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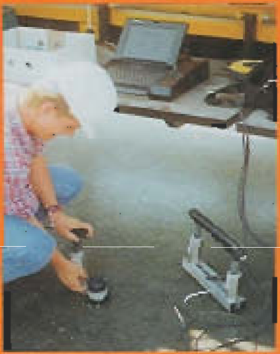

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Petrography – A Powerful Tool for Quality Assurance and Failure Investigation of Construction Materials

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Introduction

Petrography is the 150-year old science of geology that deals with the textural, compositional, and mineralogical description and classification of terrestrial (igneous, sedimentary, and metamorphic) and extraterrestrial (lunar and meteoritic) rocks. Classical petrography encompasses detailed macroscopical and microscopical examinations and supplementary physicochemical analysis needed for the detailed description and classification of natural rocks. Since clinker, cement, pozzolan, slag, aggregate, concrete, masonry units, mortar, grout, plaster, building stone, tile, terrazzo, and various other construction materials are essentially made using or processing various natural rocks, petrography has significant applications in the characterization and analyses of these materials. Since the mid-nineteenth century, petrography has been used for the analysis of portland cement – one of the most widely used construction materials in modern civilization. The application of petrography in the examination of concrete dates back to the early twentieth century. Despite its early heritage, it is has been only during the last 3 to 4 decades that the construction industry has started to accept this century-old science in the routine quality evaluation and particularly in failure investigation of various construction materials.

This article provides a detailed overview of the advancements and various applications of petrography in the construction industry.

Applications

Petrography has broad applications in the construction industry, such as in :

- **Materials Characterization** – Compositional, textural, and microstructural analysis of construction materials to determine and evaluate their bulk composition and ingredients.
- **Quality Evaluation** – Evaluation of the overall quality of a material; its integrity; assessment of durability and short and long-term performances; conformance to project specifications; evaluation of the effects of manufacturing and construction procedures on various properties of a construction material; and evaluation of the original material in a restoration project to determine a suitable repair material.
- **Failure Investigation** – Diagnosing various distresses in a material in service due to improper or inferior quality of the material; improper construction practices; inadequate design; and severe exposure conditions causing various physical and/or chemical attacks in the material during its service life.
- **Depth and Degree of Deterioration** – Determination of the depth or extent of deterioration in a structure and its severity prior to the assessment of repair and rehabilitation schemes.
- **Evaluating Materials, Methods, and Efficiency of Repair and Rehabilitation** – Evaluation of the performance of a repair material, suitability of a repair material and method in a project, extent of repair needed, condition evaluation before and after repair, and cause(s) of repair failures.

Construction Materials

Following is a list of various construction materials (and their ASTM specifications in parentheses) that can be examined in detail by petrography. Materials to be examined can be raw materials, powdered samples, fragments, saw-cut sections, drilled cores, broken pieces from a structure, crushed products, recycled products, etc.

- **Cementitious materials** including portland cement clinker, portland cement (ASTM C 150), blended hydraulic cement (ASTM C 595), fly ash (ASTM C 618, C 593), ground granulated blast furnace slag (ASTM C 989), silica fume or microsilica (ASTM C 1240), volcanic ash, metakaoline and other pozzolanic materials, masonry cement (ASTM C 91), stucco cement (ASTM C 1328), plaster cement (ASTM C 926), mortar cement (ASTM C 1329), hydraulic cement (ASTM C 1157), expansive hydraulic cement (ASTM C 845), etc.

- **Aggregates** used in concrete, mortar, grout, plaster, concrete masonry units, etc. that are classified as normalweight aggregate (gravel, crushed stone, and natural or manufactured sand; ASTM C 33, C 294), lightweight aggregates (air cooled slag, blast-furnace slag, volcanic cinder, scoria, and breccias), expanded clay, shale and slate, etc.; (ASTM C 330, C 331, and C 332), heavyweight aggregates (e.g., iron oxides), slag aggregates, and recycled concrete aggregates.

- **Portland cement concrete** and its various modifications, e.g., ready-mixed concrete (ASTM C 94); blended cement concrete; precast-prestressed-post tensioned concrete; fiber-reinforced concrete (ASTM C 1116); polymer-modified concrete (ASTM C 1439); high strength concrete; and high performance concrete. Portland cement concrete is by far the most widely examined construction material in petrography.

- Miscellaneous **portland cement-based products** such as portland cement plaster, stucco, (ASTM C 926), shotcrete (ASTM C 1141, 1436, and 1480), patching and anchoring grout, metallic and mineral surface hardener (dry shake), and portland cement overlay.

- **Clay Masonry Units** – Facing bricks (ASTM C 216), glazed bricks and tiles (ASTM C 126), building bricks (ASTM C 62), structural clay facing tiles (ASTM C 212), structural clay load-bearing and non-load-bearing tiles (ASTM C 34, 56), hollow bricks (ASTM C 652), thin veneer brick units, fire clay and high alumina refractory bricks (ASTM C 27), sewer and manhole bricks (ASTM C 32), insulating fire bricks (ASTM C 155), light and heavy vehicular traffic paving bricks (ASTM C 902, 1272), bricks for fireplaces (ASTM C 1261), chemical resistant bricks (ASTM C 279), silica refractory brick (ASTM C 416), and industrial floor bricks (ASTM C 410).

- **Concrete Masonry Units** – Concrete building bricks (ASTM C 55), sand-lime brick (ASTM C 73), load bearing concrete masonry units (ASTM C 90), nonload-bearing concrete masonry units (ASTM C 129), prefaced concrete masonry units (ASTM C 744), solid interlocking concrete paving units (ASTM C 936), concrete grid paving units (ASTM C 1319).

- **Stone Masonry Units** – Building Stones - Marble (ASTM C 503), limestone (ASTM C 568), granite (ASTM C 615), sandstone (quartz-based stone, ASTM C 616), and slate (ASTM C 629), dimension (ashlar) stone and rubble stone masonry units.

