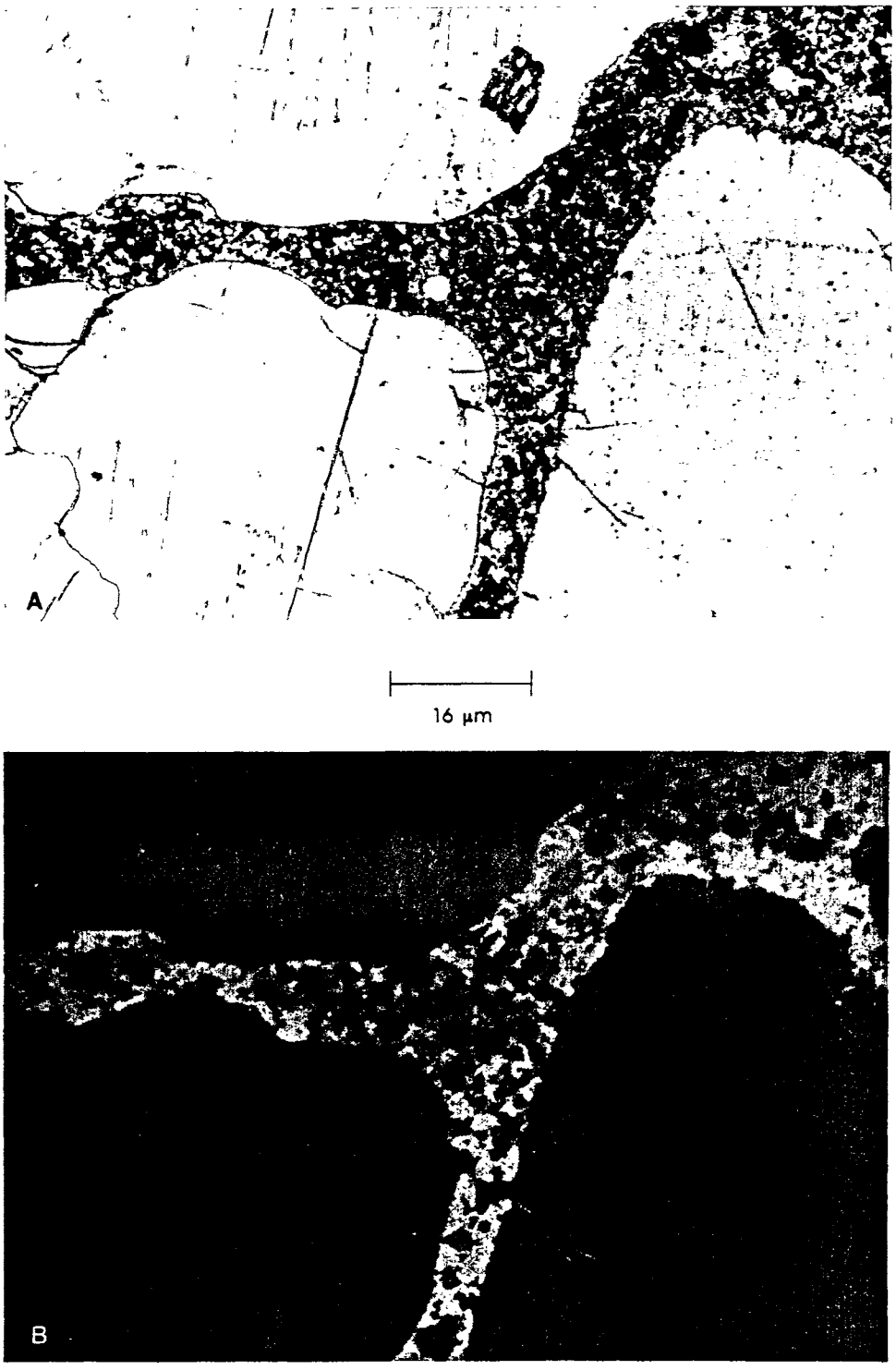


Figure 13-12 THIN SECTION OF HCC FABRICATED WITH SMOOTH, ROUNDED SAND. A. Viewed with plane polarized light. B. Same view as A with ultraviolet illumination causing fluorescence in the pore structure impregnated with dye. Notice the even texture of the paste.

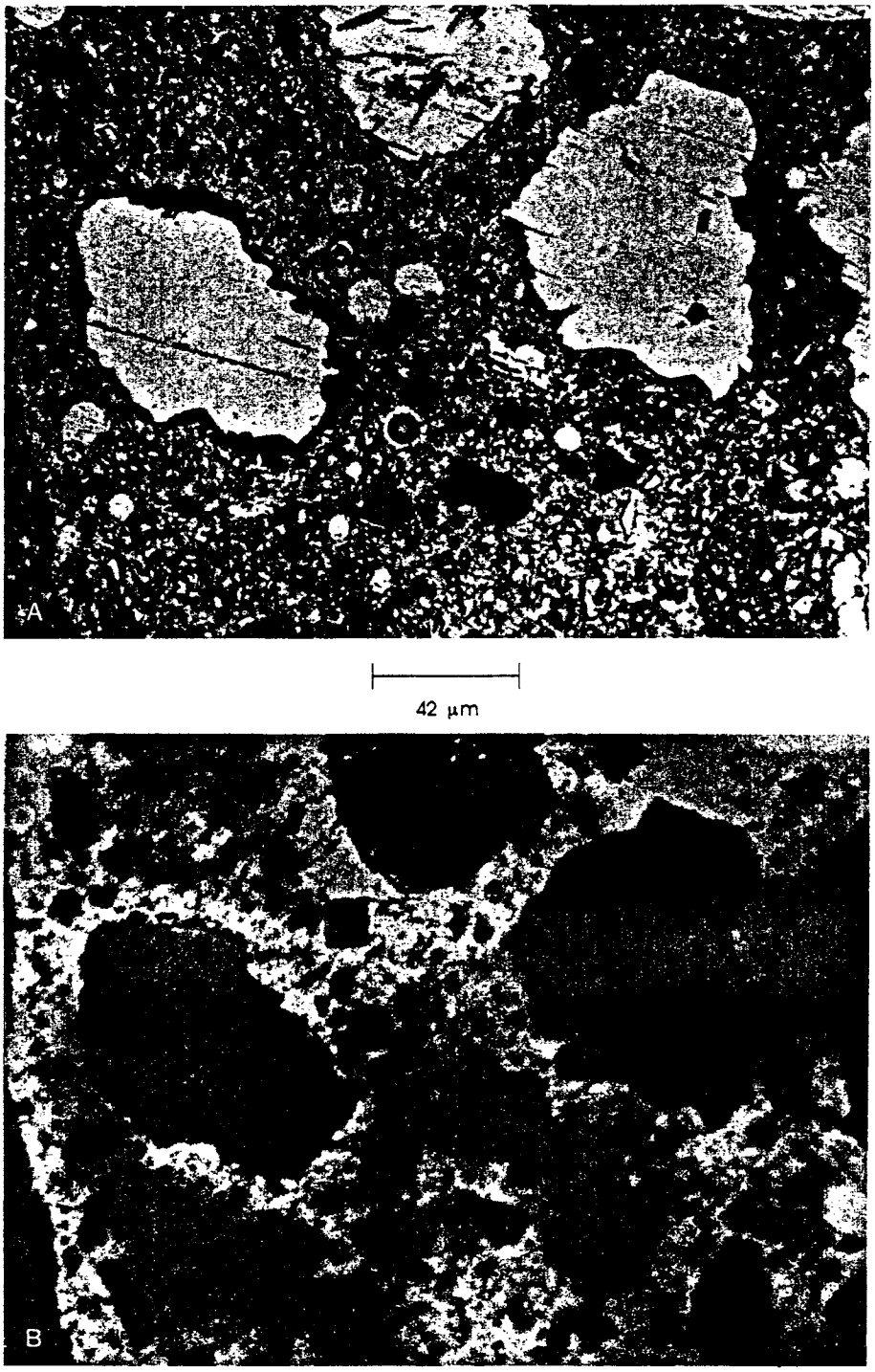


Figure 13-13 THIN SECTION OF HCC FABRICATED WITH ANGULAR, DIRTY SAND. There are numerous reentrant angles. **A.** Viewed with plane polarized light. **B.** Same view as **A** with ultraviolet illumination causing fluorescence that delineates the pore structure. Notice the clumping of the cement grains, abundance of pores (shown by the fluorescence) at the edge of the sand, structure of the clay coatings, and general uneven texture of the paste. Such uneven texture indicates zones of weakness through the HCC.

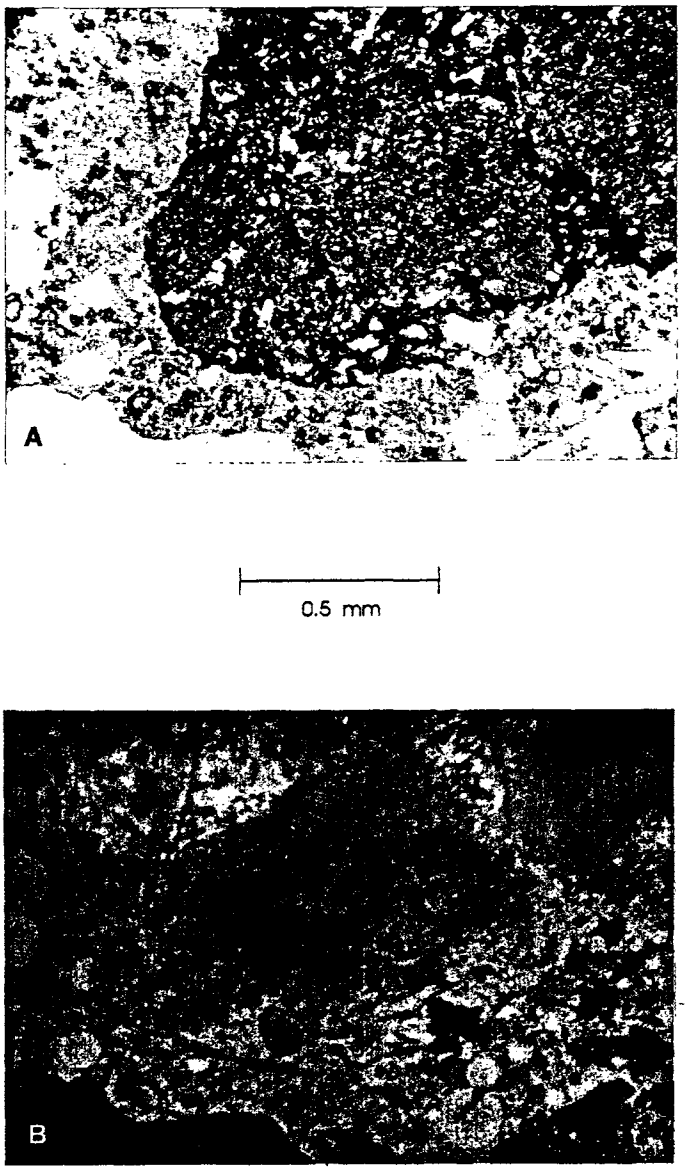


Figure 13-14 THIN SECTION OF POROUS, IRON-STAINED PARTICLE OF SAND. **A.** Viewed with plane polarized light. **B.** Viewed with incident ultraviolet illumination causing fluorescence of the dye in the pore structure of the sand grain and indicating a zone of water accumulation and weakness.

