Aggregate and Concrete Petrography

Insights into Aggregate, Concrete, and Issues that can affect their Performance

Presented by:
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Outline

• Definition
• ASTM Methods
• Aggregate Petrography
  – How To
  – Iron Sulfides Issue
• Concrete Petrography
  – Why and How To
  – Surface Defects related to finishing
  – Examples of ASR Damaged Concretes
• Update on ASTM Committees on Aggregate Reactions and ACI Durability & Aggregate Committees
What is Petrography?

- A branch of geology
- Merriam Webster Dictionary:
  “the description and systematic classification of rocks”
- Concrete is essentially a man-made rock
  - Applies the same techniques used for rock, to examine and describe aggregate for use in concrete, and the microstructural characteristics of hardened concrete.

- Megascopic & Microscopic
  - Sometimes scratch and sniff isn’t enough
Concrete Petrography – ASTM Methods

- **ASTM C 295** – Standard Guide for Petrographic Exam 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 C1723** – Standard Guide for Examination of Hardened Concrete Using Scanning Electron Microscopy
- **ASTM C1324** – Standard Test Method for Examination and Analysis of Hardened Masonry Mortar
Petrographic Exam of Aggregate for use in Concrete

ASTM C295

– Looking for characteristics that will affect the performance of the concrete.

– Describe and classify the material

– Determine relative abundance of constituents, especially those which may have a bearing on performance.
  • Freeze thaw susceptible, AAR, sulfates, swelling clays, flat elongated

– Compare aggregate from new sources with samples of aggregate with known performance records

– Identify contaminants
Petrographic Exam of Aggregate for use in Concrete
ASTM C295 – HOW TO

• Sieve size fractions
• Sort aggregate in each size fraction by identifiable lithology - rock type/color/grain size/texture using stereo-optical microscope
  – Identify potentially deleterious components and sort within each sorted lithology
  • Investigate further
    – SEM/EDS
    – Thin section – polarized light microscopy (PLM), SEM/EDS
    – X-ray Diffraction
    – X-ray Fluorescence, CSA A23.2-26A
Iron Sulfides

- Minerals most common: pyrite and pyrrhotite
  - No established limits for rejection of aggregate in N. America

Pyrite, FeS$_2$

Pyrrhotite, Fe$_{(1-x)}$S, $x \leq 0.125$
Oxidation → Secondary Minerals → Expansion

\[ 4\text{FeS}_2 + 15 \text{O}_2 + 10 \text{H}_2\text{O} \rightarrow 4 \text{FeOOH} \text{(goethite)} + 2 \text{H}_2\text{SO}_4 \]

\[ \text{Fe}_{(1-x)} \text{S} + \text{O}_2 + (3-x)\text{H}_2\text{O} \rightarrow (1-x) \text{FeOOH} \text{(goethite)} + \text{H}_2\text{SO}_4 \]

Pyrrhotite is much reactive than pyrite.

\[ \text{H}_2\text{SO}_4 + \text{Ca(OH)}_2 \rightarrow \text{CaSO}_4 \cdot 2\text{H}_2\text{O} \text{(gypsum)} \]

\[ \text{HŠ} + \text{CH} \rightarrow \text{CŠH}_2 \]

\[ \text{Ca}_3\text{Al}_2\text{O}_6 + 3 \text{CaSO}_4 \cdot 2\text{H}_2\text{O} \text{(gypsum)} + 26 \text{H}_2\text{O} \rightarrow \text{Ca}_6\text{Al}_2(\text{SO}_4)_3(\text{OH})_{12} \cdot 26\text{H}_2\text{O} \text{(ettringite)} \]

\[ \text{C}_3\text{A} + 3 \text{CŠH}_2 + 26 \text{H} \rightarrow \text{C}_6\text{AŠ}_3\text{H}_{32} \]

\[ \text{H}_2\text{SO}_4 + \text{C-S-H} + \text{Ca(OH)}_2 + \text{CO}_2 + 12 \text{H}_2\text{O} \rightarrow \text{Ca}_3\text{Si(OH)}_6(\text{CO}_3)(\text{SO}_4) \cdot 12\text{H}_2\text{O} \text{(thaumasite)} \]

\[ \text{HŠ} + \text{C-S-H} + \text{CH} + \text{Ĉ} + \text{H} \rightarrow \text{C}_3\text{SŠĈH}_{15} \]
Oslo Region had been plagued with sulfate attack from the pyrrhotite-containing “alum shales”.

