Application of Petrography and Other Methods in Quality Assurance & Failure Investigation of Construction Materials

Dipayan Jana, President
Construction Materials Consultants, Inc.
www.cmc-concrete.com
Strategy used in Quality Assurance & Failure Investigation of Construction Materials

Background Information, Communication

Techniques, Examinations, Investigation

- Field Investigation, Photographs & Sample Selection
- Petrographic Examination
- Chemical Testing
- Physical Testing
- Specialty Testing

Data Interpretation and Report Preparation
Petrography

Literally: 150-year old discipline of Geology, which deals with the description and classification of natural (igneous, sedimentary, & metamorphic) rocks. [Greek Petra = Rocks & Graphics = Picture]

**Concrete is a man-made rock**

Broadly: The science of observation and description of a material – Its composition, texture, microstructure, integrity, and overall quality

**Tools:** Light optical microscopes, Electron microscopes, X-ray diffractometer

Basic

Advanced

There are two systems in the Universe – Geology & Theology – Petrography is the connecting link.
Concrete Petrography

Application of petrography in the description of concrete and concrete-making materials, which include:

- Portland cements
- Fly ash, Ground granulated blast furnace slag, Silica fume, Metakaoline, Natural pozzolans, Microfillers
- Blended cements
- Other cementitious materials, e.g. high alumina cement, expansive cements
- Aggregates: Natural, Manufactured, Gravel, Crushed stone, Sand, Slag, Lightweight, Heavyweight, Recycled concrete
- Portland cement based concrete, mortar, plaster (stucco), grout, shotcrete, pipe, brick, block, paver
- Air-void analysis
- Special concretes: Polymer-modified, Fiber-reinforced, Shrinkage-compensating, Lightweight, High performance
- Gypsum and lime based products – mortar, plaster, grout
- Clay, stone, and concrete masonry units and mortars
- Dimension stones, tiles, terrazzo, anchoring grouts, fill materials

Best Technique to Evaluate Concrete Quality & Durability
Concrete Petrography – Modes of Examination

Structure (Field Survey)

Concrete core, Other samples (Visual Exam)

Fractured, thin, & Lapped sections (SEM)

Increasing Magnifications

Fractured & Lapped sections (Stereomicroscope)

Thin-sections; Mounts (Petrographic Microscope)
Concrete Petrography – A Few Applications