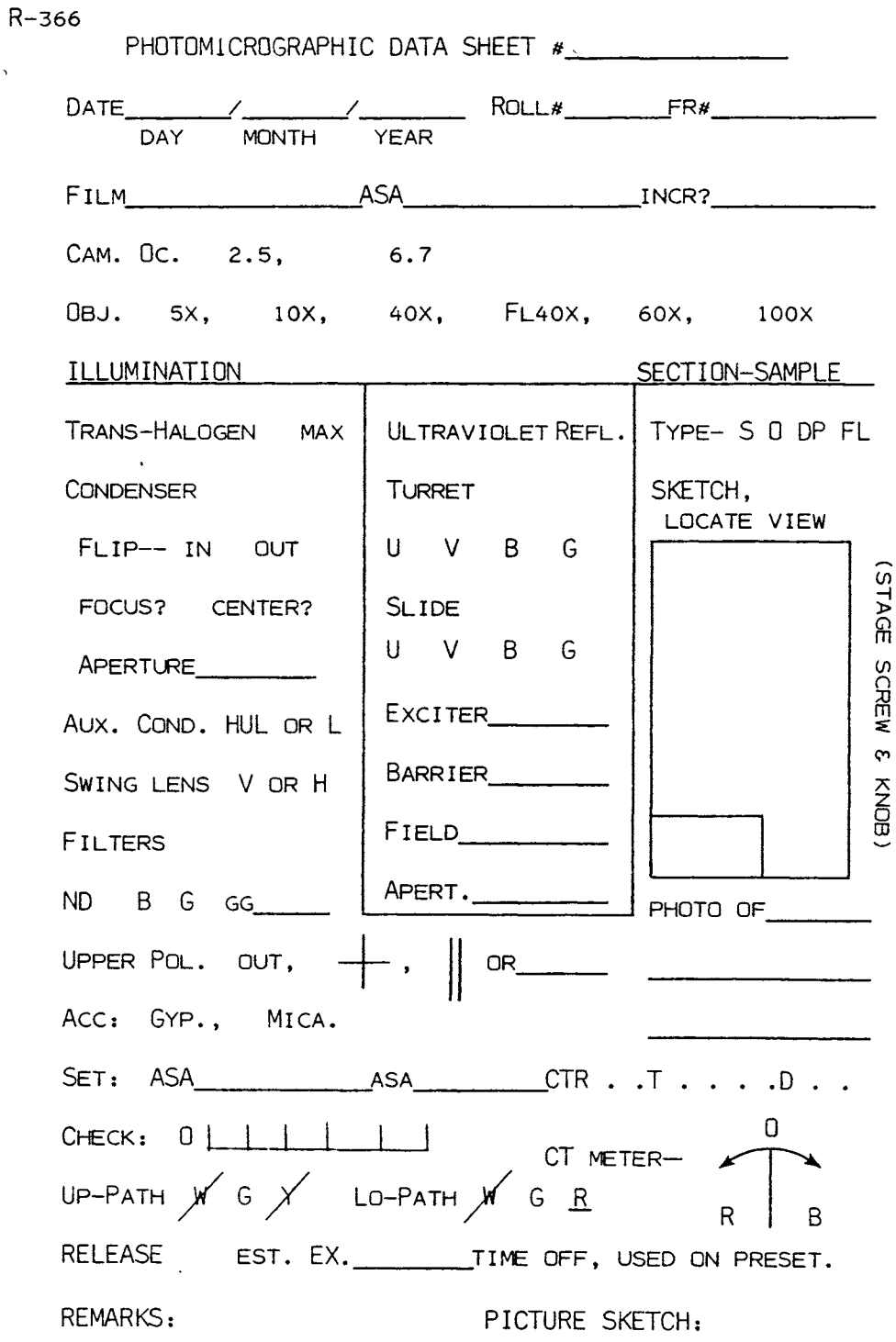


Figure 13-15 PAGE FROM VTRC P/EF PHOTOMICROSCOPY NOTEBOOK

2. Color photomicrography requirements vary with the intensity of fluorescence, but the quality is generally good with the violet settings (BG-3, DM 455, and Y-455) and the addition of Y-485. No transmitted light is used in the photomicrography of fluorescent images.

Completely satisfactory exposures for photomicrography with fluorescence or with crossed nicols have not been obtained. In both cases, it has been necessary to try many different exposure times. When the automatic exposure meter is used, the ability of the equipment to produce the correct exposure seems to depend on whether a bright or a dark object is centered in the view to be photographed. Bracketing the exposure with this light meter is done by setting the light meter for many different ISO numbers and photographing with each.

3. Standardize on a method of keeping track of all photomicrographs taken. Each exposure should have a reference number and be recorded with any data that will aid in improving future exposures. The data recorded should include the source and type of illumination, position of any intensity controls, filters used, amount of opening of all diaphragms (akin to f-stop), film used, and ISO number used to set the exposure meter. If roll film is used, a system of identifying rolls should be devised and this identification recorded with the negatives and on any contact or other archive prints. Figure 13-15 is a page in the P/EF photomicroscopy notebook used at VTRC. The information to be recorded will depend on the nature of the specimen, camera, illumination, and adjustments on the light meter or shutter control.

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READING LIST

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This is the only experimentation in alkali-carbonate reactions that I have heard of in which the reaction took place under restraint. In field situations, expansive reactions are almost always under the restraint of the surrounding concrete and other portions of the structure. Even small members are usually under some restraint. For a report of an alkali-silica reaction under restraint, see Houston, B. W. (1969). *Effects of axial restraint on length change of expanding mortar bars* (Technical Report C-69-8). Vicksburg, MS: U.S. Army Corps of Engineers Waterways Experiment Station.

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ASTM Standards

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- C 33: Standard specification for concrete aggregates.
- C 39: Standard test method for compressive strength of cylindrical concrete specimens.
- C 42: Standard test method for obtaining and testing drilled cores and sawed beams of concrete.
- C 117: Standard test method for materials finer than 75-μm (No. 200) sieve in mineral aggregates by washing.
- C 136: Standard method for sieve analysis of fine and coarse aggregates.
- C 289: Standard test method for potential reactivity of aggregates (chemical method).
- C 873: Standard test method for compressive strength of concrete cylinders cast in place in cylindrical molds.
- D 2419: Standard test method for sand equivalent value of soils and fine aggregate.
- Manual of aggregate and concrete testing (related material).

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 use such standards. These are only a few of the test methods under the jurisdiction of Committee C-9 on Concrete and Concrete Aggregates as published in the *ASTM Annual Book of Standards, Section 4, Construction, Vol. 04.02, Concrete and Aggregates.* Many of the standards in this volume, in addition to those listed here, will be needed for specialized testing of individual materials (such as epoxy rebar coating, fly ash, or ground-granulated blast furnace slag or as they may be referenced in other standards) or for testing individual properties or kinds of concrete. The entire volume should be available for reference.

Cements

Campbell, Donald H. (1986). *Microscopical examination and interpretation of portland cement and clinker.* Skokie, IL: Portland Cement Association.

The best I have seen on cement clinker. Numerous color photomicrographs.

Insley, Herbert, & Frechette, Van Derck. (1955). *Microscopy of ceramics and cements*. New York: Academic Press.

Lea, F. M., & 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.

Winchell, A. N., & Winchell H. (1964). *The microscopical characters of artificial inorganic solid substances: Optical properties of artificial minerals.* New York & London: Academic Press.

D-Cracking

Schwartz, Donald R. (1987). *D-Cracking of concrete pavements* (NCHRP Synthesis 134). Washington, DC: Transportation Research Board.

This is a short, easily absorbed synthesis of the state of the knowledge in 1987 on D-cracking due to failure of carbonate aggregate. There has been much written since but nothing so succinct. For information on D-cracking due to lack of an air-void system sufficient to protect the paste against freezing and thawing, see Andrews, L. E. (1953). *Record of experimental air-entrained concrete 10 to 14 years after construction.* (Highway Research Board Bulletin 70). Washington DC: Highway Research Board.

Paste

Powers, T. C., & Brownyard, T. L. (1948). *Studies of the physical properties of hardened portland cement paste* (Bulletin No. 22: Research Laboratories of the Portland Cement Association). Reprinted from *Journal of the American Concrete Institute*, October 1946–April 1947, Proceedings 43. Detroit.

Powers, T. C. (1962). *Physical properties of cement paste* (Bulletin No. 154: Research Laboratories of the Portland Cement Association). Reprinted from Chemistry of Cement, Proceedings of the Fourth International Symposium, Washington, 1960. Monograph 43, Vol. II, Session V, Paper V-1, pp. 577-609. Washington, DC: National Bureau of Standards.

Petrographic Methods

Beauchamp, R. H., & Williford, J. F. (1974). Metallographic methods applied to ultrathinning lunar rocks, meteorites, fossils, and other brittle materials for optical microscopy. In J. L. McCall and W. M. Muller (Eds.), *Metallographic specimen preparation: Optical and electron microscopy* (pp. 233-250). New York: Plenum Press.

Beauchamp, R. H., Williford, J. F., & Gafford, E. L. (1972). *Exploratory development and services for preparing and examining ultrathin polished sections of lunar rocks and particulates: Final report, Parts I & II* (NASA 9-11993, 211B00862): Revision 1 to NASA Manned Spacecraft Center, Houston, TX 77068. Richland, WA: Pacific Northwest Laboratories, A Division of Battelle Memorial Institute.

Of these two publications by this team of Beauchamp et al., the first is more readily available and has some very instructive photographs but the second (available only from the authors) has much more detailed information concerning the sample preparation procedures used for fabricating thin sections of lunar rock. Some of these procedures were adapted at VTRC for use in the fabrication of thin sections of concrete

[Walker, Hollis N., & Marshall, Bobby F. (1979). Methods and equipment used in preparing and examining fluorescent ultrathin sections of portland cement concrete. *Cement, Concrete, and Aggregates*, 1(1): 3-9].

Bloss, F. Donald. (1961). *An introduction to the methods of optical crystallography.* New York: Holt, Rinehart and Winston.

This work places more emphasis on cataloging mineral species by means of their birefringence than does any other text on optical crystallography or petrography I have encountered.

DeHoff, R. T., & Rhines, F. N. (1968). *Quantitative microscopy.* New York: McGraw-Hill.

Johannsen, Albert. (1968). *Manual of petrographic methods* (Facsimile of the Second Edition, 1918). New York: Hafner.

"The first attempt to give in English a comprehensive review of petrographic methods" (from the dust jacket). It elucidates the optical parameters of crystalline substances and the use of the modern type of petrographic microscope for determining the optical properties of minerals and the principles of light, harmonic motion, lenses, and other related subjects.

Kerr, Paul F. (1959). *Optical mineralogy* (3rd ed.). New York: McGraw-Hill.

Larson, E. S., & Berman, Harry. (1964). *Microscopical determination of the non-opaque minerals* (Geological Survey Bulletin No. 848). Washington, DC: Government Printing Office.

This indispensable work includes chapters on the determination of the optical constants of minerals and, most important, various arrangements of tables of the optical properties. The optical character of the mineral is considered the most important subdivision. Tables are available under each subdivision in order of the indices of refraction of the minerals.

Nicol, W. (1828-9). On a method of so far increasing the divergence of the two rays in calcareous-spar that only one image may be seen at a time. *The Edinburgh New Philosophical Journal*, 6: 83-94 (cited in Johannsen [1968]).

Ray, J. A. (1983). Things petrographic examination can and cannot do with concrete. In *Proceedings of the Fifth International Conference on Cement Microscopy.* Duncanville, TX: International Cement Microscopy Association.

Ray, J. A. (1984). Preparation of concrete samples for petrographic studies. In *Proceedings of the Sixth International Conference on Cement Microscopy.* Duncanville, TX: International Cement Microscopy Association.

Ray, J. A. (1987). Concrete problems associated with air entrainment. In *Proceedings of the Ninth International Conference on Cement Microscopy.* Duncanville, TX: International Cement Microscopy Association.

Wahlstrom E. E. (1955). *Petrographic mineralogy.* New York: Wiley.

Notable for its charts on feldspars and igneous rocks.

Wilk, W., Dobrolubov, G., & Romer, B. (1974). *Development in quality control of concrete during construction* (Report No. TRR-504). Washington, DC: Transportation Research Board.

This work is the source of the idea of using fluorescent illumination to distinguish true void areas from the areas of the paste that have little or no birefringence. Thus fluorescence can be used to locate and enhance microcracking and zones of high capil-

larity. This report has many other ideas in it than just the petrography performed with fluorescent-causing illumination, for example, quick testing for resistance to freezing and thawing and a numerical scale by which to grade each concrete.

Reference Collections

American Concrete Institute. (Revised yearly). *ACI manual of concrete practice.* Vols. 1–5. Detroit.

This manual is a very important reference and should be available to all concrete technologists.

ASTM Committee on Concrete and Concrete Aggregates, C-9 (Ed.). (1978). *Significance of tests and properties of concrete and concrete making materials* (ASTM Special Technical Publication No. 169B). Philadelphia.

This worthwhile work is a revision and expansion of ASTM Special Technical Publication (STP) No. 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. STP 169C is being prepared.

ASTM Committee on Performance of Concrete—Chemical Aspects, C-9.02.02 (Ed.). (1965). *A symposium on alkali-carbonate rock reactions* (Highway Research Record No. 45). Washington, DC: Highway Research Board.

National Bureau of Standards. (1962). *Chemistry of cement: Proceedings of the Fourth International Symposium* (Vols. 1 and 2). (NBS Monograph No. 43). Washington, DC.

This entire monograph is a classic work and includes discussions of many of the papers. Volume 2 is mainly concerned with alkali-silica reactions.

Transportation Research Board. (1974). *Cement aggregate reactions* (Report No. TRR-525). Washington, DC.

Terminology

ACI. 116R: Cement and concrete terminology. In *ACI manual of concrete practices: Part 1, Materials and general properties of concrete.* Detroit.

ASTM. C 125: Standard terminology relating to concrete and concrete aggregates. In *Annual book of ASTM standards: Volume 04.02, Concrete and aggregates.* Philadelphia.

Bates, Robert L., & Jackson, Julia A. (Eds.). (1987). *Glossary of geology* (3rd. ed.). Alexandria, VA: American Geological Institute.

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Appendix A
GLOSSARY

Acid-igneous rocks. Igneous rocks that are rich in quartz and the potassium-sodium feldspars. May contain mica, amphibole, etc. Examples are granite and granodiorite. These rocks are often light colored and/or mottled. It is common (except in extrusive volcanic rocks) for the individual grains to be large enough to be easily distinguished by the unaided eye.

Alkali-aggregate reaction. Any potentially expansive chemical reaction between the aggregate and the hydroxide ions associated with the ions of sodium and potassium in solution in the paste. Can be very deleterious (see **alkali-carbonate reaction** and **alkali-silica reaction**).

Alkali-carbonate reaction. A potentially expansive chemical reaction between a carbonate aggregate and the hydroxide ions associated with the ions of sodium and potassium in solution in the paste. The only rock known to so react is an impure dolomitic limestone with a specific internal structure. Can be very deleterious if the forces generated by the reaction exceed the cohesive forces of the concrete. See Chapter 10.