SULFATE ATTACK ON CONCRETE IN THE OSLO REGION

JOHAN MOUM and I. TH. ROSENOVIST

In the Oslo region of Norway, alum shales containing small amounts of the unstable iron sulfide, pyrrhotite, produce an unusual form of sulfate attack upon concrete placed in or near these deposits, and cause deterioration if they are used as concrete aggregate. The ground water associated with the alum shales carries ferrous sulfate and produces severe sulfate attack and the precipitation of ferric iron compounds in concrete structures made with normal portland cement. Cements of low tricalcium aluminate content resist the sulfate attack but may be subject to attack by acid solutions produced when the ferrous sulfate is oxidized. Air-entrained concrete appears to be particularly susceptible.

For 40 years the construction industry in the Oslo region has been plagued by problems of concrete deterioration and foundation failures related to the presence of slightly metamorphosed shales containing usually unstable forms of the iron sulfide mineral pyrrhotite. They are called “alum shales” or “alum slates,” and the expression “alum problem” is familiar to most people engaged in construction work.

After World War II a semi-official “Alum Shale Committee” was formed by the city of Oslo, and the Norwegian Geotechnical Institute was requested to take over the problem essentially related to the chemical, physical, and mineralogical.

WEATHERING PRODUCTS

The weathered alum shales are mostly covered by a yellow deposit of jarosite [KFe$_3$(OH)$_6$(SO$_4$)$_2$] and brown-iron ore (Fe$_3$O$_4$ · nH$_2$O). The weathering of the alum shale also yields solutions which very rapidly attack concrete made with normal portland cement. We have seen the concrete walls of an underground bomb shelter built in an alum shale area transformed into mush in about 9 months. In other cases, the attack may proceed more slowly, but generally the attack from the alum shale extracts seem to be much quicker than attack by most other aggressive waters.
Thousands of home foundations deteriorated in Trois Rivières region of Quebec. Problems reported within 3-5 years of construction.
Connecticut Pyrrhotite

Affected structures constructed as early as the 1980’s.

Over 600 complaints filed, and up to 34,130 homes are potentially at risk.
Concrete Damaged Due to Pyrrhotite Oxidation
Concrete Damaged Due to Pyrrhotite Oxidation

Cement paste replaced by thaumasite.

Gypsum formed in void spaces.
Result - Extremely Weak Concrete
Stucco Affected by Pyrite Oxidation
Stucco Affected by Pyrite Oxidation

Stereo-optical microscope

SEM
Iron sulfide typically appears as minor inclusions within aggregate.
Iron Sulfide deposits often contain multiple mineral phases. 

- pyrrhotite
- pyrite
- chalcopyrite
Multiple factors determine risk of internal sulfate attack. (not just amount of sulfides)

Concrete Porosity/Permeability

Crystal structure

Exposure

Pittsburgh Area Chapter American Concrete Institute (ACI)
Canadian Researchers early development of performance-based testing

1 – Chemical Approach
Total Sulfur by Mass %

2 – Oxygen Consumption

3 - Mortar Bar Expansion


ASTM C856 – Hardened Concrete

Condition Assessment
Failure Analysis
   Cause of cracking, surface defects
   floor covering or coating failures
Low strength
Verify materials used and general conformance to mix design

• Identify:
  – Cementitious materials
  – Aggregate type, size, distribution
  – Air type, size, distribution
  – Presence of curing compounds, sealers, other
  – Depth of carbonation

• Estimate:
  – paste content
  – air content
  – w/cm-ratio

• Describe/Evaluate:
  – Cracking – plastic, drying shrinkage, load
  – Hydration
  – Porosity & distribution
  – aggregate alteration and reactions – AAR, f/t, chemical
  – Paste/aggregate bond
  – Deleterious reactions: Corrosion, Chemical attack, Freeze thaw, etc.
  – Bond of coatings, floor coverings or overlays
Petrographer and Engineer Team

The concrete microstructural characteristics are fundamental to the performance of the concrete element.