- **Mortars and Grouts for Unit Masonry** – Lime mortars, portland cement-lime mortars (ASTM C 270), masonry cement mortars (ASTM C 270), and grouts; evaluation of aggregates for masonry mortars (ASTM C 144) and grouts ASTM C 404); and estimation of proportions of cement, lime, and sand in masonry mortars (ASTM C 270).

- **Stone Products** – Building stones, stone claddings and veneers, stone pavers, natural stones, dimension stones, and various other decorative or architectural stone products not related to stone masonry.

- **Lime and Gypsum-based products** – Quicklime (ASTM C 5), lime and limestone (ASTM C 50, 51), hydrated lime (ASTM C 207), hydraulic hydrated lime (ASTM C 141), gypsum (ASTM C 22), anchoring grouts, gypsum plasters (ASTM C 28), gypsum casting and molding plaster (ASTM C 59), gypsum Keene's cement (ASTM C 61), gypsum wallboard (ASTM C 36), dry wall products, gypsum concrete (ASTM C 317), gypsum veneer plaster (ASTM C 587), etc.

- **Asbestos** – Detection of various forms of asbestos (serpentine and amphibole minerals, e.g., tremolite, antigorite, amosite, etc.) in building materials.

- **Tile, Terrazzo, and Other Floor Coverings** – Ceramic, vinyl, and quarry tiles; portland cement based and epoxy-based terrazzos; and other resilient floor coverings.

- **Architectural cast stones** (ASTM C 1384) from historic and architectural constructions.

- Many proprietary, **shrinkage compensating, fast setting, high early strength mortars and grouts** used for patching, repair, and anchorage in concrete (ASTM C 928, 1107).

- **Soil and backfill materials** – mineralogy and composition of surface clays and their potential for expansion.

A petrographer should have a sound knowledge of all these construction materials, their manufacturing processes, applications, properties, and specifications. Experience gained from petrographic examinations of a wide variety of each of these materials provides detailed knowledge and understating on the compositional and microstructural properties of these materials. Such experience is the best tool for materials characterization and failure investigation in regular consulting practices.

Instruments

Microscopical examinations stay at the nucleus of petrography, which is often supplemented with chemical and physical tests necessary for detailed description. The following instruments are commonly used for microscopical examinations of construction materials:

- **Stereo-microscope** (reflected-light microscope), where white light from an illuminator is reflected from an "as received," saw-cut, fractured, lapped, polished, or thin section of material and examined through objective and eyepiece lenses at magnifications up to 100 times.

- **Petrographic microscope,** where a "polarized" light is transmitted through a thin-section of a sample and through objective and eyepiece lenses and examined at magnifications up to 1000 times. Both crystalline and glassy materials can be identified; specific minerals are identified by their characteristic optical properties.

- **Fluorescence-light microscope,** where a petrographic microscope is modified by: (a) inserting an excitation filter between the sample stage and polarizer to generate a shortwavelength, ultraviolet light from a polarized-light, which creates fluorescence in the sample embedded in a fluorescent dye, and (b) inserting another barrier filter to block the long wavelengths. Fluorescence highlights the open spaces, cracks, and voids in a material; and based on the variation in degree of fluorescence, the spatial variations in the porosity of a material can be assessed.

- **Metallurgical microscope** (combined reflected and transmitted light modes), where polarized light is reflected from a polished section of a sample and examined at magnifications up to 1000 times. A petrographic microscope can be modified to provide transmitted polarized light, reflected light, and fluorescent light (in both transmitted or reflected modes) observations.

- **Scanning electron microscope** (SEM) equipped with a secondary electron detector, a backscatter electron detector, and an energy-dispersive x-ray spectrometer (EDS), where accelerated electron beams are focused to a small area on a saw-cut, fractured, lapped, polished, or thin-section of material and examined at magnifications in excess of 100,000 times. A secondary electron detector provides the detailed, three-dimensional morphology of material at a high resolution from low energy secondary electrons generated near the surface region of the material after the electron bombardment. Backscatter electrons reflect back from the sample surface after the bombardment of the primary electron and provide a detailed microstructure and differentiate phases of compositions from their average atomic numbers. An EDS detector captures characteristic x-rays of elements generated from the material, which helps determine the elemental or oxide compositions of a material at a small scale, at a point, in an area, along a line, or of an area as different elemental maps.

- **X-ray diffractometer,** which determines the presence and abundance of various crystalline components (especially the fine-grained phases) in a material.

There are various other instruments used for specific purposes such as infrared and x-ray microscopes for organic and elemental analysis, environmental SEM for studying samples in SEM in wet conditions, phase contrast and transmission electron microscopes for asbestos analysis, microhardness testers, etc. The above six instruments are used most commonly for analyses of construction materials and are usually adequate for most purposes.

Chemical analyses are done by following classical wet chemistry (gravimetric) or by using various modern instruments including atomic absorption spectrometer (AAS), infrared spectrophotometer (IR), inductive-coupled plasma spectrometer (ICP), x-ray fluorescence spectrometer (XRF), and others. Thermal analyses (differential thermal analysis, thermogravimetric analysis, and differential scanning calorimetry) and bulk porosity determinations (from mercury intrusion porosimetry or gas absorption studies) are also common in many investigations. These analytical techniques are beyond the scope of this article and can be found elsewhere (Ramachandran and Beaudoin 2001).