Alkali-silica reaction. A potentially expansive chemical reaction between siliceous aggregate and the hydroxide ions associated with the ions of sodium and potassium in solution in the paste. The siliceous rocks involved in this reaction are those with an imperfect crystal structure or those that are not crystalline. Can be very deleterious if the forces generated by the expanding silica gels exceed the cohesive forces of the placement. Deteriorated HCC often exhibits oozing silica gels or the dehydrated and carbonated remains of silica gels. See Chapter 10.

Amorphous. Not crystalline; without a regular arrangement of the component atoms into a crystal structure.

Analyzer. In a petrographic microscope, a device (located above the objective) that polarizes the light after it has passed through the specimen.

Anhedral. Without bounding crystal faces. Cf. **euhedral, subhedral.**

Basic-igneous rocks. Igneous rocks that are rich in the sodium-calcium feldspars, with little or no quartz; often abundant olivine and pyroxenes. Examples are basalts and diorites. These rocks are often very dark colored and fine grained.

Becke line. In the microscopical test for the relative index of refraction, a bright line that is most visible while in motion as the focus is changed. It is the projection of the boundary of two substances of different indices of refraction. If the match between the indices is close, the line may be refracted into rainbow colors (unless monochromatic light is used). As the objective lens is raised from the subject, the Becke line appears to move toward the center of the substance with a higher index of refraction. If the line is refracted into colors, the position (movement) of the rusty red color is taken as the true position.

Beneficiation. The process of improving the chemical properties, physical properties, or both of an ore or other earth material for use in a manufacturing process or as a construction material.

Bertrand lens. A removable plano-convex lens on a slide located in the tube of a petrographic microscope above the upper nicol (analyzer); used with convergent light from the condenser to form interference figures and thus determine the number of optic axes and the optical sign. The focusable, diaphragmed Bertrand lenses are the most convenient.

Binocular. Pertaining to optical equipment. Allowing the use of both eyes together either to permit the sensation of relative distance (when two objective lenses are furnished) or provide a more natural method of viewing the subject. In modern petrographic microscopes, binocular viewing is present to relieve eye strain and cannot provide a sensation of depth. In many microscopes, the prisms necessary to split the image for use by both eyes may make determinations of the properties of the optic axis figures nearly impossible.

Biotite. A dark-colored mineral of the mica group that exhibits excellent platy cleavage; in thin section, a bird's eye texture and a birefringence of 0.033.

Birefringence. The numerical difference in the index of refraction between two rays of light separated from one light source by the properties of the substance. Birefringence can be observed when a nonisotropic substance is placed between crossed nicols. The diffraction colors seen are a product of the birefringence of the substance and its thickness. Also called *double refraction*. Low birefringence is gray though pale white, 0.005; moderate birefringence includes the first order colors up to 0.030; all above 0.030 is high to very high.

Birefringent. The property of having at least a minimal birefringence.

Bleed water. The water produced by bleeding (q.v.). The water can be seen as a wet sheen on the surface of the concrete. When abundant, it can form puddles.

Bleeding. In the hardening of concrete, a process whereby the solids of the HCC, including the cement and other fine particles, settle and water rises to the top. (Has been thought to be caused by syneresis but is probably almost entirely due to settlement.)

Calcite. Calcium carbonate, $CaCO_3$, a common mineral and a major ingredient of limestones and certain other sedimentary rocks. Often occurs as a vein mineral. Calcite is colorless or white when pure, crystallizes in the rhombohedral class, has a hardness of 3 on the Mohs scale, and dissolves with the effervescence of carbon dioxide in dilute hydrochloric acid. The transparent forms of calcite exhibit excellent double refraction that can be observed in thick slices without lenses or polarizers. The carbonates have a very high birefringence; calcite = 0.172. Cf. **dolomite.**

Carbonation. The process in portland cement concrete and in other HCCs where the calcium ions in solution or in the calcium silicate hydrates react with the carbon dioxide of the atmosphere, in the presence of moderate humidity, and become changed to calcium carbonate with impurities such as amorphous silica. On the surface of HCC, the production of calcium carbonate by this method can provide a tough surface. In the interior, it can indicate weakness; passageways for water, air, and CO_2 to enter the paste; and lowered pH of the paste, thus making the steel more susceptible to corrosion. See **depassivation.** (Not to be confused with the carbonation of soda water or the coalification of organic materials.)

Cold joint. A joining between two adjacent placements of HCC material where the earlier placement had hardened when the second was placed. May indicate little or no bond between the two concretes.

Collimate. When pertaining to light rays, to make parallel.

Conchoidal. Said of a fracture surface that is made up of smoothly curved, shell-like surfaces. True of quartz and obsidian and other massive glasses.

Conoscopic lenses. The lenses used to form interference figures. See **Bertrand lens.**

Core. When pertaining to concrete testing, a specimen cut from a hardened placement or a large block of concrete with a diamond core drill for use as a portion of the material for various testing procedures. It is preferred that the core drill have an internal diameter of at least 4 in. The core should be as deep as the placement is thick so that the entire thickness of the placement is sampled. When coring bridge decks, a small thickness of concrete is often left in the bottom of the drill hole to prevent the core from falling through the bridge and provide a bottom for the patching material. A core can be distinguished from a cylinder by the diamond-cut exterior curved surface on which cut aggregate particles will normally be exposed.

Crazing. A fine very shallow cracking that occurs in the exposed surface of concrete. It may be due to shallow freezing or minor curing flaws. Crazing often has a very fine pattern, like pattern cracking except the individual uncracked central portions are usually less than 2 in. across. Crazing may develop to the point where the surface flakes off to a depth of about 1/8 in.

Crossed nicols. The microscopical condition of having the analyzer and the polarizer in the system with their vibration directions at 90° to each other.

Crystallite. A tiny crystal, often acicular and sometimes too small to exhibit birefringence.

Cubic. Used with reference to the crystal structure of a substance to indicate three crystallographic axes of equal length and mutually perpendicular. Often referred to as an isometric crystal structure.

Cylinder. When pertaining to concrete testing, a specially cast cylindrically shaped specimen of the concrete being fabricated to provide portions of the concrete for various testing procedures. In Virginia, the cylinder is usually 4 in. in diameter and 8 in. in height. A cylinder may be distinguished from a core by the fact that the curved surface was cast in a mold with a smooth surface and may show marks of the seams of the mold.

Dedolomitization. Any of several processes in which the dolomite is removed or replaced from within a solid rock; may take place by ionic solutions in the interior or be particularly active on the rock surface. Especially used for the replacement of dolomite by calcite.

Depassivate. The process of neutralizing or removing the passivation on the surface of a metal. In concrete, usually used to mean the lowering of the pH of the material surrounding the reinforcing bars so that the passivating (protective) effect of the high pH of the paste is no longer present.

Design of the mixture. The specified proportioning of the ingredients of the concrete mixture; the document giving this information. The document should indicate the source of each of the ingredients approved for the concrete in question.

Diffraction. The process of bending light as it passes from one medium to another. Examples are the opponent bending of a straight object at the water line and bending of light by a prism.

Diffraction colors. Those colors caused by the fact that the different colors of light bend at different angles. Examples are rainbow colors and the colors produced by a prism.

Dolomite. Calcium-magnesium carbonate, $CaMg(CO_3)_2$, a common mineral in sedimentary rocks such as limestones and dolostones. Much like calcite but slightly harder and less soluble in acid. The chemical composition grades toward ankerite, with a substitution of iron and manganese for part of the magnesium. The carbonates have a very high birefringence; dolomite = 0.185. Cf. **calcite, dolostone.** Dolomite is sometimes used to mean a rock composed predominantly of the mineral dolomite.

Dolostone. A rock much like limestone in appearance but composed mainly of the mineral dolomite. Frequently, the rock is called dolomite.

Effervesce. To bubble, splatter, etc. by the emission of a gas due to chemical action. *Example:* $CaCO_3$ when acted on by acid.

Efflorescence. A surface encrustation caused by the evaporation of solutions seeping out onto the surface of rock or concrete.

Euhedral. Completely bounded by its own regularly developed crystal faces. Cf. **anhedral, subhedral.**

Exudation. Something oozed out, usually a fluid solution.

Feldspar. A mineral of one of the two major feldspar groups: the potassium-sodium group and the sodium-calcium (plagioclase) group. The feldspars are aluminum silicates and are common in low-temperature veins and in all types of igneous rocks. They are monoclinic or triclinic, have two optic axes, and range in color from colorless through various pastels to dark gray. They have nearly perfect cleavage in at least two directions at close to 90° to each other. They are often euhedral or subhedral even when intergrown with other minerals and each other. The birefringence is moderate, generally between 0.006 and 0.011. They can be recognized by their cleavage and intergrowths.

Grading. Said of an aggregate and used to describe the distribution of the sizes of the particles therein. *Well-graded* aggregate has a large variety of particle sizes and fills a space well. *Poorly graded aggregate, or gap-graded aggregate,* has particular sizes missing. When used by a geologist, *good grading* indicates a definite sorting of grain sizes and *excellent grading* indicates that the various grain sizes are well separated and deposited in different portions of the sediment.

Greenschist facies or greenstone facies. The stage of metamorphism in which greenstones occur. The rock is rich in chlorite, actinolite, epidote, or all three, occasionally with significant quantities of feldspars and quartz. Cf. **greenstone.**

Greenstone. A compact rock formed by the metamorphism of basic to ultrabasic igneous (sometimes volcanic) rock. The metamorphism has proceeded to the greenschist facies.

Groundmass. The phase of a rock that is so fine grained that individual minerals cannot be identified. Commonly said of the interstitial material surrounding the larger crystals in an igneous rock or of the unidentifiable mudlike matrix of a sedimentary rock.

Hexagonal. Used with reference to the crystal structure of a substance to indicate three crystallographic axes, of equal length, at 120° to each other and all perpendicular to a fourth axis.

Hornblende. A dark-colored mineral of the amphibole group of monoclinic silicates, all of which exhibit cleavage parallel to the long axis at about 56° and 124°. Birefringence is about 0.023. Occurs in acid-igneous rocks.

Igneous. Used to indicate rock that has formed by cooling or the escape of fluids from molten portions of the earth's crust either at or near the surface or at great depth. Examples are granites, lavas, and diorites. They occur in massive formations, flows, veins, dikes, and sills. The term includes those vein rocks that are formed from hydrothermal, ion-rich fluids and that produce a large variety of minerals and often gigantic crystal sizes (i.e., pegmatites). On occasion, used to indicate rock (which may be truly metamorphic) that seems to have the characteristics of igneous rock.

Index of refraction. The ratio of the speed of light in a vacuum to the speed of light in the substance under consideration.

Index of refraction oil. Immersion oil used as a standard medium to which to compare subject substances. Prepared sets of such oils are commercially available. Because the index of refraction of these oils may change with time, they should be periodically checked using a refractometer or solid standards.

Insoluble impurities. The substances found in the interstices of a carbonate rock material that cannot be dissolved by warm, dilute hydrochloric acid. These substances may include clay, quartz, micas, feldspars, and pyrites.

Interference color. The highest order of color seen in a mineral when viewed with the petrographic microscope with crossed nicols. The highest colors will be seen when the two crystallographic axes that have the greatest difference in refractive power are parallel to the stage and when the axes are at 45° to the nicols. The interference color is a product of the birefringence and the thickness of the substance. Charts of the interference colors are available in most books on the use of the petrographic microscope. These charts show 0.000 birefringence as black, increasing to gray, white, yellow, and orange, through the spectrum. When the spectrum starts to repeat, the light is more brilliant and the color bands are increasingly blurred. In about the sixth repeat (the sixth order), the light is a brilliant white in which no specific color can be distinguished. This is the typical appearance of the carbonate minerals at 30 mm (0.030 mm; 0.0018 in.) in thickness. In a random view of any mineral, the interference colors seen will depend on the thickness, angle of the viewing, and angle to the polarization of the nicols. Some particles will be banded from very low to the highest color produced by the substance. Other particles of the same substance when viewed with only equivalent crystallographic axes parallel to the stage will show almost no interference and will be seen as nearly black.

Interference figure. A pattern of light and shadow and bands of interference colors produced on the back lens of the objective in the petrographic microscope by the use of the accessory convergent lens in the condenser. The figure can be viewed as projected on the back lens surface or with the Bertrand lens. The interference figure provides data concerning the crystal structure and the spread of the optic axes. With the gypsum plate or quartz wedge accessories, the optic sign of the mineral can be determined.

Isometric. The crystallographic or optical property of a substance of having three crystallographic axes of equal length at mutual right angles. An isometric mineral has no birefringence unless it has been distorted or subjected to stress.

Isotropic. The property of being crystallographically the same in all directions. Includes isometric and amorphous.

Laitance. A layer of weak material containing cement, calcium hydroxide, and aggregate fines brought to the surface of concrete by bleed water. The amount is increased by a high water-cement ratio, overworking, or improper finishing (see ACI 116).

Lapping. The process of producing a smooth surface by grinding away projecting portions.

Limestone. A common sedimentary rock mainly composed of the mineral calcite, $CaCO_3$. Includes the lithified chemically deposited benthonic (deep water, bottom) type as well as the indurated collections of shells and fragments of shells. Usually restricted to meaning only oceanic rocks but can include the fresh water limestone, travertine, as well. May be restricted to rocks mainly composed of calcite, but is sometimes used to include the other carbonate rocks, especially dolostones.