- Aggregate size, grading, quality, reactivity
- Cement type, grain-size, degree of cement hydration
- Water-cementitious materials ratio (Strength, Durability)
- Density, porosity, permeability (Strength, Durability)
- Interfaces in concrete – Aggregate-Paste interface (Strength)
- Depth of carbonation (Corrosion of steel)
- Air void system, Air content (Strength, Freeze-thaw durability)
- Cracking due to shrinkage or expansion; Shrinkage potential (Curling)
- Chemical alterations by acid / sulfate / CO₂ / leaching
- Physical alterations by fire or frost attack
- Diagnosing causes of various surface distresses (scaling, spalling, delamination)
- Effects of various pozzolans and other chemical admixtures in densifying the concrete
- Type, composition, overall quality
<table>
<thead>
<tr>
<th>Field Investigation &amp; Physico-chemical Testing</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Field Survey</strong></td>
</tr>
<tr>
<td>- Condition survey (ACI 201)</td>
</tr>
<tr>
<td>- Surface distress (ACI 201)</td>
</tr>
<tr>
<td>- Delamination survey (ASTM D 4580)</td>
</tr>
<tr>
<td>- Relative Humidity Profile (ASTM F 2170)</td>
</tr>
<tr>
<td>- Moisture vapor transmission rate (ASTM F 1869)</td>
</tr>
<tr>
<td><strong>Chemical Tests</strong></td>
</tr>
<tr>
<td>- Oxide Analysis</td>
</tr>
<tr>
<td>- Cement content</td>
</tr>
<tr>
<td>- Chloride Analyses</td>
</tr>
<tr>
<td>- Chloride Profile</td>
</tr>
<tr>
<td>- Sulfate Analysis</td>
</tr>
<tr>
<td>- Sulfate Profile</td>
</tr>
<tr>
<td>- Organic Admixtures</td>
</tr>
<tr>
<td>- Sealer analysis</td>
</tr>
<tr>
<td><strong>Techniques</strong></td>
</tr>
<tr>
<td>Wet chemical, Gravimetric, XRF, FT-IR, AA, DTA, TGA, DSC</td>
</tr>
<tr>
<td><strong>Physical Tests</strong></td>
</tr>
<tr>
<td>- Compressive strength</td>
</tr>
<tr>
<td>- Flexural strength</td>
</tr>
<tr>
<td>- Split-tensile</td>
</tr>
<tr>
<td>- Modulus of Rupture</td>
</tr>
<tr>
<td>- Length change</td>
</tr>
<tr>
<td>- Alkali-aggregate reactivity (AAR)</td>
</tr>
<tr>
<td>- Specific gravity, Absorption</td>
</tr>
<tr>
<td>- Freeze-thaw durability</td>
</tr>
<tr>
<td>- Deicing salt scaling</td>
</tr>
<tr>
<td>- Chloride permeability</td>
</tr>
<tr>
<td>- Blaine Fineness</td>
</tr>
<tr>
<td>- Sieve Analysis</td>
</tr>
<tr>
<td>- Sulfate soundness</td>
</tr>
<tr>
<td>- Abrasion Resistance</td>
</tr>
<tr>
<td><strong>Specialty Tests</strong></td>
</tr>
<tr>
<td>- Accelerated weathering [Wetting-drying, Freezing-thawing, Heating-cooling cycles]</td>
</tr>
<tr>
<td>- Aggregate-induced staining</td>
</tr>
<tr>
<td>- North Carolina test for corrosion inhibitor</td>
</tr>
<tr>
<td>- Penn-DOT air-void analysis</td>
</tr>
<tr>
<td>- NYC Freeze-thaw tests</td>
</tr>
<tr>
<td>- Project-specific tests</td>
</tr>
</tbody>
</table>
Concrete & Concrete-Making Materials
- Quality Assurance

(1) Aggregate quality (ASTM C 33, C 295)
(2) Aggregate reactivity (ASTM C 289, C 586)
(3) Portland cement (ASTM C 150)
(4) Pozzolan (ASTM C 685, C 989)
(5) Air-void system (ASTM C 457)
(6) Pozzolan - Type and amount
(7) Water-cementitious materials ratio (w/cm)
(8) Contaminants
(9) Freeze-thaw resistance (ASTM C 666)
(10) Deicing salt-scaling resistance (ASTM C 672)
(11) Chloride Permeability (ASTM C 1202)
(12) Potential expansion of hardened concrete by alkali-aggregate reaction (ASTM C 1260, C 1105)
(13) Potential expansion of concrete in moisture
(14) Fiber – Presence, Type, and Amount
(15) Surface Sealer – Presence & Depth
(16) Quality of concrete cover over reinforcement
(17) Concrete - Topping bond; Bonding agent
(18) Degree of consolidation
(19) Retempering
(20) Joint spacing, timing of installation, depth, rebar depth and location and other design issues in a slab
(21) Depth of extent of distressed concrete for removal
(22) Overall quality and integrity
Concrete & Concrete-Making Materials
- Failure Investigation

(1) Scaling
(2) Popout
(3) Mortar flaking
(4) Abrasion
(5) Dusting
(6) Discoloration, Staining
(7) Blistering
(8) Delamination
(9) Spalling
(10) Corrosion of steel in concrete
    (Carbonation-induced & Chloride-induced)
(11) Frost attack – Early freezing, Late freezing
(12) Cracking – Shrinkage cracks, structural cracks
(13) Curling
(14) Low strength
(15) Alkali-aggregate reaction - Cracking
(16) Acid attack – Loss of mass & strength
(17) Sulfate attack – Loss of mass & strength, Cracking
(18) Delayed hydration of free lime & magnesia
(19) Seawater attack
(20) Physical salt attack - Scaling
(21) Leaching & Efflorescence – Surface discoloration
(22) Fire attack – Cracking, Loss of strength
(23) Concrete burn
(24) Setting-related – Delayed, Accelerated, False, Flash
(25) Concrete pipe (Sewage, PCCP) distress
(26) Concrete chimney distress