Sampling and Sample Preparation

Samples selected for petrographic examination must be representative of the larger sample or structure of the concern. The sampling strategy is more crucial in failure investigation, where samples from distressed and relatively less distressed or sound areas are usually selected for determining the cause and different degree and extent of deterioration. Following a detailed field investigation, samples should be collected from the distressed and relatively sound or less distressed areas in pristine conditions for laboratory analysis. The number of samples should be adequate to cover the area of interest. In most cases, drilled cores, saw-cut sections, or freshly broken pieces are collected. Concrete sampling can be done following the recommendations of ASTM C 823 "Standard Practice for Examination and Sampling of Hardened Concrete in Construction". Materials received for petrographic examinations are first examined visually in an "as received" condition, and then microscopically on freshly fractured sections, saw-cut sections, lapped sections, thin sections (0.2 to 0.3 mm thick, capable of transmitting light), polished sections, and oil immersion mounts using the microscopes described earlier. Materials should be adequately photographed to record the pristine condition prior to any destructive sample preparation procedures described below.

Depending on the nature of the material, various methods of sample preparation are followed in microscopical examinations to obtain as much compositional and microstructural information as possible.

- **Sectioning** for reducing a sample to a manageable size by using diamond saws.
- **Lapping or grinding** to achieve a smooth, flat surface using lapidary (e.g., cast iron) wheels and abrasives (e.g., SiC, alumina powder, diamond).
- **Thin sectioning** to observe the mineralogy and microstructure of a material by successive precision sectioning and controlled, fine grinding by hand or more efficiently by using thin-sectioning equipment.
- **Polishing** to achieve a shiny surface is done on polishing wheels with cloths and diamond or other fine abrasive pastes.
- **Etching or staining** a polished section with a chemical reagent highlights a particular component by absorbing into that component and removing a surface layer in the solution, or, in the latter, by reacting with the component of interest.
- **Powder mount or oil-immersion mount** preparation of material is used to examine the composition of a specific area of interest in a material. A needle is used to pick up a small portion of the sample, and its constituents are examined in a petrographic microscope by immersing it in an oil of a known refractive index. Minerals are identified by their characteristic refractive indices and other optical properties.

The method and extent of sample preparation should be based upon the kind of information needed for the purpose of the examination. Freshly fractured sections are helpful in studying the composition, properties, and conditions of a material in a stereomicroscope that have not been weathered or altered by the environment or other means. Lapped cross sections are useful for examinations of the overall composition and microstructure of a material at low (10X) to moderate (100X) magnifications by using a stereomicroscope. Thin sections are useful for examining finer details in composition, mineralogy, and microstructure in a petrographic microscope at magnifications of up to 1000X or in an SEM at much higher magnifications. Polished sections are useful for examination in metallurgical microscopes, staining/etching, and in SEM. Powder mounts or oil immersion mounts are useful in examining the composition of any small area of interest (usually in a freshly fractured section) from any location in the sample using a petrographic microscope or SEM. Saw-cut sections are sometimes used for either light or electron microscopes. Treating samples with a fluorescent or colored dye-mixed epoxy or alcohol can highlight cracks, voids, and porous areas. Epoxy impregnation improves the overall integrity of broken or fragile samples. Appropriate preparation of a sample is a crucial step in the examination, which, if not done properly, can destroy or prevent the gathering of useful information from a material.

Standards and Methodology

There are four industry standards from the American Society for Testing and Materials (ASTM) on petrographic examinations of aggregate, concrete, and mortar:

- **ASTM C 295** : "Standard Guide for Petrographic Examination of Aggregates for Concrete";
- **ASTM C 457** : "Standard Test Method for Microscopical Determination of Parameters of the Air-Void System in Hardened Concrete";
- **ASTM C 856** : "Standard Practice for Petrographic Examination of Hardened Concrete";
- **ASTM C 1324** : "Standard Test Method for Examination and Analysis of Hardened Masonry Mortar"

The European standards on petrography include: BSI 812, Part 104 "Method for qualitative and quantitative petrographic examination of aggregates" and BSI 1881, Part 124: "Methods for analysis of hardened concrete". The reference section lists several key articles on concrete petrography, which should be consulted for various petrographic methods (e.g., books and articles by Mather, Mielenz, French, St. John et al., Jana, and Walker).

Field reconnaissance is an important aspect of failure investigation and can provide valuable information for the preparation of a comprehensive petrographic report.

Beside microscopy, other common supplemental laboratory analyses for more detailed materials characterization include analyses of chloride, chloride permeability, sulfate, sulfide, oxide, cementitious materials contents, pH, abundance of various hydration products from thermal analyses, infra-red spectroscopic analysis of various organic materials, length change measurements for volume instability, compressive strength tests, freeze-thaw durability analysis, etc.

Quality and Compositional Evaluation of Cement, Aggregate and Concrete

Clinker microscopy is a tool used routinely in many cement-manufacturing plants for quality assurance and consistency in the production of clinker and cement. Procedures in clinker microscopy include: (a) examination of polished and etched/stained sections of clinkers in a reflected light microscope, (b) examination of oil immersion mounts of crushed clinker or cement and thin sections of whole or crushed clinker or cement in a petrographic microscope, (c) XRD of crushed clinker and cement, and (d) SEM examination of polished sections of clinker or cement (Campbell 1992). Microscopical determination of alite size, alite birefringence, belite color, and belite size by the Ono's method provides clinker burning conditions and a control on the production of good quality clinker and cement. Aggregate petrography, according to ASTM C 295, is a routine procedure followed in many aggregate plants for quality assurance. Routine quality assurance of concrete is not done by petrography unless there is a concern about some properties or ingredients in the concrete and their possible impact on short and long-term performance of the concrete. Some common applications of petrography for quality assurance of to-be-placed, newly placed, and old concrete are outlined below:

- Determination of the overall quality of concrete in an existing structure for assessment of its future short and long-term performance, and the impact of the existing condition in the future.
- Evaluation of aggregates to be used in concrete or mortar for potential alkali-aggregate reactivity, soundness, and frost resistance.
- Evaluation of concrete mixture proportioning, degree of mixing, retempering, consolidation, finishing, curing, and cement hydration, and their impact on the properties of newly placed concrete.
- Determination of the overall quality of concrete in an existing structure for assessment of chemical durability or its resistance to the affects of various deleterious chemical agents.