Lithology. Rock type, including mineralogy, structure-intrinsic parting, fissibility, and grain size.

Macrocrack. Cracks that can be seen with the unaided eye (often large enough to be seen when one is riding over a pavement or bridge).

Metabasalt. A basalt, fine-grained, basic igneous rock, often extrusive (volcanic lavas, near surface sills and dikes, etc.), that has undergone metamorphism. Will frequently include beds of metamorphosed volcanic debris such as ash, lapilli (pebble sized), and bombs. See **greenstone.**

Metamorphic. Pertaining to a rock formed by the processes of metamorphism. The source rock material may be sedimentary, igneous, or an earlier metamorphic.

Metamorphism. A natural process that, over the stretch of geologic time, can transform a rock from one appearance, crystal structure, and composition to another. The process can include the stresses of the folding and faulting crust, subduction at the edges of crust plates, pressures of burial at depth, and recrystallization and chemical changes caused by the various solutions and temperatures found in the earth's crust. The word *metamorphism* includes the low-grade, low-temperature processes by which fluids can rearrange the ions, atoms, and minerals of a rock to form new minerals and recrystallize minerals in new forms.

Micrite. The very fine-grained crystalline component of limestones that is thought to have been deposited as a chemical precipitate, calcium carbonate, and is considered to be a lithified ooze. The individual particles are completely anhedral and generally less than 4 μm in diameter.

Microcrack. Cracks that cannot be seen clearly seen or measured without magnification. They exist on all concrete surfaces but are extremely difficult to see on rough surfaces.

Modal (from classical petrology). Used with *analysis* or *determination* to indicate the data collection and calculations necessary to make a mathematical determination of the relative abundance of the various component solids or discrete substances that make up the whole. There is no implication of the determination of the three-dimensional spatial arrangement, shape, and size of the individual portions of the phases. When the determination is made optically, from points, chords, or areas on a plane through the substance, the results will be in percentage by volume.

Monoclinic. Used with reference to the crystal structure of a material to indicate three crystallographic axes of not necessarily equal length: two are mutually perpendicular, and the third is not.

Monocular. Pertaining to optical equipment. Allowing the use of the equipment by one eye only.

Nicol or nicol prism. In a petrographic microscope, a device that polarizes light. The lower nicol (located in the substage) polarizes the light before it reaches the specimen. The upper nicol (located above the objective) polarizes the light after it has gone through the specimen.

Nonisotropic. The property of not being the same in all directions. Used especially in reference to mineral substances and usually implies that the substance is at least slightly birefringent.

Ocular. Pertaining to optical equipment. The lens assembly nearest the eye.

Optic sign. An optical property of birefringent crystal substances. It is one of the properties by which substances can be classified and identified. Refer to **interference figure.**

Orthorhombic. Used with reference to the crystal structure of a substance to indicate three crystallographic axes of unequal length and mutually perpendicular.

Passivate. To render the surface of a metal chemically inactive. In concrete, passivation of the surface of the reinforcing bars is produced by the high pH of the cement paste.

Paste. The portion of a concrete that is not aggregate.

Phase (as used in chemistry). A distinct, mechanically separate component of a heterogeneous whole. The term may or may not refer to the physical state of the component (i.e., gas, liquid, or solid). Also used with *analysis* or *determination* in the same sense as *modal.*

Phenocrysts. The relatively large conspicuous crystals, surrounded by groundmass, finer crystals, or natural glass (such as obsidian). Found in certain igneous rocks.

Pleochroic properties. The property of a mineral such that it exhibits different colors when viewed at different angles, particularly in plane polarized light. A useful identifying property particularly well exhibited in the minerals biotite, hornblende, and tourmaline.

Polarizer. In a petrographic microscope, a device (located in the substage) for polarizing the light before it reaches the specimen.

Pozzolanic materials. Materials that combine with the lime in cement paste to produce cementitious calcium silicate hydrates and can react with the hydroxide ions associated with the ions of sodium and potassium in solution in the paste during the early stages of hydration and sequester these ions in gels that, because of their location or composition, cannot cause deleterious expansion; includes natural pozzolans, fly ash, silica gel, and GGBFS.

Pyrite. A stable, common mineral of the pyrites group, iron sulphide, FeS_2; it is opaque, with a golden metallic color and conchoidal fracture. Crystallizes in the cubic system with striated faces; common habits are the cube and pyritohedron. Com-

mon in all metamorphic rocks, even those of very low-grade metamorphism. See **pyrites.**

Pyrites or pyrite group. A group of common, opaque, iron sulphide minerals; stability and metallic color depend on the ratio of iron to sulphur. Black in color when occurring in an extremely finely divided state. The group includes pyrite, pyrrholite, and marcasite. The black variety is common in very dark-colored limestones, shales, and slates. See **pyrite.**

Quartz. A common mineral, silicon dioxide, SiO_2; crystallographically rhombohedral, colorless when pure, a major constituent of the rocks of the earth's crust. On the Mohs hardness scale, it is number 7. Occurs in six-sided prisms with pyramidal terminations in veins and in anhedral masses in almost all acidic rocks. A major constituent of most beach and river sands. Cleavage is usually visible only in very thin sections. The birefringence is moderate: 0.009.

Refraction. The change in a light wave as it passes into a substance of different density or with different optical properties. Related to diffraction but includes the splitting of a light wave into two components that travel at different speeds and whose vibration directions are at right angles to each other. Examples are the apparent bending of a straight object at the water line, the bending of a light wave by a prism, and the double refraction of crystals of substances such as calcite. See **index of refraction.**

Relief. The optical property of the degree of contrast microscopically observed, caused by the difference in index of refraction between a substance and the medium with which it is surrounded. High relief appears as a rough surface; low relief appears as a smooth surface that is hard to distinguish from the background.

Rhombohedral. Used with reference to the crystal structure of a crystalline substance to indicate a subset of the hexagonal crystal system in which the symmetry is incomplete. The two end terminations may differ, and alternate (thus opposing) prism faces may have different textural characteristics.

Screeding. The process of leveling and smoothing concrete to prepare it for the final surface texture. Often combined with vibration as part of the consolidation process.

Sedimentary. Used to indicate rock that has formed by the collection and usually the induration of materials in solution and particles derived from other rocks by the forces of weathering, gravity, running water, etc. The induration may be purely chemical and can take place on the surface or may be due to compression and solutions at depth. Cf. **metamorphism.** The source rocks may be sedimentary, igneous, or metamorphic.

Siltstone. A very fine-grained consolidated rock, the particles of which are predominantly between 1/16 and 1/256 mm across and have been removed from other rocks and transported by wind or water to the place of consolidation.

Stalactite. A cylindrical or conical deposit of solids caused by the evaporation of solutions dripping from an overhead structure such as a cave roof or bridge.

Subconchoidal. Said of a fracture surface that is nearly but less than perfectly like a conchoidal surface. Cf. **conchoidal.**

Subhedral. Incompletely bounded by its own regularly developed crystal faces. Cf. **anhedral, euhedral.**

Tetragonal. Used with reference to the crystal structure of a substance to indicate three crystallographic, mutually perpendicular axes, two of which are of equal length.

Tourmaline. A mineral of a complex silicate group of minerals that contain boron and are crystallographically rhombohedral. They occur in striated columnar crystals in many different colors. They may be found in acid-igneous rocks and veins. The birefringence of the minerals of the tourmaline group is about 0.020.

Triclinic. Used with reference to the crystal structure of a substance to indicate three crystallographic axes, of not necessarily equal length, with no restrictions on the included angles.

Appendix B
OBTAINING SPECIMENS OF HCC FOR PETROGRAPHIC EXAMINATION

B.1 OVERVIEW

The petrographer can examine any specimen(s) of concrete the client wishes to submit for his or her scrutiny, but unless the petrographer has been informed of any problem(s) the concrete has developed and how the specimens were collected relative to the location of any problem areas, the examination may not yield any meaningful information. Complete documentation describing the placement and any problems concerning it must accompany the specimens.

Unless samples of concrete are obtained according to a statistically based sampling plan (see ACI 201.1R; ASTM C 42; ASTM C 823, "Sampling Concrete in Constructions"; ASTM C 856, "Samples"), the results of any examination or testing cannot be considered to apply to any portion of the HCC not thus sampled. Information concerning the statistical sampling of concrete and concrete-making materials can be found in Abdun-Nur (1978) and Arni (1978). The bibliographies and appendix of these references provide source material to cover most sampling problems.

B.2 TYPES OF SPECIMENS

The specimens for petrographic examination may be of several types: (1) cores (drilled from the hardened concrete with a diamond core drill); (2) cylinders cast from the unhardened mixture at the time the HCC was placed; (3) fragments broken naturally or by sledge or pneumatic hammer from the placement; and (4) laboratory specimens such as mortar bars, beams, and test cylinders. It is important that the concrete in the specimens be as nearly like the HCC under investigation as practical.

1. *Cores.* Cores should be at least 4 in. in diameter and, if possible, at least 8 in. in depth. Full-depth cores are preferred. Cores must be virgin—not specimens that have been previously tested for compressive strength or used for other destructive tests. A minimum of three cores from any area concerning which petrographic information is sought and from any comparison area should be submitted. The cores must be unaltered by any testing. When cores are taken for any destructive testing, three companion cores should be taken and reserved for petrographic examination. In general, cores are more useful than cast cylinders.
2. *Cylinders.* Cylinders cast during placement may differ from the body of the HCC because of exposure to different temperatures (different maturation rate), subjected to different degrees and types of consolidation and curing. If water has been added to the mixture since it arrived at the job site, the cylinders will not be representative of the mixture placed unless they were fabricated after the water was added. When such differences are known, they should be reported in the documentation accompanying the cylinders submitted for petrographic examination.

3. *Fragments.* Fragments of concrete, particularly deteriorated concrete, must be considered to be representative only of the zone of the placement most like the fragments. Such fragments may be valuable as preliminary specimens that can be studied in order to plan further examination of the placement, a more extensive sampling, or both. If the HCC is so deteriorated that full-depth cores are impossible to obtain, pieces of cores or even fragments will have to be studied.
4. *Laboratory specimens.* Specimens of HCC produced in the laboratory may be submitted to the petrographer in order to determine the microstructural effects of various materials used or of experimental treatments of the HCC. Control specimens of HCC of known quality should be simultaneously submitted.

When the specimens of HCC submitted to the petrographer are insufficient in number, size, depth, or distribution of source locations, they must be treated as preliminary specimens that are to be examined in order to determine the necessity for a more complete examination of the placement and a more extensive sampling program.

B.3 SAMPLING PLAN

Despite the fact that most clients would prefer to take specimens of only the most questionable area (often an area they wish to remove anyway), the petrographer must become familiar with the material of the entire placement. For example, if one portion of a placement is showing distress or exhibits failure of some sort, specimens should be obtained not only from the area of failure but also from nearby HCC that was presumably of the same mixture but that is free of failure. These companion specimens should be sufficiently large and numerous to represent the "healthy" condition. They should be composed of the same materials (aggregates, cement, and admixtures) and should have been specified to have been made from the same mixture proportions. In addition, if various degrees of failure of the material exist, the specimens submitted must also represent these intermediate conditions.

The steps taken to develop a sampling plan should include the following:

- Define the nature and extent of the investigation.
- Procure a sketch or plan view of the site under investigation.
- Locate all areas of questionable material on the plan view.
- Describe the ways in which the questionable areas differ from areas that are considered good.
- Locate areas of intermediate quality and describe.
- With the intent of finding out how the materials, weather, and incidents that occurred during placement differ between the questionable areas and the good areas, collect pertinent data from the inspector's notebooks, casual observers, and, if possible, the contractor.
- Define the questions to be asked of the petrographer.

The client should furnish the petrographer with complete information concerning the sampling plan used and the sketch showing the relationships between the specimens. Specimens must be labeled so that their source location can be identified.

The results of any testing already performed on the concrete in question, the data collected, its relationship to the specimens submitted, and the reasoning used in selecting the locations sampled (see ASTM C 856, "Samples") should be reported to the petrographer.

B.4 SAMPLING PROCEDURES

The location from which the samples are obtained will depend on the objectives of the investigation at hand. Specimens of concrete should be as little damaged by the removal methods as possible; otherwise, the petrographer will not be able to ascertain which cracks are indigenous to the HCC of the placement and which were caused by the collection procedures. Core specimens are usually preferred.

B.5 SPECIAL CONSIDERATIONS

B.5.1 Air-Void Samples

Air-void determinations may be required whenever it is suspected that the air-void content is not sufficient to provide protection from freezing and thawing deterioration or whenever it is suspected that the cause of low strength might be excess air content. If the air-void content of the entire placement is in question, sampling should follow the instructions detailed in ASTM C 457, "Sampling," as follows:

> To determine the compliance of hardened concrete with requirements of specifications on the air-void content or specific surface and spacing factor of the void system, a sample of the concrete should be obtained from at least three locations in the body of concrete and microscopical measurements should be made on at least one section prepared from each of at least three of such samples.