The list goes on .......
**QUALITY ASSURANCE**

1. Clay Masonry Unit (Brick, Structural tile) -
   - Compressive strength, Absorption, Freeze-thaw durability, Efflorescence, Overall quality
2. Concrete Masonry Unit (Brick, Black, Paver) -
   - Compressive strength, Absorption, Freeze-thaw durability, Efflorescence, Overall quality
3. Stone Masonry Unit -
   - Compressive strength, Absorption, Freeze-thaw durability, Efflorescence, Overall quality
4. Mortars (ASTM C 1324) -
   - Portland cement – Lime Mortar (ASTM C 270)
   - Masonry & Mortar cement Mortar (C 91)
   - Lime Mortar
   - [Composition, Type (M-S-N-O), Strength, Sand-cement ratio, Overall quality, Comparison to existing mortar]

**FAILURE INVESTIGATION**

1. Bonds between existing masonry unit & mortar
2. Water tightness/leakage
3. Sealer – Presence & Depth
4. Mortar – Strength, Composition, Type, Comparison, Condition, Quality (C 1324)
5. Cracking
6. Efflorescence
Flooring, Coating, & Plaster
Materials Characterization & Failure Investigation

Flooring
- Ceramic & Quarry Tile
- Vinyl Tile
- Clay Tile
- Stone Tile
- Terrazzo
- Cracks, Bumps, Blisters, Discoloration, De-bonding, Curling

Coating
- Paint Failure (peeling, cracking)
- De-bonding between paint and drywall
- De-bonding between paint and concrete

Plaster
- Portland cement plaster (Stucco)
- Gypsum Plaster
- Lime Plaster
- Cracking, Shrinkage, De-bonding
Building/Dimension Stone – Limestone, Granite, Marble, Slate

Fill Materials (Slag, CLSM) – Potential for Expansion

Fast setting, shrinkage-compensating grouts – Expansion, lifting, cracking, spalling

Expansive cementitious materials

Shotcrete (Gunite)

Gypsum based products

Lime based products

Patching materials

Architectural Cast Stone

Insulating materials (Foam concrete, Zonolite grout, etc.)
A close look to some common concrete problems
Scaling – A common problem in concrete slabs

- “Local flaking or peeling away of the near surface portion of hardened concrete or mortar”

ACI Committee 116 (Cement and Concrete Terminology)

**Light** = Does not expose coarse aggregate (CA)

**Medium** = Loss of surface mortar to 5-10 mm in depth and exposure of CA

**Severe** = Loss of surface mortar to 5-10 mm in depth with some loss of mortar surrounding CA 10-20 mm in depth

**Very severe** = Loss of CA and surface mortar to a depth > 20mm
(1) Poor air entrainment
- No ‘entrained’ air, or, Low amount of ‘entrained’ air in concrete, or Low air at the surface
- Inadequate specific surface of air voids (<600 in²/in³)
- Large void spacing factor (>0.008 in.)
(2) **Finishing Improprieties**

- Premature finishing prior to the cessation of bleeding
- Finishing improprieties (e.g. prolonged finishing) that reduce air content at the surface

*Sheet Scaling by Premature finishing Before the cessation of bleeding*
(3) **Deicing salts**
- Application of salts prior to the attainment of maturity (4000 psi strength, a period of air drying)
- Application of salts on a poorly air entrained concrete
- Application of salts on an air-entrained concrete which has lost its surface air by finishing

* A properly placed, finished, cured, & air-entrained concrete should resist the effect of salts *

(4) **Softening of surface – which causes ‘abrasion’**
- Addition of water during finishing, which increases the w/cm
- Finishing with bleed water on the surface; Sprinkling of dry cement
- Inadequate curing

- Cyclic freezing of concrete at critically water-saturated condition
- Traffic load
Diagnosing Scaling – Strategy

Preliminary Investigation
- Field investigation, Photographs
- Selection of sample locations from scaled and sound areas
- Collection of saw-cut section or concrete core
- Review of concrete mix design
- Background information on concrete placement, finishing and curing
- Weather condition during placement; First exposure to winter

Laboratory Examination
- Petrographic Examination
- Air-void & Air Profile Analysis
- Chloride Analyses
Popout – Aggregate is the culprit!