- Determination of air content and other air-void parameters of concrete for assessment of freeze-thaw durability and scaling resistance.

- Estimation of the amount of total cementitious materials used, proportions of various pozzolans, and water-cementitious materials ratio for assessment of strength of concrete in a new construction.

- Evaluating the reasons for low strength of laboratory cured concrete cylinders compared to the anticipated designed strength. Petrography is the most powerful method to determine the cause of low strength, which could be from high water content, high air content, low cementitious materials content, low degree of cement hydration, early freezing of concrete, or other reasons.

Quality and Compositional Evaluation of Masonry Units and Mortar

Although physical tests (e.g., compressive strength, cold and boiling water absorption, saturation coefficient, freeze-thaw durability, modulus of rupture, and efflorescence as in ASTM C 67) are the common industry-recommended procedures for evaluating the quality of various masonry units and their conformance to many industry specifications, petrography has significant potential for:

- Evaluation of the overall quality, condition, uniformity in composition, and volumetric proportions of ingredients in an existing lime, portland cement-lime, or masonry cement mortar to prepare a matching pointing mortar. The procedure for analysis of hardened mortar by petrography is outlined in ASTM C 1324.

- Evaluation of clay, stone, and concrete masonry units for their quality, composition, condition, suitability and anticipated performances in a project, short and long-term durability, performance, and behavior in an existing structure.

Quality and Compositional Evaluation of Stone and Tile Products

Petrography has significant potential in the evaluation of building and dimension stones; their overall quality, composition, and suitability in a particular project and environment; their freezethaw durability, and their resistance to other chemical and physical agents. Petrography can also be used to evaluate the density, hardness, compaction, and overall quality of terrazzo, ceramic, porcelain, stone, quarry tile, and other floor covering products.

Diagnosing Concrete Deteriorations

The most common application of petrography is in failure investigation of various concrete structures. Following is a list of various deteriorations in concrete and other portland cement based products that can be diagnosed by detailed petrographic examinations:

- Concrete surface distress such as scaling, spalling, aggregate popout, mortar lift-off, blistering, delamination, dusting, efflorescence, discoloration, and cracking.

- Chemical attacks in concrete by acid, alkali, sulfate, carbonate, and chloride solutions.

- Cracking and spalling due to alkali-aggregate reaction.

- Corrosion of reinforcing steel in concrete.

- Cracking, spalling, and loss of strength due to frost attacks in plastic and hardened concretes.

- Cracking, spalling, and discoloration due to fire attacks in concrete - the severity and extent of fire attack in concrete can be identified by petrography.

- Loss of mass and strength in concrete exposed to a marine envirotnment by the ingress of seawater containing dissolved magnesium sulfate, chloride, and carbon dioxide; corrosion of reinforcing steel in concrete by the dissolved chloride of seawater.

- Cracking due to delayed hydration of free lime and magnesia in the paste due to prolonged exposure of concrete to moisture.

- Cracking, spalling and exfoliation due to salt weathering and salt hydration distress.

- Cracking due to various mechanisms such as plastic shrinkage, drying shrinkage, plastic settlement, crazing, freezing and thawing, alkali-aggregate reaction, corrosion of reinforcing steel in concrete, sulfate attacks, heat of hydration, fire attacks, and delayed hydration of free lime and magnesia, etc.).

- Curling and cracking of a concrete slab-on-grade due to the thermal and/or moisture differential between the slab top and bottom.

- Low strength gain due to abnormal setting behavior (quick or delayed setting) of concrete.

- De-bonding of surface sealer, protective coating, paint, and overlays.

- Weathering, acid attacks, frost attacks, chloride and carbonation induced corrosion of wires, and other environmental attacks on underground prestressed concrete cylinder pipes and reinforced concrete pipes; microbial-induced sulfuric acid attacks in concrete sewer pipes.

- Various deteriorations in portland cement plasters (stuccos) such as cracking, discoloration, de-bonding of stucco from the substrate, corrosion of wire mesh backing, etc.

Each mechanism of deterioration leaves a series of characteristic compositional, macrostructural, and microstructural evidence that is commonly searched for during petrographic examinations of concrete. Identification of this evidence helps determine the cause and extent of deterioration.

Concrete Durability Evaluation

Similar to failure investigation, petrography is routinely used for assessing the durability of concrete structures. Following is a list of various applications of petrography in the field of concrete durability:

- Assessment of the **resistance of the concrete to external aggressive agents** from the overall quality of concrete including the estimated water-cementitious materials ratio, degree of cement hydration, degree of consolidation, finishing and curing practices employed, extent of cracking, and extent of chemical alterations of concrete.

- Assessment of **chloride-induced and carbonation-induced corrosion of reinforcing steel in concrete** from the chloride profile and depth of carbonation of the concrete in relation to the depth of the corroded steel, respectively. Assessment of the materials, thickness, and quality of the concrete cover over the reinforcing steel for preventing corrosion.

- Assessment of the **freeze-thaw durability of concrete** from air entrainment; the amount, size, fineness, distribution, and spacing factor of entrained air voids; frost resistance of aggregates (evaluation of aggregates for potential frost damage in concrete such as D cracking in pavement); density/porosity of the paste; volumetric proportion of the paste; evaluation of the results of laboratory cyclic freezing and thawing and deicing salt scaling resistance tests.

- Assessment of the severity and extent of various forms of **sulfate attacks in concrete** (e.g., external sulfate attack, internal sulfate attack, chemical sulfate attack, and physical sulfate attack) from various chemical, physical, and microstructural evidence such as: (a) the bulk sulfate content of concrete and sulfate profiles from the exposed surface inward; (b) the depth and degree of decomposition of the paste; (c) the presence of calcium sulfate dihydrate (gypsum); calcium sulfoaluminate hydrates (ettringite, monosulfate); sulfate salts (sodium, calcium sulfate hydrates) and other products of sulfate attacks in the concrete, (d) extent of microcracking; and (e) the overall loss of mass and strength of the concrete.