The three locations sampled must be selected from the entire body of the placement under study according to a rigorously random plan without regard for areas of extreme deterioration. The areas exhibiting specific features should be sampled separately. These sampling guidelines may be followed for any concrete suspected to deviate from the required quality. Each sample should be large enough to allow the petrographic staff to prepare at least the minimum area of finished surface given in ASTM C 457, Table 1. The petrographer should be consulted in any case of doubt.

B.5.2 Overlay Material

B.5.2.1 Cracking

Core specimens of overlays that have cracked must be taken with special care. All cores must be the full depth of the overlay. Each core should be centered on a crack and should be examined as it is removed from the placement. If the crack extends to the bottom of the core, subsequent cores should be deep enough to include the full depth of the crack system.

B.5.2.2 Delamination

Core specimens of delaminated overlays must include at least 2 in. of the substrate concrete. If the core comes apart at the bond line during coring, the two pieces will

grind on each other and destroy the evidence of the nature of the bond. In this event, additional cores should be taken in an effort to obtain specimens of the bond itself.

B.5.3 Frozen Concrete

If freezing of the HCC while fresh is suspected, at least one specimen should be obtained from the edge of the placement, from up against the form, or from a place exposed to the ambient temperature. It is in such an exposed area that the casts of ice crystals will form first. If companion cylinders were cast and cured as was the placement, they may show ice crystal casts on the surface in contact with the mold. An ambient freezing temperature while the concrete is fresh usually affects the wearing surface only if the curing material is insufficient to retain the heat generated by the hydration of the cement or if the curing material is prematurely removed (possibly by wind).

B.5.4 Unusual Conditions

Unusual conditions may necessitate unusual methods of sampling. For example, the giant popout shown in Figure B-1A was found lying loose on a railroad tie beneath a concrete highway bridge. It was a curiosity, and we were concerned only with the reason for the popout and not with the main mass of the concrete. A method of reaching the spot on the overhead concrete was found, and the hygroscopic glass shown in Figure B-1B was recovered from the matching depression. The ordinary popouts (photographed for size contrast in Figure B-1A) were recovered loose from a highway surface. Each of these contained a fragment of porous chert at its apex. These may be considered classic popouts, pushed out of the pavement surface by the freezing and expansion of water in the porous chert. These specimens are useful reference specimens, and the chert popouts are sufficient evidence to allow the petrographer to recommend against further use of this particular aggregate in wearing courses, but the sampling procedures, although sufficient, are hardly those classically specified.

B.5.5 Aggregate Specimens

It is important that the field sampling of aggregate specimens be such that the aggregate sample is truly representative of the material proposed for use and that the ratios between the various lithologies and sizes have not been influenced by the sampling procedures (Mullen, 1978; Price, 1978).

B.6 COMPARISON OF FIELD AND LABORATORY SPECIMENS

Access to laboratory-produced specimens of HCC and the mixture proportions by which they were fabricated can prove very useful to the concrete petrographer. They provide an excellent opportunity for study of specimens of HCC produced with various experimental materials. They can also provide examples of HCC produced with a large variety of types of aggregate and numerous different admixtures so that the variations among 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 ob-

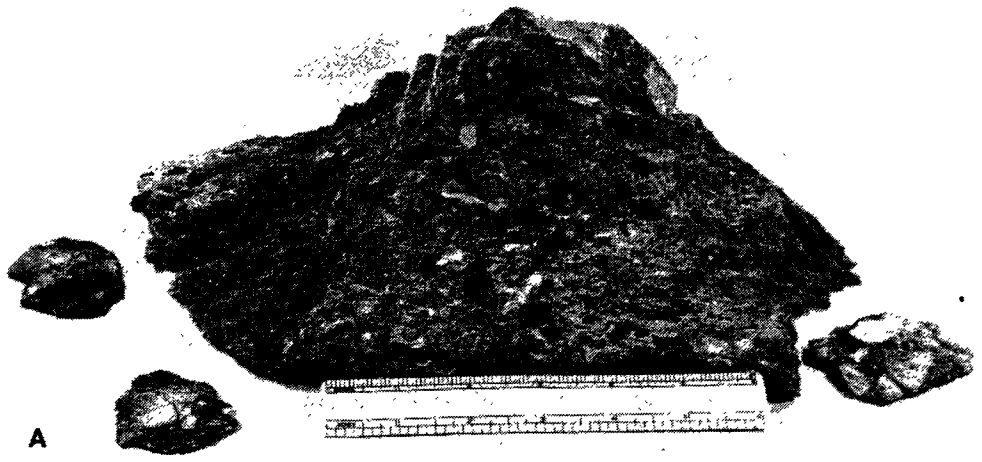




Figure B-1 POPOUT. A. A giant popout caused by a piece of hygroscopic glass. Accompanying it are several small popouts of a more usual size caused by porous chert particles. **B.** The hygroscopic glass. Natural size.

servations can be compared with the data obtained in the laboratory. The data obtained from the concrete mixing laboratory and made available to the petrographer should include the exact nature and source of the ingredients, proportions of the mixture, and results of any testing such as the following:

- slump
- unit weight
- air content when fresh
- curing method
- maturity
- compressive strength
- permeability
- results of testing for resistance to freezing and thawing
- results of any other testing

The concrete mixing laboratory and the concrete petrographic laboratory supplement each other. When the results of the testing done in the concrete mixing laboratory do not seem to make sense or do not explain the problem under consideration, petrographic examination may be able to provide illumination.

Construction problems usually require rapid solutions that cannot wait for results from 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. Such experimentation must usually be performed later under more deliberate, controlled conditions. In any case, it is widely recognized that it is difficult to duplicate bad concrete in the laboratory. Features that are due to poor workmanship or incomplete mixing are especially difficult to duplicate. 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 laboratory-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 at the construction site. All too commonly, water was added to the mixture after the concrete began to stiffen and after the air content determinations were made. This retempering (see Appendix D) is usually not documented and must be inferred from the parameters of the concrete (Mather, 1978; Meilenz, 1978).

Investigations that include the fabrication of special concrete mixtures 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 certain construction sites.

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Appendix C
CAUSES AND PREVENTION OF PLASTIC SHRINKAGE CRACKING

C.1 CAUSES

Plastic shrinkage cracks occur when the rate of evaporation from the surface of the HCC exceeds the rate of bleeding (see ACI 224R). Bleeding is a process whereby the solids of the HCC, including the cement and other fine particles, settle and water rises to the top. (The process is thought to be a form of syneresis by some.) The bleed water makes a "sheen" on the top of the HCC. When the process proceeds as it should, water is evenly distributed throughout the thickness of the HCC placement. The water sheen on the surface prevents the top portion of the HCC from becoming drier than the bottom portion; that is, the water on the surface maintains 100% humidity throughout the concrete. The condition of 100% humidity is required so that there will be sufficient water for the remainder of the hydration to take place and so that the HCC will fill the space appropriately and not shrink.

When the proper humidity is not maintained, the top portion of the HCC becomes drier than the lower portions of the HCC and shrinkage (loss of volume) occurs within the drier portion. When HCC shrinks, it can no longer fill the space it occupies. The lower portion (where there is 100% humidity) does not shrink, and, therefore, the entire body does not change size. The drier top (the smaller portion) cracks to accommodate the shrinkage but remains attached to the larger bottom portion.

When plastic shrinkage cracking of any great extent is observed, careful inquiry into the inspector's records and the observations of others who were near the placement will almost always indicate that one or more of the following occurred:

1. The drying conditions were so severe (see Fig. C-1) that the work should have been postponed until more favorable climatic conditions existed.
2. The paving train became so strung out that there was too much time for evaporation between loss of surface water and the finishing and curing operations.
3. Curing was not begun as specified.
4. Curing was not maintained as specified:
   - misting was not continued
   - the burlap dried
   - the polyethylene, burlap, or both were removed by the wind

C.2 PREVENTION

When finishing is complete and the sheen disappears by evaporation of the surface bleed water, curing procedures must begin promptly. At this point, there is no layer of water to protect the HCC from drying or maintain the 100% humidity within the HCC. If there is a wind blowing, the humidity is low, the ambient temperature is

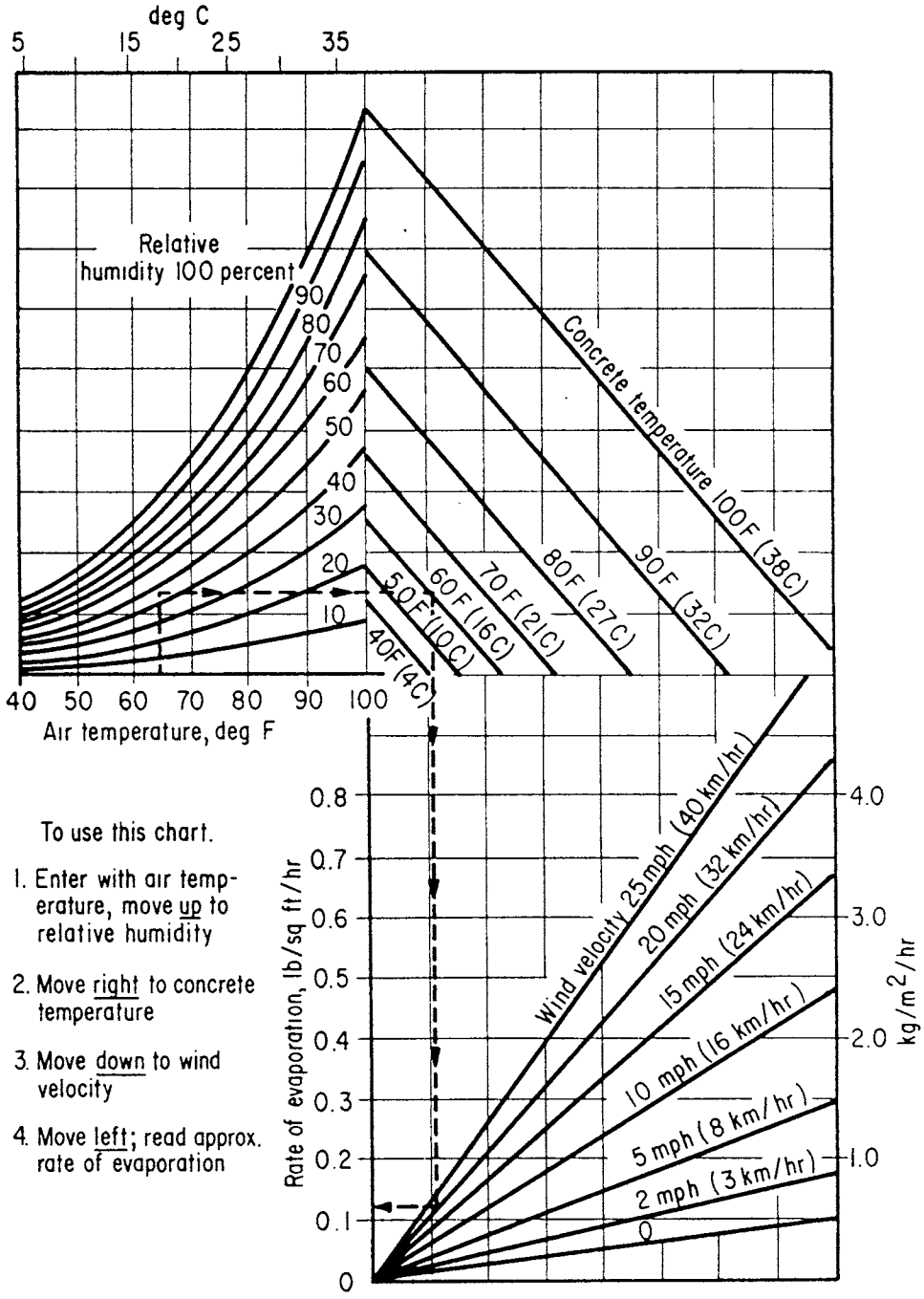


Figure C-1 EFFECT OF AMBIENT CLIMATIC CONDITIONS ON RATE OF EVAPORATION. Effect of concrete temperature, air temperature, wind velocity, and relative humidity on the rate of evaporation of surface moisture from a concrete surface. If the rate of evaporation approaches the danger point for the concrete being used, precautions against plastic shrinkage cracking are necessary (*ACI Manual of Concrete Practice*, updated yearly).

high, or the temperature of the HCC is high, the rate of evaporation will be especially rapid. Promptness is the essence of efficient curing. Figure C-1 shows how all of these conditions combine to contribute to the rate of evaporation.

For ordinary bridge deck concrete, the rate of evaporation should not exceed 0.1 lb/ft^2/hr (0.5 kg/m^2/hr). For latex-modified concrete overlays and other cement concrete with a water-cement ratio less than 0.40, the maximum evaporation rate must not exceed 0.05 lb/ft^2/hr (0.25 kg/m^2/hr) (Kuhlman, 1991).

The curing procedures must be maintained properly. When climatic conditions are very unfavorable, it may be necessary to raise the humidity by misting the air over the concrete, erecting wind breaks or sunshades, or limiting the placement of HCC to cooler nighttime hours. The moisture must be maintained in the HCC throughout the curing period. It will be necessary to remoisten any curing cover (such as burlap) periodically.