Understand and Recognize how they affect the performance of the concrete.

Experience
Merge information to Evaluate Observations

**Engineering**
- Placement
- History?
- Needs of owner
- Performance expectations
- Physical Properties

**Petrography**
- ID deterioration and potential causes
- Compositional and textural properties
- Materials used and construction procedures employed
- Current condition of concrete
- Potential for continuity
Link the Macroscopic to Microscopic

How do we get from here to there?
Process of Evaluation

1. Sample Receipt & Log-in
2. Photo-documentation

- Visual Inspection of the Specimen
- Cross Section Specimen and Polish
- Application of pH Indicator
- Stereo-Optical Microscopy of Polished Cross-Section
- Thin Section Preparation
- Polarized Light Microscopy
- Scanning Electron Microscopy with Energy Dispersive X-ray Spectroscopy
- Interpretation of Data and Reporting
Process - Sample Photo-documentation

Photo Documentation: Each sample is documented in its ‘as received’ condition
Process – As-received Examination

Sample is visually inspected and observations are recorded.

Sample is inspected with the aid of a stereo optical microscope.
Core sample is cross sectioned with a diamond saw and polished for stereo-optical microscopy examination.
Process – Cross section Exam

Carbonation Depth using phenolphthalein pH indicator

Lightly polished cross section
Process – Cross section Exam
Vacuum impregnated with a fluorescent dye

Samples are adhered to a glass slide and prepared as a thin section

Cured

And reduced
Sample Preparation

The impregnated sections are then polished with successively finer grit to produce a smooth surface.
Four optical views of the same area can produce varying assessments: extensive extent of parallel cracking and high water to cement ratio in this case.
Scanning Electron Microscope with EDS

Supplements Optical Microscopy with Compositional Information
Data Review and Compilation

• Review of visual, stereo-optical, polarized light microscopy
• Existing knowledge of defects and microstructure
• Information given by client

• Conclusions
  – Description of concrete and general observations
  – Cause of Defects with supporting observations
  – Degree of damage
Surface Distress

• Freezing and Thawing cycles
  – Microscopy to evaluate hardened air content
    • ASTM C856 for estimated content
    • ASTM C457 for measured content
• Microscopy give clues to work practices
  – Finishing & Curing
Affects of Improper Finishing on Surface Durability

Surface Defects/Premature Wear
Blisters or Delamination, Scaling, mortar flaking or popoffs, Crazing Cracks

• Premature Finishing
  – Finish bleed water into the surface
  – Entrapment of bleed water and air beneath the surface

• Late Finishing
  – Re-tempering
  – Uneven hydration, porosity, and surface texture
Affects of Improper Finishing on Surface Durability

• Overworking
  – Reduces entrained air along the surface
  – Excessive mortar buildup

• Inadequate Curing
  – Plastic and drying shrinkage cracks
  – Surface crusting and loss of plasticity
  – Mortar flaking over large aggregate
Common Causes of Poor Surface Durability Related to Finishing
Premature Finishing Entrapped Bleed Water
Common Causes of Poor Surface Durability Related to Finishing
Common Causes of Poor Surface Durability Related to Finishing
Common Causes of Poor Surface Durability Related to Finishing
Common Causes of Poor Surface Durability Related to Finishing
Increased W/Cm-ratio at surface
Common Causes of Poor Surface Durability Related to Finishing

Surface

Depth of 1”
Common Causes of Poor Surface Durability Related to Finishing
Surface Distress: Scaling

Finishing Issue: Re-tempering & addition of water
Surface Distress: Scaling

Due to Freeze thaw damage caused by Over working during finishing causing a decrease in air content in top 1/8” of surface.
Surface Durability Study