- The classical **external (chemical) sulfate attack** occurs in concrete structures exposed to sulfate-rich environments that contain solutions of sodium, calcium, magnesium, or ammonium sulfates. Sulfates from the environment enter into the concrete through cracks or through capillary pore spaces (in permeable concrete); the solutions react with the cement hydration products (e.g., calcium hydroxide, calcium aluminate hydrate, monosulfate) and form gypsum and/or ettringite. The formation of these reaction products causes expansion, which if occurs in the confined spaces in the paste, can cause cracking. Severe cracking eventually causes spalling, loss of mass, and loss of strength. Sulfate attack is usually identified by combined petrographic and chemical analyses, which determine: (a) the presence of a higher amount of sulfate in concrete than that contributed from the portland cement, and (b) the presence of gypsum and ettringite in the cracks, voids, and in confined spaces in the paste.

- **Internal sulfate attacks** occur in the presence of high sulfate in aggregates (in *sulfate* form such as gypsum or anhydrite or in *sulfide* form such as pyrite or marcasite), and/or from sulfates in the paste that have been initially absorbed into the hardened paste, subsequently released in the presence of moisture, and precipitated in confined spaces in the paste causing expansion and subsequent cracking. Delayed ettringite formation (DEF) is such an internal sulfate attack. It occurs when sulfate is forcefully absorbed into the hardened paste during high-temperature curing of precast concrete, or, in concrete containing portland cements with high sulfate contents, or in concrete made using other high-sulfate cement. The absorbed sulfates in the hardened paste subsequently dissolve out during prolonged exposure of the concrete to the moisture. The dissolved sulfates produce ettringite in the hardened paste by reacting with the cement hydration products, which causes cracking and peripheral separations around the aggregate particles due to the bulk expansion of the paste relative to the aggregates. The gaps around the aggregates are often partially or completely filled with secondary ettringite precipitates.

- **Physical sulfate attack,** sometimes also called physical salt attack or salt hydration distress, occurs in concrete structures exposed to a sulfate-rich environment and in fluctuating temperatures and relative humidity conditions that cause reversible phase transformations of water-soluble sulfate salts between different hydration states. The transformation from an anhydrous or a less hydrous state to a more hydrous state (e.g., from thenardite, Na_2SO_4 to mirabilite $Na_2SO_4.10H_20$) causes a significant solid volume expansion, which can cause scaling, exfoliation, spalling, and cracking of the concrete. This type of attack is also common in many stone masonry structures exposed to a sulfaterich soil. The sulfate salts precipitate as white efflorescence deposits on the exposed surfaces of concrete. Sulfate solution usually moves upward through the capillary pores of concrete or stone along the surface region by osmosis. Petrography is the best method to use in diagnosing this type of attack by analyzing the salts using optical microscopy, SEM, and x-ray diffraction.

- Assessment of the extent and damaging action of **alkali-aggregate reactions** in concrete from: (a) the type, composition, location, and abundance of alkali-silica gel in the paste, cracks, and voids in the concrete; (b) reaction rims in potentially alkali-silica or alkali carbonate reactive aggregate particles; and (c) microcracking in concrete extending from reactive aggregate particles into the paste. The presence of potential alkali-silica reactive particles (chert, flint, chalcedony, tridymite, cristobalite, volcanic rocks, strained quartz and quartzite, greywacke, and glass), alkali-carbonate reactive particles (calcitic dolomite and dolomitic limestone containing large euhedral crystals of dolomite in a fine-grained matrix of calcite, dolomite, quartz, and clay), and the evidence and products of their reactions with cement alkalis in pore solutions of concrete can be diagnosed only by petrography.

- Assessment of **acid-induced corrosion** of concrete from the depth of alteration of the cement paste; leaching, decomposition, and loss of the calcium hydroxide component of cement hydration from the paste; and the degree of dissolution of calcareous aggregates by acidic solutions.

- Assessment of **abrasion resistance of concrete** from hardness, density, degree of curing, water-cementitious materials ratio, type of aggregates, and the presence of hardener at the wearing surface (and in near-surface region) of the concrete.

Concrete Repair and Rehabilitation

Since proper diagnosis of a problem is the key to successful repair or rehabilitation, petrography has significant applications in repair technology from the stage of implementation of a strategy to the effectiveness and performance of a repair material (Jana 2005).

- Diagnosing the causes of concrete deterioration prior to the repair.
- Determining the extent and severity of the deterioration and the amount of deteriorated concrete to be repaired.
- Evaluating a suitable repair material for a particular deterioration.
- Evaluating the preparation of the surface of the deteriorated concrete substrate prior to the placement of the repair material.
- Evaluating the bond between a repair material and the original concrete.
- Determining the degree of infiltration of a repair material in the cracks.

- Evaluating the effectiveness of a proposed repair scheme in a small test area.
- Evaluating the improvement of the overall condition of a structure by a repair material and method by comparing the conditions before and after the repair.
- Investigating the causes of a repair failure.

Diagnosing Problems in Clay, Stone, and Concrete Masonry Structures

Petrographic examinations of masonry units and mortars can provide important information during investigation of the following masonry problems:

- Water leakage through masonry.
- Cracking and spalling of jointing mortars and masonry.
- Reasons for an inadequate bond between masonry units and jointing mortars.
- Efflorescence, staining, and other discoloration on masonry walls.
- Disintegration of jointing mortars, grouts, and sealants.
- Distress in a masonry wall and foundation due to cyclic freezing and thawing.
- Evaluating the effectiveness of a surface sealer or coating or the reasons for coating failure on a masonry wall.
- Distress in dimension stone cladding such as volume instability (bowing or dishing), cracking, discoloration, de-bonding, moisture and/or thermal hysteresis, etc.
- Deterioration of clay, stone, and concrete pavers from cyclic freezing and thawing, wetting and drying, heating and cooling, abrasion, impact, disintegration, heaving, and efflorescence.