- “The breaking away of small portions of a concrete surface due to localized internal pressure that leaves a shallow, typically conical depression”

ACI Committee 116 (Cement and Concrete Terminology)

Small = Holes up to 0.4 in. (10 mm) in diameter
Medium = Holes 0.4 to 2 in. (10 to 50 mm) in diameter
Large = Holes > 2 in. (50 mm) in diameter

- Fracture plane develops through the aggregate
- A portion of aggregate remains at the base of the conical depression
(1) Porous, water-absorptive aggregates having low modulus of elasticity
   (a) Freezing at critically water-saturation condition
   (b) Swelling by moisture absorption during cyclic wetting.

   Clay-rich sedimentary and metamorphic rocks e.g., shale, ferruginous shale, argillite; porous chert, soft fine-grained limestone and dolomite, water absorptive (hygroscopic) glass particles.

(2) Alkali-reactive aggregates (Alkali-aggregate reaction)

   Aggregates having certain silica minerals (e.g., strained quartz, chalcedony, reactive chert, tridymite, cristobalite), silica glass, siliceous glassy volcanic rocks, limestone with reactive silica inclusions.

   (a) Reactive components react with cement alkali and form alkali-silica gel.
   (b) Absorption of moisture by the gel causes expansions and subsequent popout.

(3) Unsound impurities in aggregates

   (a) Delayed hydration of hard-burned lime, hard-burned dolomite, and free magnesia (periclase) present as impurities
   (b) Oxidation of pyrite (ferrous sulfide) present as inclusions in some aggregates like dolomite and shale, and the presence of coal in aggregates

*Petrographic Examination is the best method to diagnose popouts*
Diagnosing Popout – Strategy

Preliminary Investigation
- Field investigation, Photographs
- Selection of sample locations from scaled and sound areas
- Collection of saw-cut section or concrete core
- Review of concrete aggregates

Laboratory Examination
- Petrographic Examination

Cross-sections of concrete cores
Mortar Flaking – A different type of scaling

- Loss of surface mortar from above the top side of near surface aggregate particles

1. Prolonged finishing of thin surface mortar over the top side of near-surface aggregate particles

2. Inadequate curing, rapid loss of water from the surface which cannot be re-saturated by the bleed water due to blockage by aggregate

Evaporation of surface water
↑ ↑ ↑

Finished surface

Bleed water blockage by a coarse aggregate particle

Crack formation

Weak bond between surface mortar and near surface aggregates due to repeated finishing

Lift-off

Original finished surface

Incipient Lift-off

Scaled surface

Future Scaled surface
Mortar Flaking – A different type of scaling

- Loss of surface mortar from above the top side of near surface aggregate particles
Mortar Flaking – A different type of scaling

- Loss of surface mortar from above the top side of near surface aggregate particles
Delamination – A common culprit of warehouse floor

- “A separation along a plane parallel to a surface, .............in case of a concrete slab, a horizontal splitting, cracking, or separation within a slab in a plane roughly parallel to, and generally near, the upper surface; found most frequency in bridge decks and caused by the corrosion of reinforcing steel or freezing and thawing; similar to spalling, scaling, or peeling except that delamination affects large areas and can often only be detected by nondestructive tests, such as tapping or chain dragging.”

ACI Committee 116 (Cement and Concrete Terminology)

Common Causes

1. Air entrainment in concrete which will receive a hard machine trowel finish
2. Premature finishing before the cessation of bleeding
3. Prolonged finishing
4. Factors that increase bleeding duration, rate and capacity
5. Surface crusting, top-down stiffening, premature finishing
6. Corrosion of reinforcing steel in concrete
7. Cyclic freezing
Delamination – A common culprit of warehouse floor

Strategy

1. Field survey – Chain drag, Tapping, Electro-mechanical sounding
2. Sample selection from hollow-sounded areas
3. Petrographic Examination
4. Air profile analysis

Cross-sections of concrete cores from delaminated slabs where delamination was detected by hollow resonance during tapping with a metal rod.
Delamination – Two common causes

Using an air-entrained concrete in a slab to be machine troweled

Premature Finishing

Delamination – Two common causes

Using an air-entrained concrete in a slab to be machine troweled

Premature Finishing
Delamination

Using an air-entrained concrete in a slab to be machine troweled

Core 2: \( A = 6.0\% \), \( \alpha = 545 \text{ in}^2/\text{in}^3 \), \( \bar{L} = 0.0087 \text{ in.} \)

A typical air-profile in a delaminated slab made using air-entrained concrete

Cross-section of a core

Zone of stable air

Zone of decreasing air

Delamination @ 0.2 in.

