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

$$4FeS_2 + 15\ O_2 + 10\ H_2O \rightarrow 4\ FeOOH\ (goethite) + 2\ H_2SO_4$$

$$Fe_{(1\text{-}x)}S + O_2 + (3\text{-}x)H_2O \rightarrow (1\text{-}x)\ FeOOH\ (goethite) + H_2SO_4$$

Pyrrhotite is much reactive than pyrite.

$$H_2SO_4 + Ca(OH)_2 \rightarrow CaSO_4{\cdot}2H_2O\ (gypsum)$$

$$H\hat{S} + CH \rightarrow C\hat{S}H_2$$

$$Ca_3Al_2O_6 + 3\ CaSO_4{\cdot}2H_2O\ (gypsum) + 26\ H_2O \rightarrow Ca_6Al_2(SO_4)_3(OH)_{12}{\cdot}26H_2O\ (ettringite)$$

$$C_3A + 3\ C\hat{S}H_2 + 26\ H \rightarrow C_6A\hat{S}_3H_{32}$$

$$H_2SO_4 + C\text{-}S\text{-}H + Ca(OH)_2 + CO_2 + 12\ H_2O \rightarrow Ca_3Si(OH)_6(CO_3)(SO_4){\cdot}12H_2O\ (thaumasite)$$

$$H\hat{S} + C\text{-}S\text{-}H + CH + \hat{C} + H \rightarrow C_3S\hat{S}\hat{C}H_{15}$$

Oslo Region had been plagued with sulfate attack from the pyrrhotite-containing "alum shales".

SULFATE ATTACK ON CONCRETE IN THE OSLO REGION

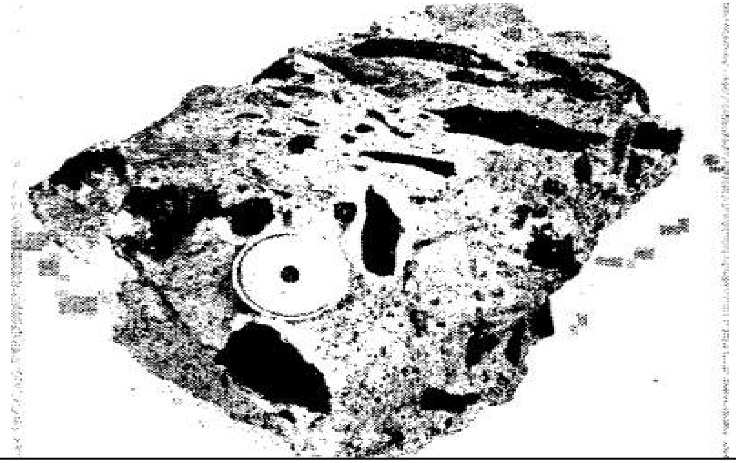
JOHAN MOUM and I. TH. ROSENQVIST

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 regi plagued with problems of concrete deterioration and foundatio related to the presence of slightly metamorphosed shales contai usually unstable form of the iron sulfide mineral pyrrhotite. Th called "alum shales"* or "alum slates," and the expression "the problem" is familiar to most people engaged in construction work

After World War II a semiofficial "Alum Shale Committee" w Oslo, and the Norwegian Geotechnical Institute was requested to take over

Both internal and external sulfate attack were documented.



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 ($Fe_2O_3 \cdot nH_2O$).

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.

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Quebec pledges $17M more for pyrrhotite-damaged homes

Government also lowers amount of problematic mineral in foundation concrete required to access fun

By Stephen Smith, CBC News Posted: Jan 06, 2017 2:18 PM ET | Last Updated: Jan 06, 2017 7:16 PM ET

This house is one of hundreds that have undergone repairs to fix damage caused by pyrrhotite.

34 shares

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Quebec is allocating $17 million more for homeowners in the Mauricie and Central Quebec regions affected by pyrrhotite.

The Liberal government announced the new funds Friday at a news conference in Trois-Rivières, 140 kilometres northeast of Montreal.

Pyrrhotite is a mineral that expands when exposed to humidity and oxygen. Its presence in aggregate can lead to cracks in concrete structures.

That problem has wreaked havoc on the foundations of thousands of homes and commercial buildings in the Mauricie and Central Quebec regions built between 1996 and 2008 with aggregate from a local quarry.

- Homeowners demand $25M from Ottawa for structural damage

Lower threshold

In addition to the new funds, the government also announced Friday that it was lowering the volume of pyrrhotite in concrete that's required to access the funds from 0.3 to 0.23 per cent.