- 3 Concrete Pads 10’ x 24’, 5-6” depth
- 3 mixes

<table>
<thead>
<tr>
<th>Test Pad</th>
<th>W/C Ratio</th>
<th>Slump</th>
<th>Entrained air</th>
<th>Temperature</th>
<th>28-day Compressive Strength (psi)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.45</td>
<td>5 ¾”</td>
<td>6.8%</td>
<td>62 °F</td>
<td>4310</td>
</tr>
<tr>
<td>2</td>
<td>0.42</td>
<td>3 ½”</td>
<td>5.6%</td>
<td>64 °F</td>
<td>4930</td>
</tr>
<tr>
<td>3</td>
<td>0.39</td>
<td>1 ½”</td>
<td>4.3%</td>
<td>67 °F</td>
<td>5510</td>
</tr>
</tbody>
</table>

Early Finish  | On-time Finish with Cure | On-Time Finish without Cure | Late Finish
A              | B                        | C                          | D
Surface Durability Study
Surface Durability Study
What Did We Do

• The slabs were placed in the fall and we extracted cores the following spring.

• Petrographic examinations were performed following ASTM C856 and C1723
Affects of Early Finishing of Surface Durability Study

0.45 W/C ratio

0.42 W/C ratio

0.39 W/C ratio
Affects of Late Finishing of Surface Durability Study

Field of View 2.6 mm

Field of View 10.0 mm
Microstructure of Properly Timed Finish with and without a Curing Agent

- 0.45 W/C ratio
- 0.42 W/C ratio
- 0.39 W/C ratio
Conclusions Discussion

• Microstructural differences were slight.
  – We were not able to re-create the gross differences we wanted to express with the study.
  – They were so slight that we did not anticipate any significant difference in abrasion test results and so have not performed them as of yet.

• Factors:
  – Mix designs not too different.
  – Experienced and Certified ACI Concrete Flatwork Finishers on the job!!

• It is likely that most of the time when durability becomes an issue, it is in extreme cases of poor finishing OR extreme use.
Petrography of Concrete Affected by Alkali Silica Reaction

- Diagnose – confirm ASR as cause for damage
  - Additional mechanisms present?
  - Extent of damage - rating

- Condition, Damage rating index (DRI)

- Prognosis – comment on potential for further deterioration due to ASR

- Monitor Damage over time - DRI

- Evaluate mortar bars or prisms post testing to confirm ASR as cause for expansion
ASR Diagnosis
ASR – Diagnosis & Prognosis Info

Aged/Old Gel in crack in paste

Fresh gel within crack in aggregate
Alkali Silica Reaction Case Study

- Airport runway tarmac placed in 2010
  - ASR damage presented within 2 years
- Slab depth of 18 inches
- Highly Reactive fine aggregate – Andesite
- Cracking due to ASR depth of slab
Alkali Silica Reaction Case Study
Alkali Silica Reaction – Case study

Andesite Fine Aggregate in thin section
ASR – Repair Evaluation
ASR – Repair Evaluation
ASR Practice Document(s)

• ASTM C1778 Standard Guide for Reducing the Risk of Deleterious Alkali-Aggregate Reaction in Concrete

• AASHTO R-80 Standard Practice for Determining the Reactivity of Concrete Aggregates and Selecting Appropriate Measures for Preventing Deleterious Expansion in New Concrete Construction
ASTM C1778 Approved
New Fig. 1
Flow Chart Showing
General sequence of
laboratory test for
evaluating aggregate for
potential for AAR
What’s New

• ASTM
  – Committee 9.50 Aggregate Reactions in Concrete
    • Merged AAR Practice sub-committee with AAR test methods sub-committees

• ACI
  – Durability 201
    • Sub-committee on Aggregate Reactions to cover ASR, Iron Sulfides, RCA, ?
      – Soon to publish a technote on iron sulfides
      – Adding a section on iron sulfides to 201.2R-16 Guide to Durable Concrete document
  – Aggregates 221
    • Updating 221R-96: Guide for Use of Normal Weight and Heavyweight Aggregates in Concrete (Reapproved 2001)
    • Updating 221.1R.98 Report on Alkali-Aggregate Reactivity (Reapproved 2008)
      – Potential to do this as joint document with 201
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