Diagnosing Distress in Tiles and Other Floor Coverings

During the past year, the author has used petrography in various investigations of tile and floor covering failures such as:

- Distress in ceramic tile floors such as cracking, de-bonding from the setting bed mortar, popping, discoloration, disintegration of the setting bed mortar and jointing grouts, and moisture penetration related distress.
- Distress in vinyl tile floors such as discoloration, bumps, blisters, and de-bonding from the substrate due to moisture entrapment, or adhesive failure.
- Distress in terrazzo floors due to cracking, de-bonding of terrazzo from setting bed mortar, discoloration, marble chipping, and other deteriorations.
- De-bonding of resilient floor coverings due to high moisture emission through the concrete substrate, alkali-aggregate reaction in the concrete substrate (which is often concentrated at the surface region by moisture upwelling and moisture-induced alkali enrichment at the surface region), adhesive failure, or other reasons.
- Blistering, cracking, crazing, and de-bonding of paint from various substrates ranging from concrete floors to drywall.

Diagnosing Miscellaneous Deteriorations

In addition to examinations of cementitious materials, aggregate, concrete, mortar, masonry, tile, stone and other products, petrography has also been used in the following cases:

- Examination of soil and other subbase materials for the presence of potentially expansive materials in the presence of moisture (e.g., certain clays, metallic materials, iron sulfide).
- Following the above line of examination, investigation of possible reasons for heaving of concrete or asphaltic pavements (either frost-induced or moisture related expansion of subbase materials).
- Comparing an unknown cementitious material from a sewage pipe with a known material to investigate the alleged blockage of the sewage line by the unknown material.
- Examinations of various slag products (steel slag, blast-furnace slag, air-cooled slag, granulated slag) for their potential use in concrete and in other building materials and their potential unsoundness.
- Examination of fireproofing materials; asbestiform minerals for possible health hazards; galvanized metals for possible corrosion-induced deteriorations, etc.

- Investigation of plaster deteriorations in many in-ground swimming pools, such as spot etching, cracking, staining, discoloration, spalling, blistering, etc.

Petrographic Report

A petrographic report is a concise and informative document of petrographic examinations of a material that provides answers to the purpose of the investigation based on which an engineer, architect, or others decide the future course of action. The report should include a detailed **descriptive section** about the project background, field reconnaissance (if any), descriptions (appearance, condition, integrity, weight, dimensions) and photographs of samples as received, methodology applied, overall composition and microstructure of the material and its components, and chemical and/or microstructural evidence of any deficiency or deterioration. Based on the accumulated petrographic data, an **interpretative section** should put the findings together into meaningful answers as to the purpose of the examination. The accuracy and adequacy of the descriptive and interpretative sections depend upon the depth of knowledge of the petrographer in: (a) classical petrography, (b) the behavior and properties of construction materials, (c) construction procedures and their impact on the properties and performance of the material, (d) the effects of the environment on the material, (e) the extent and details of the examination, and (f) his or her ability to interpret the descriptive findings in a scientific manner.

Depending on the material being examined, the descriptive section should include items such as:

- The purpose and scope of the investigation, background information about the project, existing field conditions from the client or from field reconnaissance, age, construction procedures used, weather conditions during construction, any relevant documents related to the project, and sampling locations.
- Field observations and reports of the condition survey, if any; field photographs of the condition of the structure.
- Sampling – overall condition of the sample as received, dimensions, weight, visible distress (if any), and photographs of samples prior to any sample preparation.
- Methods and instruments used during examinations; and techniques of sample preparations employed.
- Aggregates – Type, lithology, mineralogy, shape, roundness or angularity, grading, distribution, type and amount of reactive particles, evidence of alkali-aggregate reaction in the concrete, soundness, frost resistance, coating, alteration, and cracking.
- Hardened Cement Paste – Color, density, hardness, porosity, water drop absorption, texture and luster of freshly fractured surfaces; presence and estimated volumetric proportions of residual portland cement and other cementitious materials; size, shape, distribution and proportions of calcium hydroxide crystals of cement hydration; degree of cement hydration; presence of fly ash, slag, or silica fume particles; evidence of any chemical alteration or decomposition of paste; severity and depth of carbonation; and estimated water-cementitious materials ratio from textural and compositional properties of paste.
- Air – Proportion of entrained and entrapped air, air void size, shape, and distribution; the overall air-void system; and the efficiency of the air-void system in providing the necessary resistance of the paste against distress due to cyclic freezing and thawing.
- Interfaces – The nature of transition zones between the paste and other fillers in the concrete such as aggregates, reinforcing steel, fibers, etc.; intimate or weak aggregatepaste bond; importance of interfacial zones in the properties and performance; improvements of transition zones using various microfillers in the concrete.
- Secondary Deposits – Type, location, distribution, composition, mineralogy, amount, depth of occurrence, and the significance of their presence.
- Cracking – Macro and micro cracking, surface width, depth, and configuration of cracks; deposits in cracks, evidences associated with cracks regarding the cause(s) of cracking.
- Degree of consolidation of the concrete; evidence of honeycombing; retempering; the presence of any cold joints; contraction, isolation or construction joints – depth, width, presence or absence and type of joint sealant; evidence of plastic freezing of concrete;

evidence of inadequate curing of the concrete; evidence of improper finishing manipulations.

- Evidence of various physical and chemical deteriorations in concrete such as the products of reactions between the external chemical agents (e.g., acid, alkali, sulfate, salt, seawater) and cement hydration products, cracking, loss of mass, characteristic microstructural features of a typical deterioration, discoloration, chemical alterations, etc.