Air Content (%) at a particular depth
Using an air-entrained concrete in a slab to be machine troweled

- Above Delamination
- Below delamination
- Air-entrained concrete

Increasing depth from the surface:

- Dense near-surface mortar
- Incipient delamination
- Delamination

Near surface  
Near the plane of delamination  
In the body
Delamination – A common culprit of warehouse floor

- Evaporation of bleed water; Surface drying
- Provide initial curing
- Finishing ends before final set
- Provide intermediate curing
- Prolonged finishing (possibility of delamination)

Penetration Resistance

- Transport, Placing, Consolidation, Strike-off, Bull float
- Possibility of delamination if finishing starts here
- Floating, Brooming, Troweling

Initial Set

Time since batching

Final Set

Final Curing

Bleeding ends

“Window of finishability”
Delamination – By Corrosion of Reinforcing Steel in Concrete
Abrasion – Dusting – Discoloration

**Abrasion**
- High w/cm in concrete
- High w/cm at the surface
  - Finishing-induced
  - Excessive bleeding
- Inadequate curing

**Dusting**
- Carbonation
- High w/cm at the surface
  - Finishing-induced
  - Excessive bleeding
- Inadequate curing
- Laitance

**Discoloration / Staining**
- Differential w/cm at the surface
- High rate of evaporation of water from the surface
- Polyethylene sheet on surface for curing
- Calcium chloride admixture
- Iron oxide minerals in aggregates

Petrographic Examination
Corrosion of reinforcing steel in concrete

- Chloride-induced corrosion
- Carbonation-induced corrosion

The Corrosion Process

Up to 600 times increase in volume of rust compared to that of the original metal
Corrosion of reinforcing steel in concrete

- Chloride-induced corrosion (Can Occur @ high pH)
- Carbonation-induced corrosion (Occurs @ pH <11.5)

**Petrographic Examination**
- Quality and thickness of concrete cover
- Depth and extent of corrosion
- Depth of carbonation
- Condition of steel, Loss of cross section of reinforcing steel
- Microcracking in concrete due to expansion by formation of corrosion products

**Chloride Analysis**
- Chloride-induced corrosion
- Chloride profile from top to the level of corroded rebar and downwards

Corrosion – Cracking – Spalling – Corrosion Cycle

Concrete spalling by rebar corrosion
Corrosion of reinforcing steel in concrete

Chloride-induced corrosion

Chloride profile

Top surface

Cover

Cross-section of a core

Chloride content (% by wt. of cement)

Threshold chloride

CMC, Inc.
Corrosion of reinforcing steel in concrete

Carbonation-induced corrosion

Depth of carbonation depends on the quality (density, impermeability) of concrete.

- **Carbonation front**
  - Uniform depth of carbonation in an un-cracked concrete
  - Deeper depth of carbonation in a cracked concrete

*Carbonation reduces the pH, destroys the passive oxide film around reinforcing steel and promotes corrosion*
Frost attacks – Early (plastic) & Late (hardened) freezing of concrete

Strategy

- Petrographic Examinations (ASTM C 856)
- Air-void analysis (ASTM C 457)
- Freeze-thaw durability test (ASTM C 666)

Early Freezing – Ice crystal imprints

Late Freezing – Surface subparallel cracks
Cracking – A common problem

_Crack occurs when tensile stresses due to volume change exceed the tensile strength of concrete_

1. **Shrinkage cracks**
   - Plastic Shrinkage
   - Drying Shrinkage
   - Carbonation shrinkage

2. **Cracking due to expansion**
   - AAR,
   - Sulfate attack
   - Fire & Frost attacks,
   - Steel Corrosion
   - Hydration of Free Lime & Magnesia

3. **Structural or Mechanical cracks, Thermal Cracks, Overloading**
Shrinkage Cracking – A common problem in slab