The lower threshold and extra funds will help an estimated 400 homeowners and brings Quebec's total assistance for homeowners coping with pyrrhotite damage to $52 million.

Homeowner Marc Dubord welcomed the decision to lower the pyrrhotite threshold. The concrete in his foundation has a volume of .25 per cent

Related Stories

- What's in the federal budget for Quebec, and what's missing
- Homeowners demand $25M from Ottawa for structural damage

Ottawa to spend $30M helping Quebec homeowners with pyrrhotite problems

Prime Minister Justin Trudeau looks at the foundation of a house, in Trois-Rivieres, Que., on Wednesday, April 6, 201 CANADIAN PRESS/Paul Chiasson)

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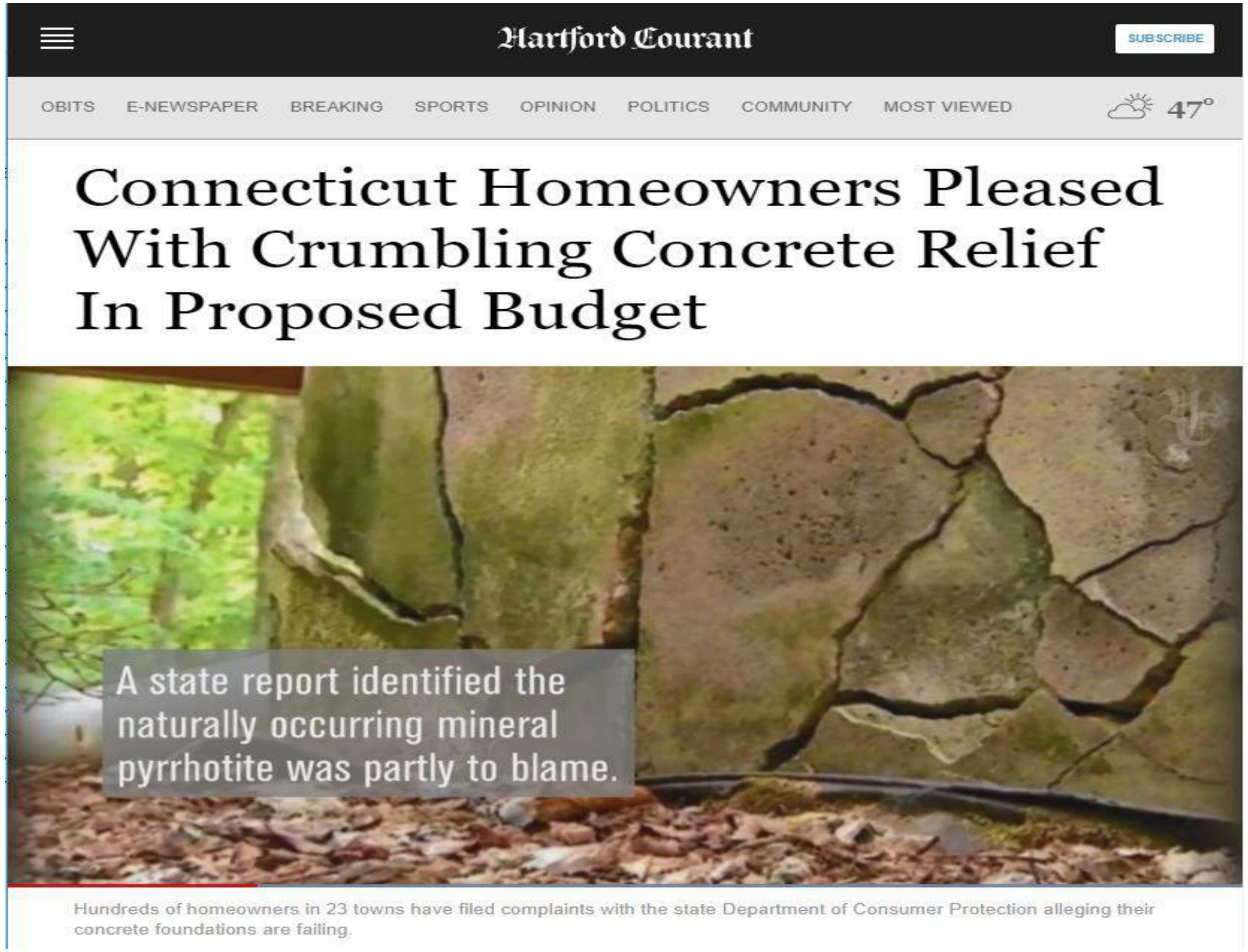
The Canadian Press
Published Wednesday, April 6, 2016 4:10PM EDT
Last Updated Wednesday, April 6, 2016 5:46PM EDT