- Miscellaneous shrinkage-compensating, fast-setting, high early strength proprietary and repair materials – composition, mineralogy, texture, microstructure, evidences of distress or abnormalities, evidence of potential expansion, and results of length change measurements.

- Portland Cement Plaster (Stucco) – Thickness of scratch (base) coat, brown coat, and finish coat; thickness conformance to industry recommendations (ASTM C 926); bond between the coats; de-bonding; cracking; composition and mineralogy of individual coats; aggregate types, composition, mineralogy, soundness, reactivity, and other properties, properties of paste, and air content in each coat; evidence of chemical alterations, freezing, corrosion of metal lath, tearing of building paper, and other problems in the plaster; deterioration of plaster in swimming pools.

- Masonry Mortar – Estimated volumetric proportions of portland cement or masonry cement, sand, and hydrated time in mortar; type of mortar; evidence of plastic freezing of mortar; degree of hydration; type, grading, color, density, composition, and soundness of sand aggregate; composition of cementitious materials used; physical properties of paste; degree of carbonation; oversanded or under sanded nature of the mortar; evidence of loss of water due to absorption in masonry units; cracks transecting mortar or along mortarmasonry interfaces.

- Masonry units – Type, color, density, absorption, saturation coefficient, efflorescence, composition, unit dimensions, surface texture, bond to jointing mortars, cracks, spalls, and evidence of any other distress.

- Lime and Gypsum-based products – Texture, mineralogy, chemical composition, and microstructure of these products; evidences of any deteriorations.

- Photographs and photomicrographs of samples depicting various features mentioned in the report. Photographs are not only helpful in describing various textural and compositional features of a material but also provide a better visualization of the evidences responsible for the distress. Photographs taken at various scales should be an integral part of a good petrographic report. Photographs should be taken in the field using a 35 mm or digital camera, and in the laboratory of the "as received" sample using a digital camera or scanning the pristine or lapped sections of samples in a flatbed scanner. Photomicrographs should be taken by attaching a commercial or research-grade digital camera in the trinocular heads of a stereomicroscope, petrographic (and fluorescent-light) microscopes, and by using appropriate digital imaging attachments in an SEM-EDS. Good photo editing and image analysis software are needed to enhance and emphasize the photographs and areas of interest.

The interpretative section of the report should address various issues such as :

- Understanding the meaning and significance of field and laboratory findings in relation to the purpose of the examination and whether or not the information obtained from the descriptive section adequately addresses the purpose of the examination.

- Diagnosing the causes of failure or undesirable performance of a material or a structure from the background information, field evidences (if any), and the detailed findings of petrographic examinations.

- Usually, a report does not provide recommendations for future repair schemes or courses of action unless it is based on consultation with (and approval of) the engineers, architects, or other personnel involved in the project.

Limitations

Despite its wide-ranging applications, like any other branch of science, petrography has certain limitations, which should be remembered during investigation of a material:

- Petrography provides a semi-quantitative estimation of the proportion of cementitious materials and the water-cementitious materials ratio of hardened concrete. The accuracy of this determination depends upon the number of samples examined, the construction practices, the type and number of reference materials used for comparison of the water-cementitious materials ratio, the degree of cement hydration, spatial variations of these properties in a structure, experience, and other factors. The strength of a material cannot be assessed without direct or in situ strength testing. Petrography, however, can determine whether or not the strength of a material is higher or lower than the strength of another similar material and the reasons for strength variations.

- The types of chemical admixtures and various property-enhancing organic chemicals added in a construction material cannot be assessed without doing the appropriate supplementary chemical analysis. However, the effects of such chemicals on the microstructure and properties of materials can be assessed.

- The absolute age of a material cannot be assessed. The relative age of cracks in a structure in the same concrete and exposure conditions can be assessed from determining the depths of carbonation.

- The arial (lateral) extent of deterioration cannot be assessed without a field and nondestructive survey or adequate sampling of the entire area. The extent of deterioration at a particular location, however, can be assessed from a core.

- Depending upon the project and the purpose of the examinations, the extent of information obtainable from petrographic examination may depend upon the sampling strategy (e.g., number, location, and extent of sampling); therefore, caution is needed to extend the conclusions from one or a couple of samples to the broader scale in the structure.

Conclusion

This article provides a broad outline of various applications of petrography in the construction industry. Table 1 provides a list of various properties and mechanisms of deteriorations than can be examined and diagnosed by petrography and other conventional methods of testing. The most common application of petrography is in failure investigation, which provides a wealth of information for evaluating future performance and possible repair schemes. Many cement, aggregate, masonry, stone, and ready mixed concrete industries use petrography for routine quality assurance of their respective products. Hopefully, this article should increase the general awareness among engineers, architects, and other construction individuals about the importance of petrography in quality assurance and especially in failure investigation of construction materials and structures.

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References

ASTM C 295 (1998): Standard Guide for Petrographic Examination of Aggregates for Concrete, In: ASTM Vol. 4.02 (Concrete and Aggregates), Philadelphia, USA, American Society for Testing and Materials.

ASTM C 457 (1998): Standard Test Method for Microscopical Determination of Parameters of the Air-Void System in Hardened Concrete, In: ASTM Vol. 4.02 (Concrete and Aggregates), Philadelphia, USA, American Society for Testing and Materials.

ASTM C 823 (2000): Standard Practice for Examination and Sampling of Hardened Concrete in Constructions In: ASTM Vol. 4.02 (Concrete and Aggregates), Philadelphia, USA, American Society for Testing and Materials.

ASTM C 856 (2004): Standard Practice for Petrographic Examination of Hardened Concrete, In: ASTM Vol. 4.02 (Concrete and Aggregates), Philadelphia, USA, American Society for Testing and Materials.