Random cracking on a slab-on-grade

No control joints!  Deeper level of reinforcing steel!
Slab Curling

Differential moisture and/or thermal conditions causing differential volume changes between the top and bottom surfaces of a slab

- A slab curls up when top dries (shrinks) and/or cools (contracts) more than the bottom
- A slab curls down when top heats up (expands) more by the sun than the bottom

Factors that affect Shrinkage & Amount of Curling

- Aggregates – Size, Grading, Quality
- Cements – Shrinkage potential (Type I & III > II)
- Admixtures – Cl-containing, Chemical
- Compressive strength & Modulus of Elasticity
- Water-cement ratio, High water, High Cement, High Slump
- Concrete shrinkage potential (Curling ∝ Drying shrinkage)
- Temperature and moisture differentials; Curing
- Slab thickness; Joint Spacing; Reinforcing amount and depth
- Placement of slab on a wet subbase and slab exposed to low RH condition increases the shrinkage gradient and amount of curl

Diagnosing factors Responsible for Curling

- Field Investigation (Relative humidity profile)
- Petrographic Examination

Curling + Linear Shrinkage → Cracking
Common Problems

- Surface distresses – Scaling, Spalling, Delamination, Staining
- Corrosion of reinforcing steel in concrete (Chloride and Carbonation induced)
- Cyclic freezing and deicing salt scaling
- Alkali-aggregate reaction
- De-bonding

Common Tests

- Field Survey
- Petrographic Examination
- Chloride content and chloride profile analysis
- Compressive strength test
Low Strength!

How low is ‘low’?

- High air content
- High water-cementitious materials ratio (w/cm)
- Weak aggregate-paste bond (Bleeding, Void clustering, High w/cm)
- Improper concrete ingredients – Low cement, different cementitious material(s), aggregate, pozzolan
- Poor Curing, Early Freezing, Early exposure to sun
- Improper Testing, Improper sample preparation for the test, Improper handling
- Sample – large diameter, high l/d, diameter/aggregate size < 2, moisture condition (air dry test > moist test), load-vs.-cast direction, rebar, poor consolidation, core location
- Field concrete – Physico-chemical attacks (leaching, acid/sulfate attack, fire/frost attack, AAR, etc.)

Why ‘low’?

For field or lab-cured cylinders (ASTM C 39):
1. An average of any 3 consecutive lab-cured cylinder strength tests < \( f'_c \)
2. An individual test (i.e. average of 2 cylinders) falls below \( f'_c \) by more than 500 psi
3. Strength of field-cured cylinder at the age of \( f'_c \) is < 85% of that of companion lab-cured cylinders

For cores (ASTM C 42):
1. The average strength of 3 cores is < 85% of \( f'_c \)
2. The strength of a core is < 75% of \( f'_c \)

How to Diagnose?

- Compressive strength test
- Petrographic Examination
- Air void analysis

CMC, Inc.
Low Strength!

Excessive air!
Fire attack - Alterations

(1) Discoloration of paste

- Normal grey to Pink/Red discoloration @ 250-300°C (due to dehydration and oxidation of iron hydroxide phases in aggregate)
- Pink/Red to Purple grey discoloration @ 500-600 °C (due to reaction between ferric oxide and lime -> light colored Calcium ferrite)

(2) Loss of strength

- Dehydration of calcium silicate hydrate – the main cementitious component of concrete
- Over 80% loss of strength by 650 °C

(3) Thermal cracking & Weakening of Aggregate-Paste interface
due to stresses developed from:

- Differential linear coefficient of thermal expansion of aggregate minerals along different axes (thermal anisotropy)
- Expansion of aggregate and shrinkage of paste
Fire attack – Alterations (Cont’d)

(4) Mineralogical Changes

- Loss of free water from ambient to 150-200°C
- Loss of structural water of cement hydrated above 200 °C
- Dehydration of Ca(OH)$_2$ @ 400-450°C, patchy anisotropy of paste with pale yellow-beige birefringence at > 450°C due to dehydration of C-S-H and CH and formation of C-S and CaO
- Transformation of quartz from alpha-to-beta form @ 573°C
- Almost complete dehydration of cement hydrates @ 500-650°C
- De-carbonation of limestone to CaO and liberation of CO$_2$ @ 900°C