C.3 LEGAL INVESTIGATIONS

The client or an adversarial witness in a court proceeding may wish to inquire concerning the following:

1. Was the shrinkage cracking noticed before the finishing operations were complete?
2. Did the finishing procedures tend to hide the cracking (see Fig. 4-1)?
3. Did the inspector warn the contractor against proceeding with the placement for any reason, at any time? Why?
4. What corrections in procedure did the inspector recommend?
5. What were the data concerning concrete temperature, air temperature, humidity, wind velocity, and direction? If the climatological data are not available from the inspector's records, they can usually be obtained from a nearby airport.

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Appendix D
RETEMPERING

D.1 OVERVIEW

Retempering is the process of changing the consistency of a concrete mixture by adding water and remixing. As it is common to send the concrete to the placement site with slightly less water than the maximum that may be used, it is expected that a specified amount of water can be added if necessary. The contractor may add the water because the mixture arrives at the site in a condition that would make placement and finishing difficult. These difficult HCCs are often termed "harsh mixtures." They lack workability. (The only quantitative measure of workability is slump.) (See 6.1 and 8.4; Gaynor & Meininger, 1983; Pigeon, Saucier, & Plante, 1990.)

The usual cause of a harsh mixture is a sand with a high void content (see Appendix E). Sands with a high void content are usually irregular in shape with an abundance of re-entrant angles and internal fractures and voids. Iron-stained clay coatings are common. Other causes of concrete that seems too dry are improper grading (size distribution) of the aggregate and the presence of mud or mud coatings on the aggregate. Additionally, a deficiency of fine aggregate or coarse aggregate that is oversized or has a very poor particle shape can create fresh concretes with a difficult texture.

Mixtures with a low water-cement ratio (below 0.45) can be difficult to place unless an effective water reducer is used. A good air-void system or the presence of fly ash as substituting for part of the cement can help make a mixture with a low water-cement ratio more workable. Apparently, the air acts as a fluid and the particles of fly ash are more equant than those of cement and act as ball bearings.

D.2 A LIKELY RETEMPERING SCENARIO

Rims of cement on the aggregate and knots of cement in the paste (see 8.7) suggest that the following typical scenario may have occurred. When the ready-mix truck arrived at the job site, it was quickly noted that the fresh HCC had a rough texture and looked as if it required more water. If the mixture was designed to have a low water-cement ratio, each of the aggregate particles in the mixture was completely coated with this very adhesive mixture. Such HCC may be very difficult to place unless a sufficient quantity of a water-reducing admixture was used. If the coarse aggregate is oversized or has a poor shape or the sand is present in an insufficient amount, is unusually angular, contains many cracks, or has many re-entrant angles, the mixture will look stiff and difficult to place (harsh mixtures). It is common under such circumstances for water to be added to the mixture to increase the slump and workability. The additional water must not increase the total water above that designed for the mixture lest the concrete become weakened because of the higher water-cement ratio.

D.3 EFFECT OF RETEMPERING

D.3.1 On HCC Paste

When water is added after hydration of the cement has begun and mixing restarted, it commonly happens (especially in mixtures with a low water-cement ratio) that the water is not distributed throughout the entire mixture but is mixed only into the larger spaces between the aggregates. The material already adhering to the aggregates remains as a rim of darker material with a low water-cement ratio around the aggregate particles and in the re-entrant angles. Patches of the original paste (unaltered by the additional water) may remain and can be found completely surrounded by the paste of higher water content. The problems of incomplete mixing are akin to the problems encountered in certain cooking situations. With gravy or white sauce, the thickening agent (such as flour) must be completely mixed with the cool water before the flour is affected by heat and begins to hydrate. If the flour and hot water mixture becomes too coherent, it may be impossible to add more water and create a smooth paste. The added water will mix with only a portion of the paste, and lumps of flour coated with stiff hydrated material will remain no matter how much mixing takes place.

Whenever water is added to the mixture without additional cement being added, the water-cement ratio is raised. The higher the water-cement ratio, the weaker the HCC. When more than the allowable amount of water for a given amount of cement is added to the mixture, the HCC will not have the designed strength. When the rims indicating incomplete mixing are present, a large portion of the cement can be concentrated in the thin bands of very rich paste around the aggregate and in the lumps of the original paste. The remainder of the paste is relatively depleted of cement and is thereby weaker than would be expected from the water-cement ratio calculated from the originally delivered mixture plus the additional water. Thus, it can be seen that areas of HCC with a high water-cement ratio can exist in close proximity to areas with a low water-cement ratio.

It must be remembered that any material is only as strong as its weakest zone. Stress in HCC in service or in a testing apparatus will cause cracking. Cracks will always follow the zones of weakness. In HCCs that have paste areas with different water-cement ratios, the cracks are going to develop in the areas of higher water-cement ratio and thus the strength will be dependent on the extent and continuity of those areas.

The skeptic will mention the fact that the bond between the aggregate and the paste in many HCCs is the weakest area and say that the dark rims of high cement content eliminate this problem. Although this is true, the fact that weak bond areas are not as continuous throughout the paste as are the light-colored areas with a high water-cement ratio (low cement content, high water content) obviates the value of rims with a high cement content as bonding agents.

D.3.2 On Air Voids

D.3.2.1 Quantity

Air-entraining agents are more active in the presence of additional water. When retempering has occurred and the mixing has not been complete, petrographic ex-

amination will show that many portions of the paste have a much higher void content than does the HCC of the rims and dark patches. Thus, the weakness of the portion with a high water-cement ratio is compounded by the portion containing more than its proportionate share of air voids. In moderate cases, the spacing factor of the air-void system may change very little because the spacing factor is most dependent on the very small voids. Pigeon et al. (1990) reported that there was little change in the spacing factor in the mixtures they studied if the retempering did not increase the slump by more than 4 in. (100 mm).

D.3.2.2 Shape

When remixing takes place after some coalescence of the HCC has occurred, the remixing may occur after the individual integrity of some of the small air voids has formed. In such cases, many of these voids will retain their surface area but lose their original spherical shape and become ovoid, or squashed, or develop an angular shape. Many angular voids may be seen in Figure 8-1.

D.3.2.3 Size

Retempering can cause an increase in the size of air voids, the number of air voids, or both. The size of the voids caused by retempering as evidenced by the microscopical examination shows that the larger voids (more than 1 mm across) nearly all occur within the portion with the higher water-cement ratio. In normal, well-proportioned HCC, the percentage of voids whose diameter expressed on the surface examined exceeds 1 mm should be less than 2% of the total concrete.

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Appendix E
AGGREGATES USED IN HCC

E.1 OVERVIEW

Usually, the aggregates used in HCC are naturally occurring earth materials that have been crushed, graded, and washed as needed to meet the requirements of the concrete being produced. The amount of beneficiation required will depend on the nature of the aggregate and the requirements of the specifications. Often, transporting the aggregate is more costly than obtaining it from the quarry (presumably cleaned and sized). Therefore, aggregate sources near the concrete production plant are often preferred over sources of higher quality material located at a greater distance. The preparation of aggregate specimens for petrographic examination is described briefly in 5.5.

Not all natural rocks are suitable for use as aggregate. The material used must pass certain tests as specified in ASTM C 33 and any specification document provided by the client, customer, or purchaser. In Virginia, the document is the *Road and Bridge Specifications* (1991).

The petrographic description of the aggregate should be guided by ASTM C 125 (standard terminology for concrete) and ASTM C 294 (descriptive nomenclature for aggregates). The procedures given in ASTM C 295 (petrographic examination of aggregates) can be used when a supply of the aggregate is available. Detailed information may be found in Dolar-Mantuani (1983), Meilenz (1978), Mullen (1978), Ozol (1978), Price (1978), and Schmitt (1990). Basic texts on petrology, petrography, and mineralogy should be available and familiar to all persons doing this work (see the Reading List). Any person performing petrographic examinations of HCC or aggregates or who is engaged in specifying aggregate properties should carefully study the literature on earth materials and the works on concrete aggregates.

The HCC petrographer who generally works with concretes fabricated in a given area, such as a state or group of states, will find that the more familiar he or she becomes with the aggregates from that area the easier the aggregate identification will become. A collection of reference aggregates is very helpful. The aggregates should be identified as to quarry, approximate date quarried, geologic formation, and rock type. The collection should include specimens of concrete containing the aggregate as well as specimens of the unused aggregate.

The ability of aggregate to withstand the stresses induced during the mixing of HCC is very important, and, therefore, aggregates must be able to conform to the abrasion resistance specified in ASTM C 33 (specification for concrete aggregates) as tested in accordance with ASTM C 131 (resistance to degradation of small-size coarse aggregate) or ASTM C 535 (resistance to degradation of large-size coarse aggregate). Commonly, the external characteristics of the aggregate (such as distribution of the particle sizes, shape, texture, surface coatings, type of fracture, surface area, and so forth) are more important to the behavior of aggregates in HCC than is the mineral and chemical composition of the aggregate. Mullen (1978) provided an excellent explanation of these properties. A summary of some of these testing procedures may be found in the ASTM *Manual of Aggregate and Concrete Testing*

(1990). The fine aggregate (that passing a No. 4 [4.75 mm] sieve) requires specialized testing (Gaynor & Meininger, 1983).

E.2 COARSE AGGREGATE

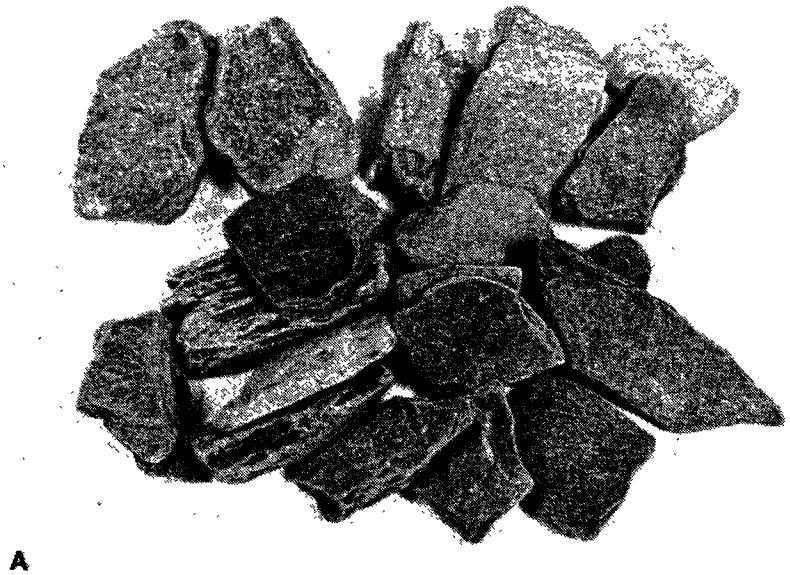
Coarse aggregate (that retained on a No. 4 [4.75 mm] sieve) for use in HCC is selected mainly on the basis of durability, size, general shape, mineral composition, economy, and availability. Figures E-1 and E-2 illustrate aggregate particle shapes that are avoided when economic reasons permit.

The requirements of the proposed HCC placement must be fully considered. A placement that has many reinforcing bars close together will require a much smaller coarse aggregate than one with no reinforcement. Preplaced aggregate is often very large and always lacking in the finer size (see Lamberton [1978]). In certain cases, the specific gravity or mineral composition of the aggregate is important. A high specific gravity will make it more difficult to prevent segregation, but the use of aggregate of high specific gravity may be dictated by availability of aggregate or specific use of the concrete. Refer to any specifying document of the client to determine the fitness of a coarse aggregate to meet the requirements of the HCC (see E.6).

E.3 FINE AGGREGATE

Sand (fine aggregate) for use in concrete should be tested for shape and surface smoothness. If the particles have angular shapes with abundant re-entrant angles, the sand has a high void content and a high water demand. It takes more fluid (or cement paste) to surround an angular particle than to surround an equant particle. Among solid shapes, the ratio of surface area to volume is smallest for spheres and largest for extremely lath-shaped particles and particles with deep re-entrant angles and internal cracks and cavities. If the fluid present is insufficient to coat the surface area, the concrete mixture is harsh and difficult to place and finish. This condition is perceived, during construction, as a need for more water.

It is almost impossible to estimate the void content of a sand from a petrographic examination of a finely lapped slab because the visual contrast between the paste and the sand particles is very low unless the sand is stained or coated. The particle shape is tested by determining the void content by means of Virginia Test Method No. 5 (Virginia Department of Transportation), the test method described in Gaynor and Meininger (1983), or ASTM C 29. The test measures the unforced packability of the aggregate and thus indirectly measures the water demand of the aggregate. A concrete with a high water demand will often prompt the performance of a retempering procedure. In thin section, the outline of the sand particles can be easily distinguished from the paste by means of the birefringence of the sand particle. (Very few sand particles have birefringence as low as that of the paste.) In fluorescent microscopy, where the thin section is impregnated with a fluorescent dye, the outline of the aggregate is emphasized because the aggregate is not illuminated by fluorescence and the porous paste is. The weak zones caused by the high water demand are exceedingly porous, contain very large capillaries, and become brightly illuminated by the fluorescence of the impregnating dye (see Figs. 13-13 and 13-14). Gaynor and Meininger (1983) also detailed a few of the other tests sand should pass.



A



B

Figure E-1 SHALY PARTICLE SHAPE. A. Particle shape of a crushed slate aggregate. **B.** Pavement surface with a flaw caused freezing and thawing of water trapped under a particle of the aggregate shown in **A.**



Figure E-2 AGGREGATE PARTICLES FROM FISSILE GNEISS. A particle shape such as this can cause a high water demand and popouts such as those shown in Figure E-1B.