Table 1: Importance of petrographic examinations

	Visual Examination (1)	Nondestructive Testing (2)	Chemical Analysis (3)	Physical Testing (4)	Petrography (5)
Abnormal setting of cement			■		■
Acid attack			■		■
Aggregate – Type, Grading					■
Aggregate soundness & reactivity			■	■	■
Aggregate popouts	■				■
Air content – Hardened					■
Air-void System					■
Alkali attack			■		
Alkali-aggregate reaction			■	■	■
Autogenous healing of cracks					■
Backfill – Potential expansion			■		■
Cement and concrete burns			■		■
Cementitious materials – types and proportions			■	■	■
Chloride content			■		
Chloride permeability			■		■
Compressive Strength		■		■	
Corrosion of Steel in Concrete	■	■			■
Cracking due to expansion	■	■		■	■
Cracking due to shrinkage	■			■	■
Consolidation	■	■			■
Delamination	■	■			■
Density, Unit weight, Specific Gravity				■	
Depth and condition of reinforcing steel in concrete	■	■			■
Depth of carbonation					■
Depth of penetration of epoxy in cracks		■			■
Discoloration	■		■		■
Disintegration	■	■			■
Durability	■	■	■	■	■
Efflorescence	■				■
Fire attack	■			■	■
Floor covering failure	■		■	■	■
Voids, Honeycombing		■			■
Low strength and low strength gain		■		■	■
Masonry efflorescence			■		■
Masonry water leakage	■	■			■
Masonry mortar failure	■	■	■		■
Masonry unit (clay, stone, and concrete) - properties and distress	■	■		■	■
Moisture content		■	■		

Pavement deterioration	■	■	■	■	■
Permeability				■	■
Repair patch failure	■		■		■
Resistance to freezing and thawing				■	■
Scaling of concrete surface	■				■
Scaling resistance				■	■
Spalling of concrete	■	■			■
Sulfate attacks	■		■		■
Stucco (plaster) failure	■		■		■
Sulfate resistance			■	■	■
Swimming pool deterioration	■		■		■
Tile debonding, cracking	■			■	■
Terrazzo failure	■				■
Uniformity	■	■			■
Relative age from depth of carbonation					■
Water-cement ratio			■		■
Weathering resistance	■	■		■	■

(1) Visual examinations include observations made during the condition survey, preliminary and detailed examination of the structure from where the sample was taken, and detailed examination of the sample as received, prior to any sample preparation.

(2) Nondestructive testing includes Acoustic Impact and Emissions, Gamma Radiography, Rebound Hammer, Ultrasonic pulse, Windsor probe, Infrared Thermography, Radar, Chain drag survey, and other tests.

(3) Chemical Analyses include: analysis of cement content, chloride content, sulfate content, elemental analysis, and determination of moisture content.

(4) Physical testing includes testing of compressive, flexural and tensile strengths, length change measurements, freeze-thaw durability measurement, accelerated wetting-drying, heating-cooling, freezing-thawing tests, rapid chloride permeability test, etc.

(5) Petrography includes examinations using light and electron optical microscopes, x-ray diffraction, and supplementary chemical analysis.

ASTM C 1324 (2004): Standard Test Method for Examination and Analysis of Hardened Masonry Mortar, In: ASTM International's Masonry Standards for the Building Industry, Fifth Edition, Philadelphia, USA, American Society for Testing and Materials.

BSI 1881 (1988): Testing concrete Part 124, Methods for analysis of hardened concrete.

BSI 812 (1994): Method for qualitative and quantitative petrographic examination of aggregates, Part 104.

Campbell, D.H. (1992): Microscopical examination and interpretation of portland cement and clinker, Construction Technology Laboratories, Skokie, Illinois, USA, Portland Cement Association.

French, W.J. (1991): Concrete Petrography: A Review, Quarterly Journal of Engineering Geology, 24 (1): 17-48.
Jana, D. (2005) Petrography and Concrete Repair – A Link is Needed, Concrete International, 27 (1): 37-39.

Jana, D., and Cole, A.A. (1997): Microscopy – A Practical Solution to Concrete Problems, Concrete Industry Board Bulletin, 33 (3): 18-22.

Jana, D. (2005): Concrete Petrography – Past, Present, and Future, 10th Euroseminar on Microscopy Applied to Building Materials, Scotland.

Mather, K. (1952): Applications of light microscopy in concrete research, Symposium on Light Microscopy, ASTM STP 143: 51-70.

Mather, K. (1966): Petrographic Examination of Hardened Concrete, In: Symposium on Significance of Tests and Properties of Concrete and Concrete-Making Materials, ASTM 169A, Am. Soc. Testing. Mats: 125-143.

Mielenz, R.C. (1962): Petrography Applied to Portland-Cement Concrete, In: T. Fluhr and R. F. Leggert (eds.), Reviews in Engineering Geology, The Geological Society of America, Vol. 1: 1-38.

Mielenz, R.C. (1964): Diagnosing Concrete Failures, Stanton Walker Lecture Series on the Materials Science, University of Maryland, College Park, Maryland, USA: 5-47.

Mielenz, R.C. (1994): Petrographic Evaluation of Concrete Aggregates, In: Klieger, P. and Lamond, J.F. (eds.), Significance of Tests and Properties of Concrete and Concrete-Making Materials, ASTM STP 169C, American Society for Testing and Materials, Philadelphia, USA: 341-364.

Ramachandran, V.S., and Beaudoin, J J., eds (2001): Handbook of Analytical Techniques in Concrete Science and Technology, Noyes Publications, New Jersey.

St. John, D.A., Poole, A.W., and Sims, I. (1998): Concrete Petrography – A handbook of investigative techniques, London, Arnold.

Walker, H.N. (1992): Petrographic Methods of Examining Hardened Concrete: A Petrographic Manual, Virginia Transportation Research Council, Richmond, Virginia, USA: 1-286.

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