Connecticut Pyrrhotite


Hartford Courant
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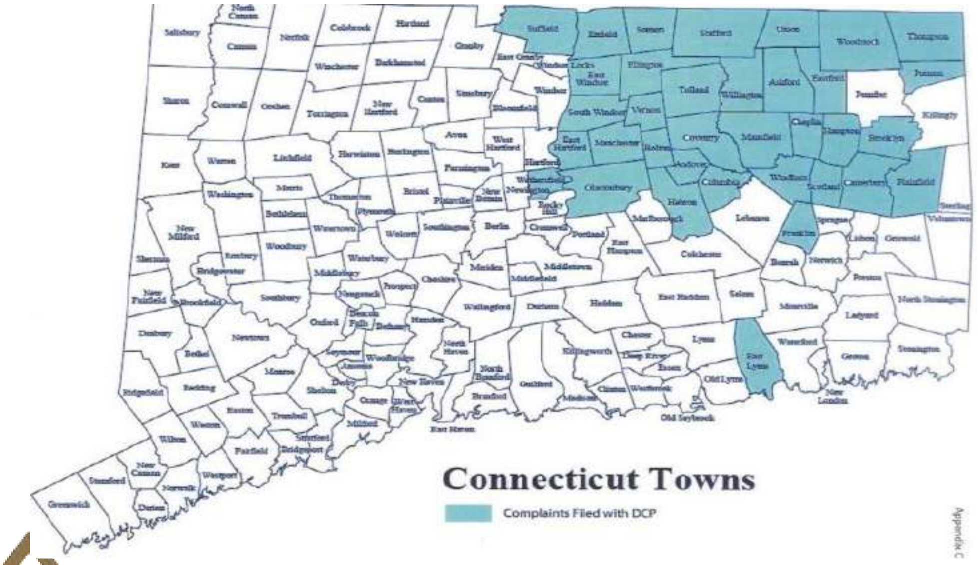
Connecticut Homeowners Pleased With Crumbling Concrete Relief In Proposed Budget

A state report identified the naturally occurring mineral pyrrhotite was partly to blame.

Hundreds of homeowners in 23 towns have filed complaints with the state Department of Consumer Protection alleging their concrete foundations are failing.

Affected structures constructed as early as the 1980's.

Over 600 complaints filed, and up to 34,130 homes are potentially at risk.



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Concrete Damaged Due to Pyrrhotite Oxidation