(5) Concrete spalling

- Explosive spalling at the early exposure to fire
- Slow spalling along surface parallel thermal cracks
- Steaming of internal moisture by the advancing heat-front

(6) Post-fire cracking

- Re-hydration of CaO formed from hydration of calcium hydroxide or de-carbonation of limestone during fire
Fire attack – Loss of strength & Discoloration of paste

Over 80% loss of strength by 600°C

Class 20 (w/c 0.65) → Class 30 (w/c 0.4)

Colour changes:
- normal
- pink
- whitish grey
- buff

Temperature (°C)

Percentage of nominal strength

Fire-damage factor
Fire attack in a concrete silo
Pink discoloration of paste towards the fire-exposed side

Carbonation, Thermal cracking
Dehydration of Ca(OH)$_2$
Normal paste with Ca(OH)$_2$

Lapped section

Thin-section photomicrographs

Microcracking
Dark paste below the carbonated zone
Normal paste below the dark paste

Side exposed to fire

CMC, Inc.
Alkali-Aggregate Reactions (AAR)

Alkali-Silica Reactions (ASR)

(1) Reactive silica in aggregate + Cement alkalis = Gel
(2) Gel absorbs moisture $\rightarrow$ Expand $\rightarrow$ Cracking

Alkali-Carbonate Reactions (ACR)

(1) Dolomite in Reactive carbonate rocks + Cement alkalis = Calcite + Brucite
(2) Expansion by - Moisture absorption of clay in the rock,
   - ASR,
   - In-situ formation of Calcite / Brucite

Expansion, Map cracking
Alkali-Silica Reactions (ASR)

Reactive aggregate has:

*Reactive silica component:* Silica glass, tridymite, cristobalite, flint, chert, opal, chalcedony, glasses in acid and intermediate volcanic rocks

Tests:
- Aggregates: ASTM C 289 (Chemical test), Petrography (ASTM C 295)
- Hardened Concrete: ASTM C 1260 (Mortar Bar), ASTM C 1293 (Length Change), Petrography (ASTM C 856)

Petrographic Diagnosis:
- Reactive silica components
- Reaction Rims
- Reaction products (ASR Gel)
- Cracking of aggregates and paste
Alkali-silica reactive components in thin-sections of concrete
Alkali-silica gel in concrete: No Gel – No ASR

Fractured section

Voids lined with gel

Thin-section

Freshly fractured section treated with uranyl acetate solution and exposed to UV-light

Oil-immersion mount
Alkali-Carbonate Reactions (ACR)

Reactive aggregate:

*Characteristic composition:* (1) Calcitic-dolomite or Dolomitic limestone - Calcite/Dolomite 1:1,
(2) Insoluble residue contain large amount of clay (15-20%)

*Characteristic texture:* Large, rhombic crystals of dolomite set in a finer-grained matrix of calcite, quartz, and clay

De-dolomitization reaction:

(1) Dolomite in carbonate aggregate + Alkali hydroxide = Calcite + Brucite + Alkali Carbonate
(2) Alkali Carbonate + Calcium hydroxide = Alkali hydroxide + Calcite

Expansion by:

(1) Absorption of moisture by clay in carbonate rock matrix, or
(2) Reaction of reactive silica in carbonate rocks with alkali to form ASR gel, or
(3) De-dolomitization reaction, in-situ formation of brucite and calcite

Petrographic evidence of ACR:

A decrease of dolomite rhombs towards the margins of reactive rocks, Cracking, Reaction Rims

Tests:

Aggregates: ASTM C 586 (Rock-prism test), Petrography (ASTM C 856)
Hardened Concrete: ASTM C 1105 (Length Change), Petrography (ASTM C 856)
Alkali-Carbonate Reactions (ACR)

Characteristic texture of a reactive carbonate rock
Alkali-Carbonate Reactions (ACR)


CALIBRATION CURVE

% DOLOMITE IN CARBONATE ROCK

X-RAY INTENSITY RATIO

CALCITE 3.03 Å

DOLOMITE 2.88 Å

CMC, Inc.
Sulfate Attack

**Chemical sulfate attack**

- Sodium sulfate
- Calcium sulfate
- Magnesium sulfate
- Ammonium sulfate

Cement Hydrates
- Calcium hydroxide
- Calcium aluminate hydrate
- Calcium monosulfoaluminate