E.4 IDENTIFICATION OF ROCK TYPE

When required, the exact mineralogical identification of natural and artificial aggregates can be performed by classical petrographic methods. Monomineralic aggregates and the minerals of which the rock is composed can be properly identified. The methods used may include determining the optical properties by use of the petrographic microscope when the aggregate is examined in grain mount, thin section, or both; various methods of determining the chemicals present; determining the crystal structure by x-ray diffraction; and matching x-ray diffraction patterns for positive identification. There are many excellent books written on these methods, and courses on these methods are taught in the materials engineering and geology departments of numerous universities (see the Reading List).

The naming of rocks that are composed of mixtures of several minerals (as are most rocks) will require some knowledge of rock classification methods, i.e., how the ratios between the various minerals influence the name of the rock. The names of sedimentary rocks are usually easy to understand and use. Such terms as *sandy limestone* and *fissile shale* are self-explanatory. The major type of confusion results with a word such as *marble*. To an architect, an engineer, or a building contractor, the word means any decorative, polishable stone. To them, the word refers to the appearance of the rock and its engineering properties. To a geologist, the word means carbonate rock that has undergone metamorphism. Metamorphism includes recrystallization, mineral and fossil replacement, folding, crushing, and/or recementation. In general, the engineer is unconcerned with the geologic origin of a rock material. The specific geologic names of the igneous rocks can be confusing because

they involve very specific grain-size classification, mineral identification, and often quantitative determination of the relative amounts of the types of feldspar and the mafic minerals present. The contractor or engineer finds such exact classification unnecessary and will often call all coarse-grained, light-to-medium colored, hard, tough rocks *granites* and all fine-grained, hard, dark rocks *trap rocks*.

When the equipment is readily available, x-ray diffraction, x-ray fluorescence, scanning electron microscopy, and transmitted electron microscopy can be used for exact mineral identification. Such identification is necessary only when the distinction between similar rocks might elucidate the reasons for differences in durability or other behavior when the aggregate in question is used in HCC.

Most often, the exact identification is not necessary. Usually, the most detailed examinations are required when alkali-aggregate reactions are suspected or carbonate rocks are suspected of causing D-cracking (Schwartz, 1987). For more data on alkali-reactive aggregates, refer to Chapter 10 and the associated figures and references, the related ASTM standards (Reading List), and the section on thin-section preparation (5.2).

E.5 AGGREGATE CONCERNS IN D-CRACKING

D-cracking that is due to destruction of the aggregate by freezing and thawing has been well documented in the literature, and much information concerning it can be easily found (Schwartz, 1987). Both the alkali-carbonate reaction and D-cracking involve dolomitic limestones whose composition includes a large portion of insoluble material. Dolomites involved in D-cracking are thought to require a certain fine pore structure and/or contain a minor amount of iron, strontium, or both in their crystal structure (Dubberke & Marks, 1987; Schwartz, 1987). A specific pore structure and iron or strontium do not seem to be necessary conditions for the alkali-carbonate reaction. The crack patterns produced by these two reactions are very different. D-cracking deterioration is first evident at the edges and near the joints of a pavement. The cracking is parallel to the joint or edge (see Fig. E-3 and compare with Figs. 10-15 through 10-17). The alkali-carbonate reaction is an expansive chemical reaction with the alkalies, whereas D-cracking is caused by freezing and thawing of moisture in the particles of certain susceptible dolomites and does not involve alkalies.

I have had no experience with D-cracking because it has not been found in Virginia. Schwartz (1987) stated:

> D-cracking is a form of portland cement deterioration associated primarily with the use of coarse aggregates in the concrete that disintegrate when they become saturated and are subject to repeated cycles of freezing and thawing. It is defined by a characteristic crack pattern that appears at the wearing surface of the pavement as a series of closely spaced fine cracks adjacent and generally parallel to transverse and longitudinal joints and cracks and to the free edges of the pavement (p. 5).
>
> It is generally accepted that pore size is the most important characteristic of coarse aggregate influencing its susceptibility to D-cracking (p. 10).
>
> It is generally agreed that the brand or composition of cement does not significantly influence D-cracking (p. 11).

In Iowa, Dubberke and Marks (1987) found that D-cracking is not necessarily related to the pore structure of the aggregate and came to the conclusions that the in-

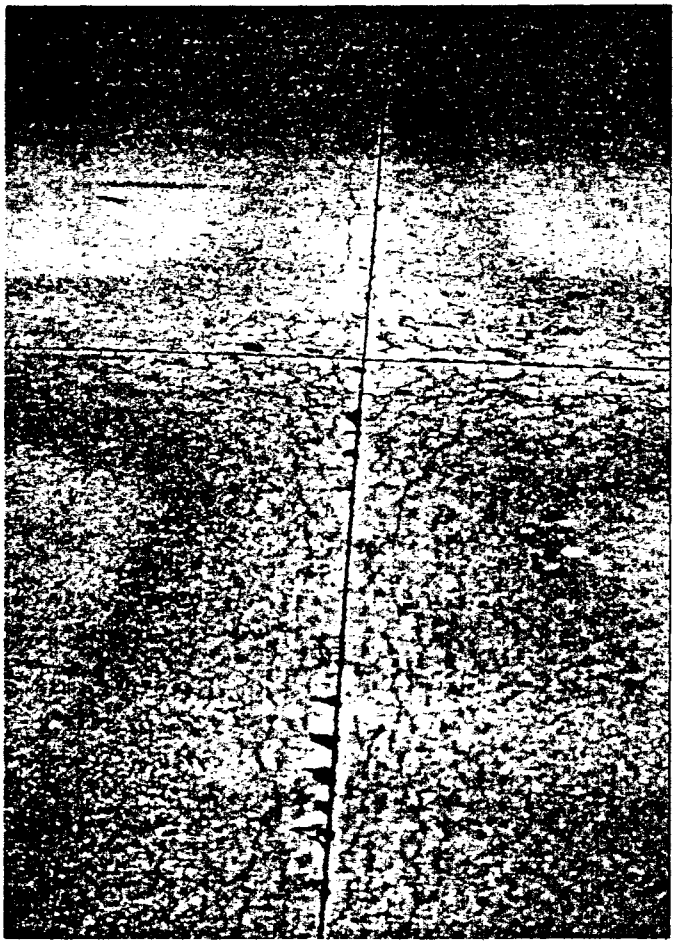


Figure E-3 D-CRACKING. D-cracking of a pavement due to destruction of the aggregate by freezing and thawing. The cracking parallel to the joint and wrapping around at the juncture of joints is indicative of D-cracking.

cidence of D-cracking is higher with ferroan-dolomite aggregates than with other compositions and D-cracking may be due to or hastened by a chemical reaction with deicing salts.

E.6 SPECIAL PURPOSE AGGREGATES

E.6.1 Skid Resistance

Skid resistance is usually desired for any aggregates used in a surface of HCC that supports traffic. Skid resistance requires that the surface of an HCC pavement be fabricated with hard, nonpolishing aggregates. This generally means that nonsiliceous carbonate rocks cannot be used. In areas where carbonate aggregate is much less expensive than harder aggregate from more distant sources, two-course construction may be the most economic alternative. Not all types of siliceous aggregate provide the same wearing-surface microtexture as others. Despite their hardness, some quartz and feldspar pebbles and quartzitic and granitic rocks may tend to wear with a rounded surface and to polish (see Fig. E-4). Others with certain zones of weakness such as certain granites and graywackes wear and then break with a microtexture that creates a very skid-resistant wearing surface (see Fig. E-5) (Webb, 1970).



Figure E-4 TRAFFIC-WORN ROUNDED SURFACE OF FELDSPAR AGGREGATE PARTICLE. This will not provide good skid resistance. The scale is in millimeters.

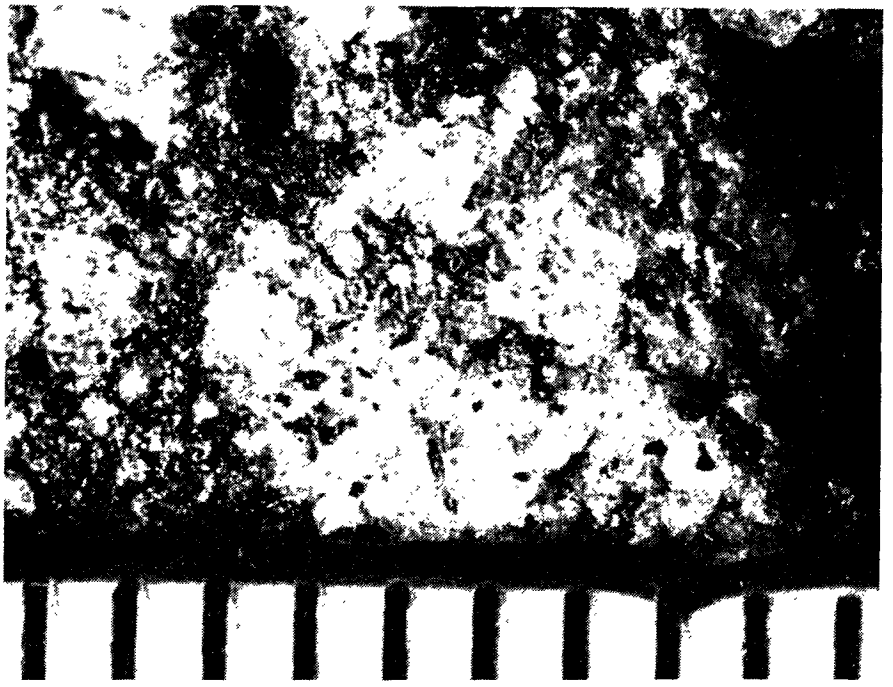


Figure E-5 TRAFFIC-WORN SURFACE OF GRANITE AGGREGATE PARTICLE. This has zones of weakness to provide an irregular skid-resistant surface. The scale is in millimeters.

E.6.2 Lightweight Aggregates

Lightweight aggregates may be specified whenever the weight of conventional aggregates might be a problem. They are frequently used for long span bridges to alleviate the dead load on the support structures. They may be used when bridge decks require widening and it is considered more economical to widen with the more expensive expanded aggregates than to increase the strength of the support structure.

Manufactured lightweight aggregates are usually shale or slate that has been expanded by treatment at very high heat. The exterior of the particles becomes fluid, and the gases and vapors inside expand to create a very porous substance with a fused exterior shell. Figure E-6 shows this sort of aggregate exposed on a lapped slice of HCC.

These expanded aggregates vary considerably with the source material used and the nature of the heat treatment (temperature, time, oxidation conditions, etc.). Much depends on the depth and continuity of the fused surface of the aggregate particles. If this surface is continuous, the aggregate will have a low permeability despite its high porosity.

It is suspected that the chemical composition of the fused layer may have an important effect on the ability of these aggregates to bond with cement paste. Certain expanded aggregate materials produce concretes in which the bond between the aggregate and the paste is extremely good. In other cases, the bond is no better than would be expected of a quartz-pebble aggregate. In the examples of a good bond,

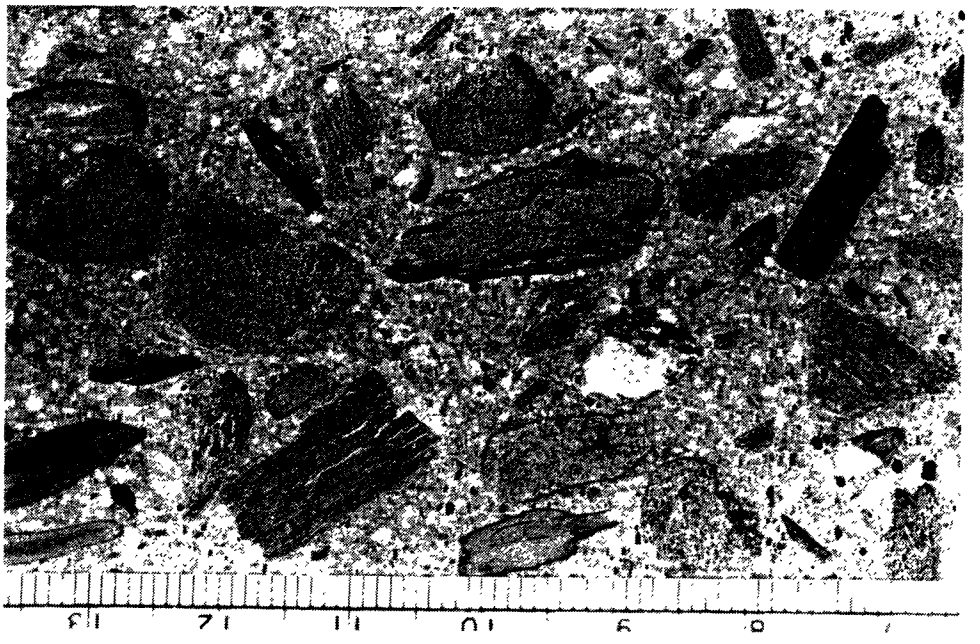


Figure E-6 LAPPED SLICE OF HCC FABRICATED WITH EXPANDED LIGHTWEIGHT AGGREGATE. The scale is in millimeters.

the aggregate surface does not seem to have an attraction for water, as does quartz and many other highly siliceous materials. These layers of water on the surface of aggregate particles create space along the bond line in the finished concrete that, whether or not it fills with calcium hydroxide, is a zone of weakness in the concrete and a possible channel for water, salt solutions, and other liquid materials. It has been shown that this attraction for water that lessens the paste-aggregate bond is most often present in materials that are acidic by nature whereas the materials that may be considered mafic or alkaline, such as the carbonates and iron-rich minerals, generally have a much tighter bond (Walker, 1972b). It has not yet been shown that the chemical composition of the fused layer in expanded aggregate has a direct effect on the properties of the bond. I suspect that such research would be likely to yield interesting and useful results.