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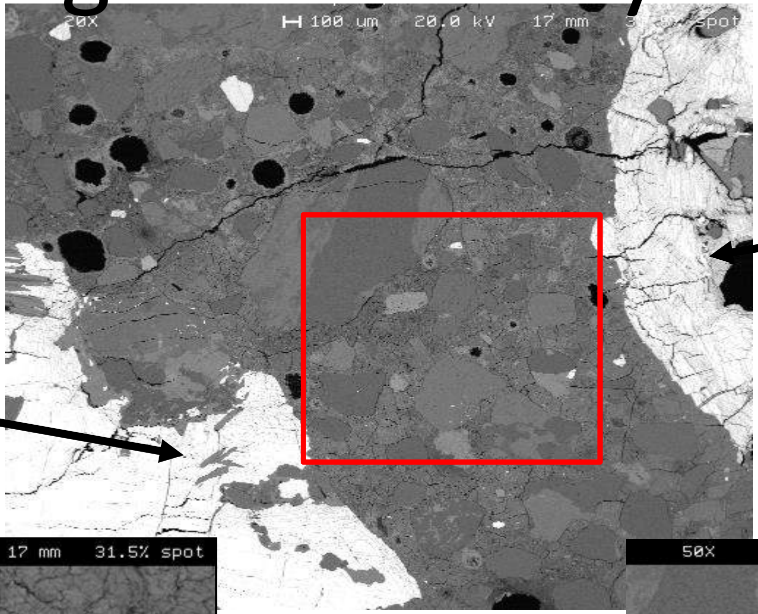
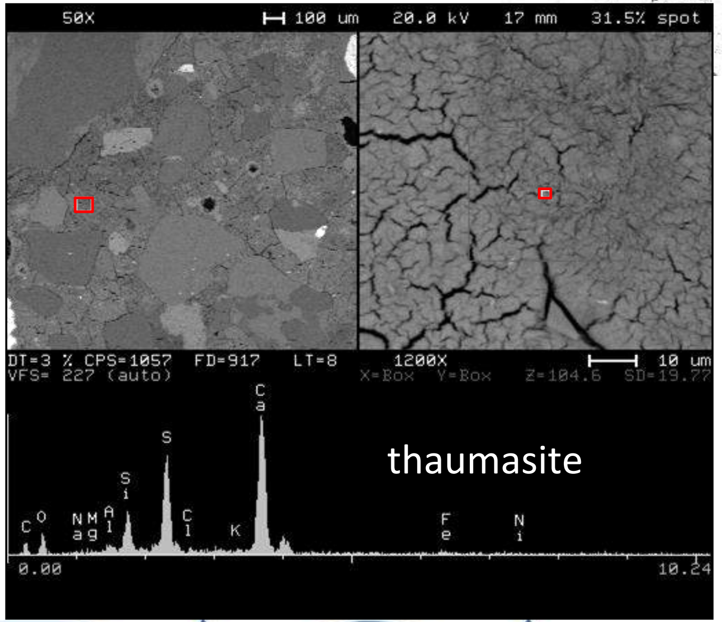
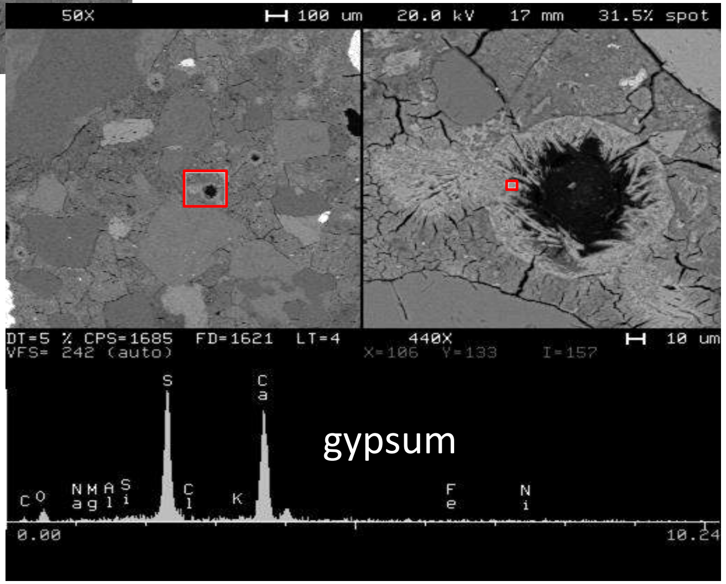


pyrrhotite

pyrrhotite



Cement paste replaced by thaumasite.



Gypsum formed in void spaces.