External (Classical) Sulfate Attack

- In Paste = Delayed ettringite formation (DEF)
- In Aggregate = Gypsum, Pyrite

Internal Sulfate Attack

- Expansion, Cracking, Loss of strength
- Loss of mass

Physical sulfate attack

- Reversible phase transformations of water-soluble sulfate salts between various anhydrous/hydrous forms due to cyclic variations in T & RH (Thenardite ⇔ Mirabilite)
- Crystallization of sulfate salts in pore spaces (Salt weathering)

Expansion, Cracking, Loss of strength
Loss of mass

Scaling
Acid Attack

Being alkaline in nature, exposure to acidic solutions causes loss of mass and loss of strength of portland cement concrete.

Corrosion of column bases exposed to acidic solution from an acid-containment tank.
Problems in Masonry
Fill Materials – Potential Expansion

Materials:

- All kinds of demolished materials
- Slag
- Gypsum based products
- Recycled portland cement concrete

Common Tests:

- X-ray diffraction
- Petrographic Examination
- Chemical analyses

Potential Expansion:

- Absorption of moisture by clay (swelling clays)
- Sulfate – aluminate reactions (gypsum plus portland cement based products)
- Hydration of free lime, magnesia
- AAR, Cyclic freezing (frost heave), Corrosion of steel
Fast-setting grouts

Por-Rok, Super Por-Rok, Quik-Rok, Anchor All, Pave Patch

Advertised Benefits

- Rapid setting
- High early strength, Excellent late strength
- Shrinkage-compensating (Anchoring by early expansion of 0.1-0.2%)
- Excellent freeze-thaw resistance
- Workability, Pourability, Patchability
- Exterior application (water run offs)

Yet Common Problems!

- Cracking & Spalling of adjacent concrete
- Lifting of rail posts
- Internal cracking of patching grouts
- Softening of grouts
- Corrosion of rail posts

Materials? Or Workmanship?
Expansion of Fast-setting anchoring & patching grouts

Uplifting of rail posts and spalling of concrete. Quik-Rok was used.
What causes the problem?

1. Sulfate-aluminate reactions in mature (hardened) grout forming ettringite
2. Cyclic freezing at critically moisture-saturated condition
3. Corrosion of rail post at the low pH environment of gypsum based grout (pH = 6-8)
4. Softening of gypsum in the presence of moisture

<table>
<thead>
<tr>
<th>Brand</th>
<th>Sulfate</th>
<th>Aluminate</th>
<th>Product</th>
</tr>
</thead>
<tbody>
<tr>
<td>Quik-Rok</td>
<td>Gypsum</td>
<td>Portland cement</td>
<td>Ettringite</td>
</tr>
<tr>
<td>Por-Rok</td>
<td>Gypsum</td>
<td>High alumina cement</td>
<td>Ettringite</td>
</tr>
<tr>
<td>Super Por-Rok</td>
<td>Portland cement</td>
<td>Portland cement</td>
<td>Ettringite</td>
</tr>
<tr>
<td>Anchor All</td>
<td>Gypsum</td>
<td>Portland cement</td>
<td>Ettringite</td>
</tr>
</tbody>
</table>

Gypsum-based grouts should not be used in moist, outdoor environment
Zero-slump precast concrete
Tests of PCCP:
- Petrographic Examination
- Absorption, Density,
- Volume of Permeable voids
- Chloride profile
Oil immersion mounts

Lime

Gypsum

X-ray diffraction pattern of gypsum plaster

G = Gypsum from the plaster
Q = Quartz from the sand

Degrees, 2θ

X-ray Intensity
Still many more examples to list!

Call me with any questions!

Let’s go to the conclusions
Petrographic examination is an excellent method for materials characterization and failure investigation of construction materials.

Field Examination and adequate background information are the essential requirements for any failure investigation.

Various types of surface distresses, lower-than-designed compressive strength, and corrosion of steel in concrete are the most frequently-encountered concrete problems – which can be diagnosed by petrographic examination and physico-chemical tests.

Proper diagnosis of a problem is essential to provide recommendations for the appropriate repair. Petrographic examination is essential for proper diagnosis, based on which an engineer can prescribe a repair scheme.

Thank you!