Certain rocks can be used as lightweight aggregates without heat treatment. These may include volcanic ashes, tuffs, and pumices.

E.6.3 Radiation Shielding

Concrete for radiation shielding is designed using heavy-weight or special composition aggregates so that the maximum amount of radiation may be contained. These requirements are dealt with in ASTM C 637 and ASTM C 638. Many of the aggregate minerals used in radiation shielding are opaque, and identification cannot be determined with the petrographic microscope. If the exact mineral identification is required, x-ray diffraction and some form of chemical testing or examination with a metallographic microscope will have to be performed.

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Appendix F
PREVENTION OF A DESTRUCTIVE ALKALI-SILICA REACTION

F.1 METHODS OF PREVENTION

Table F-1 lists the methods of preventing the deterioration caused by the alkali-silica reaction.

Table F-1
METHODS OF PREVENTING DESTRUCTIVE ALKALI-SILICA REACTION

1. Specify a low-alkali cement (not often practical).
2. Specify nonreactive aggregates (if possible).
3. Use an effective pozzolanic additive or blended cement such as:
   - fly ash (most class F, some class C) (byproduct of coal-burning electrical plants)
   - ground granulated blast-furnace slag (GGBFS) (byproduct of iron-steel industry)
   - silica fume (byproduct of the silicon reduction process)
   - natural pozzolan (no known U.S. sources)

Note: At the present time, there is no reason to believe that pozzolans will ameliorate destructive alkali-carbonate reactions.

F.2 USE OF POZZOLANIC MATERIAL

The mechanism(s) by which a pozzolanic additive or a pozzolanic cement reduces the expansion of the alkali-silica reaction is not well understood. There are three mechanisms that may be active:

1. The pozzolanic material may react with the hydroxide ions associated with the sodium and potassium ions in solution in the paste during the early stages of hydration and sequester the deleterious ions in gels that, because of their location or composition, cannot cause deleterious expansion. If all the alkalies are contained in such gels, no expansive gel will form at a later stage.
2. All pozzolanic materials combine with the lime in cement paste to produce cementitious calcium silicate hydrates and thus reduce the quantity of the lime in the HCC. This general reduction in the amount of lime may change the ability of the gel pockets to imbibe water.
3. The pozzolans promote the formation of a concrete that is much less permeable than concretes fabricated without them. Thus, a concrete is produced through which solutions cannot move as readily. Concrete fabricated with GGBFS as 60% of its cementitious material has been found to be 1/10 to 1/100 as permeable as comparable concrete without GGBFS at equal strength (B. Mather, personal communication, October 1991).

All three of these mechanisms may be active concurrently. In any case, it is well established that sufficient pozzolanic material (in the case of silica fume, sometimes only 4% of the weight of the cement) will dramatically reduce the expansion and prevent alkali-silica deterioration. The appropriate amount can be determined in accordance with ASTM C 441.

In general, the replacement of a portion of the cement by a reasonable amount of an efficient pozzolan has few contraindications. When combined with the lime of cement, a pozzolan forms a cementitious material. It was extensively used by the Romans. Good quality GGBFS has been successfully used in amounts up to 65% of the cement. Fly ash may be used in up to 20% of the cement. Silica fume is costly and in Virginia is not usually used in amounts larger than 8%. Large amounts of pozzolan (except silica fume) can delay final set and, thus, can necessitate the delay of form removal in cold weather (see Chapter 11). The final compressive strength of the HCC can, however, be as high or higher than it would have been if a portion of the cement had not been replaced. If quality control is not good, certain pozzolans can create other problems.

If fly ash has a large carbon content, it will adsorb organic chemicals, such as air-entraining agents. The carbon in fly ash acts as does activated charcoal. Its large surface area is capable of absorbing large amounts of organic chemicals. This is the reason activated charcoal is used as a filter in aquariums. When thus adsorbed, the air-entraining agent does not work efficiently. The resulting HCC may not contain an air-void system with the parameters necessary for a concrete protected from freezing and thawing distress. The carbon content can be measured by the amount of loss of mass on ignition (refer to ASTM C 311). As far as is known, neither GGBFS nor silica fume causes such problems. When used as a replacement for the cement, GGBFS is economically attractive and produces a concrete resistant to many types of chemical deterioration because it not only is excellent for preventing alkali-silica expansion but also produces a concrete of low permeability and thus inhibits the circulation of solutions.

See the Reading List for a list of applicable ASTM standards. 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, use such standards, and take all precautions necessary for the safety of personnel and applicable equipment.

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Appendix G
QUESTIONS AND SUGGESTIONS FOR FURTHER RESEARCH

G.1 OVERVIEW

It is obvious that there is much work still to be done in the field of petrographic concrete research and investigation. The concrete petrographer must be able to decipher the construction problems as they occur and be ready to advise engineers as to how to avoid the problems in the future. Research is required to answer the questions raised in many places throughout this manual (and others I have not mentioned) and investigate new materials and methods as they become available. New quarrying methods, new methods of construction, uses of waste material as aggregate, and new methods of obtaining laboratory data will, of course, continue to make the field a continually growing one that should always be interesting (provided funds can be found to further the necessary experimentation and investigation).

G.2 CONSTRUCTION METHODS AND MATERIALS

As with so many sciences, there are many changes and advances being made in the study and analysis of HCC by means of all of 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 (R. D. Walker, 1978).

G.2.1 Aggregate Shortages

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

G.2.2 Cement Alkalies

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 provisions for overcoming the problems the higher-alkali cements will create when used with the alkali-reactive aggregates.

G.2.3 Concrete

Concrete that is returned to the plant in a ready-mix truck is presently discarded, and the truck washed out to prevent the mixture setting up 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 also go into a settling pond before it is returned to the environment.

It has been found that this concrete can be kept in stasis over a night or a weekend if a retarder is added to the returned excess concrete. When downtime has elapsed, an accelerator can be added to neutralize the retarder and the concrete can again be ready for use.

However, it is easy to make a mistake on the dosage of these chemicals. It will require the efforts of many types of concrete technologists to discover the proper methods of conserving concrete by the use of them. As it becomes common to conserve concrete and protect the environment by such methods, the dosage errors possible will become more common and concrete petrographers will have to be on the lookout for the results of such errors.

The concrete petrographer should be continuously on the alert for the sort of problems that can be brought about by the use of new materials and techniques and should be ready to devise methods of detecting them, quantifying them, and reporting them to engineers.

G.3 CONCRETE LABORATORY EQUIPMENT

New concrete laboratory equipment becomes available and more useful as inventive persons become interested in concrete technology. New methods of using existing equipment add to our ability to discover more information concerning the different types of HCC. New advances in instrumental analysis occur all the time. The petrographer should make himself or herself aware of any new equipment and methods that could be used to advantage in petrographic examinations of HCC. If funds are available, every concrete petrographic laboratory should be equipped with the modern equipment that is presently known to be useful in the study of HCC. Such equipment includes 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 might be answered.

G.3.1 Equipment for Air-Void Analysis

The equipment for the determination of the parameters of the air-void system has become varied and in some cases quite sophisticated since the early days of these determinations. At one time, there were laboratories that used two persons to make an air-void determination by the point-count method. One person was the microscopist, who verbally reported observations to the other person, who manually recorded the data with paper and pencil. Today, with the use of image analysis, once the specimen is properly prepared and the equipment properly adjusted, the analysis can proceed without the presence of any human being. This is much faster than any of the older methods and eliminates the problems of training technicians in these microscopical techniques and the problems of operator fatigue. There are,

however, many concrete laboratories wherein it is preferred that the analysis be performed by a human microscopist and the specimen be uncolored by dyes or fillers. It is felt that only a human microscopist can make the subtle judgments that are often necessary. Also, there are many concrete laboratories that have not been able to budget for image analysis equipment. It can probably be assumed that some version of the microscopical methods, in which the human eye is the detector, will exist for a long time to come.

Image analysis or other methods yet to be invented may be able to determine air-void parameters not considered by ASTM C 457. Such additional parameters may lead to new theories concerning the air-void system and methods of controlling it. There has been concern for a few strange occurrences of concrete that by all the known parameters of the air-void system should be able to withstand the rigors of the weather and deicing but that have failed under field conditions. This problem was addressed by Buckingham and Spaw (1988). They are working on methods of using image analysis and a large number of the specimens (to make up for the heterogeneity of concrete) to determine the distribution of spacing factors in a concrete. This is in contrast to the classical method of merely considering the average spacing factor. It is thought that the concretes that fail without discernible reason may have an uneven distribution of air voids (and thereby an uneven distribution of spacing factors), and thus there may be areas within these concretes that are unprotected by the air-void system.

There are other possible explanations for failures that cannot be explained by deficiencies of the air-void system detected by conventional methods: (1) insufficient sampling, (2) improper sampling, and (3) improper performance of the determination of the air-void parameters.

G.3.2 Equipment for Detecting Ionic Permeability

The rapid chloride ion permeability test devised by D. Whiting (1981) is a good method for obtaining the relative permeability of HCC to chloride ions (and by analogy permeability to many other ions). 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. A comparison of the results of this or similar tests could be correlated with the features visible to a petrographer (with the stereomicroscope, the P/EF microscope, or both), and new insights could be gained as to the meaning of the visible features of HCC.

G.4 FREEZING AND THAWING OF CONCRETE

The specification for the percentage of air to be entrained in concrete resistant to freezing and thawing 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. Although the RH within the concrete can be measured, 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 freezing and thawing 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. I am 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 any protection?

Is it possible to have a concrete so completely saturated and subjected to a 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?

G.5 ALKALI-AGGREGATE REACTIONS

We know that the alkali-aggregate reactions require reactive aggregate, alkalies in the cement or in contaminants, and water. We are fairly certain that the alkali-carbonate reactions are active at a lower alkali content 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 sufficient good-quality pozzolanic material will prevent these reactions indefinitely. So far, this theory has not been around long enough to be substantiated. 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.

All of these reactions have had numerous theories about their exact mechanisms 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 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 solely due to the sodium and potassium salts present in a particular kind of deicing chemical used or does pure NaCl also increase the distress? Is there any similar effect on the alkali-carbonate reaction?

G.6 CONTENTS OF CEMENT IN CONCRETE

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 very rarely that a concrete supplier either by accident or design fails to supply concrete with the sufficient cement content 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 requirement, and may not be as durable as would be expected from the design of the mixture. There have been many tests devised to de-

termine 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 inform the contractor what to do when any problem arises that has 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 sufficient water-reducing admixture additive of an approved type be employed should be enforced. The petrographer can be instrumental in the devising and enforcement of proper specifications.

G.7 DETERMINATION OF AGGREGATE SIZE

At the present time, VTRC is not able to make a determination by optical means of the amount of a specific size or the ratio between sizes of aggregate in hardened HCC unless the size range of particle to be determined differs in lithology or color from other particles of aggregate occurring in the HCC. (If all the paste was dissolved away, a sieve analysis would be possible.) An optical method of determining particle sizing could be the comparison of results of determining chord lengths across the particles (by linear traverse or image analysis), with similar determinations made on hardened laboratory-fabricated HCC mixtures with known particle sizing. A large number of standard specimens would be required to determine the sizes in the mixture within meaningful limits. To date, it has not been felt that such work would be worth the time and expense, but if the problem occurred frequently with specific aggregate sources, it might be used in the future. Kite and Walker (1978) investigated particle angularity by chord length measurements, but, as far as is known, no effort has been made to determine sizes of irregularly shaped particles by such a method.

G.8 USE OF WASTE MATERIALS IN CONCRETE

Some of the waste materials of mining and manufacturing processes are rarely used for any purpose and merely collect or are stored on land that is thus wasted and unsightly. Fly ash, silica fume, and GGBFS were once merely waste products and are now used (after beneficiation) as valuable particulate ingredients in HCC. Certain mining operations set aside overburdens that may be composed of rocks that might be used when sized and sintered for lightweight aggregate. The utility industry has excess bottom slag that may be used as aggregate under controlled conditions. There has been much hypothesizing and some experimentation concerning the use of various other waste materials in HCC. It would solve many problems if the human race could use its nonrecyclable and excess crushed glass and plastic, garbage, waste paper, mildly radioactive waste, slag (all types), cinders, and other waste as aggregate in building materials. (We are already aware of some of the problems that might occur if the waste products used as aggregate are not stable in the concrete paste environment.) As yet, it seems that most technical people are skeptical of the stability, durability, and safety of HCC constructed with these kinds of aggregate. It is hoped that the future will provide better repositories for our waste than the ubiquitous landfill.

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