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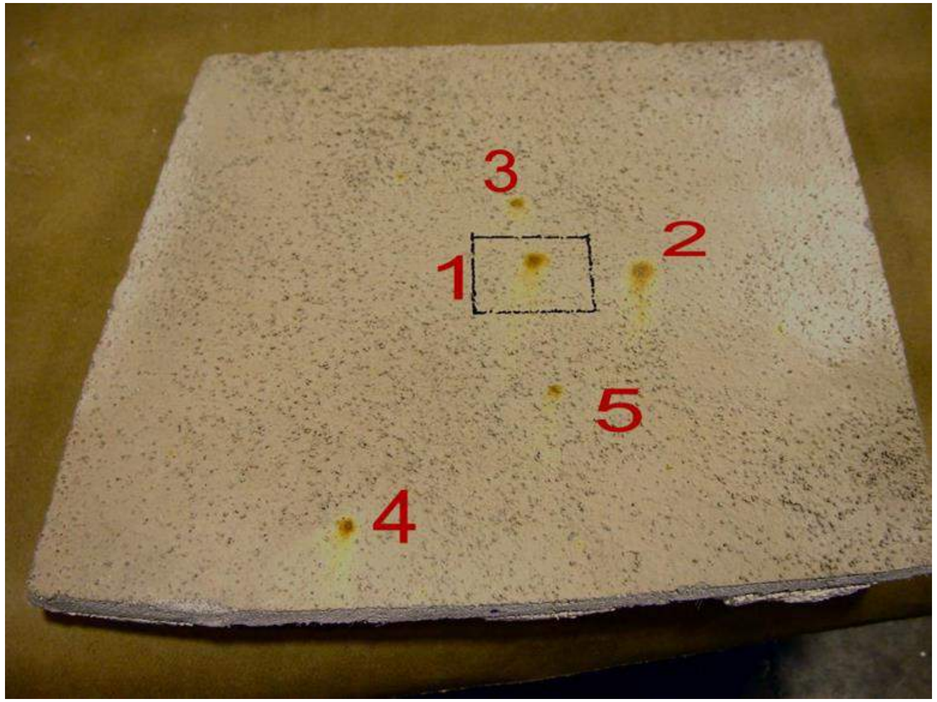
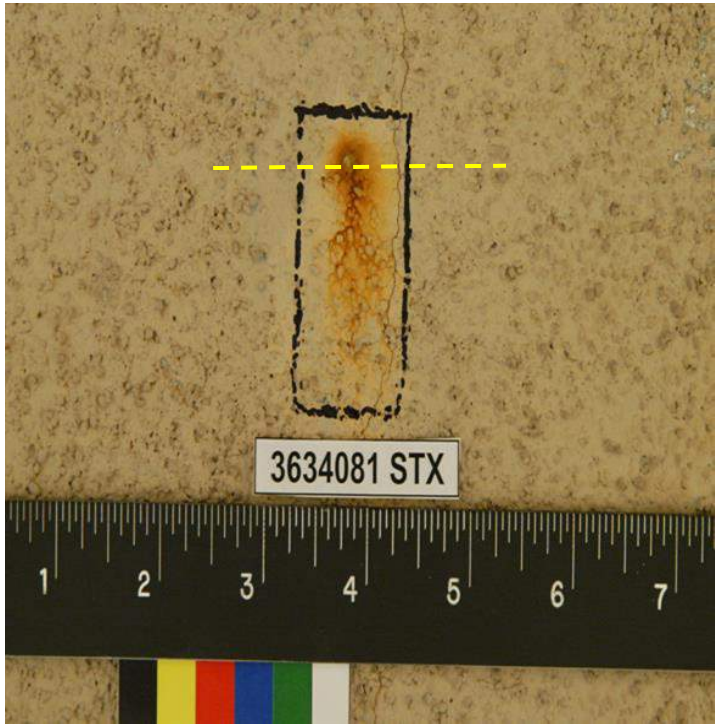
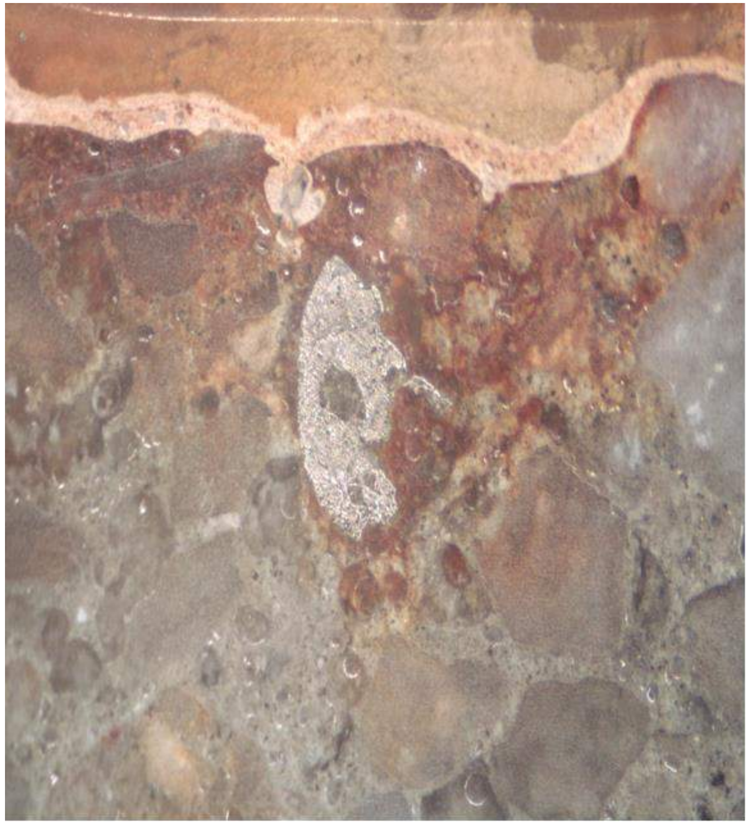
Result - Extremely Weak Concrete

Stucco Affected by Pyrite Oxidation

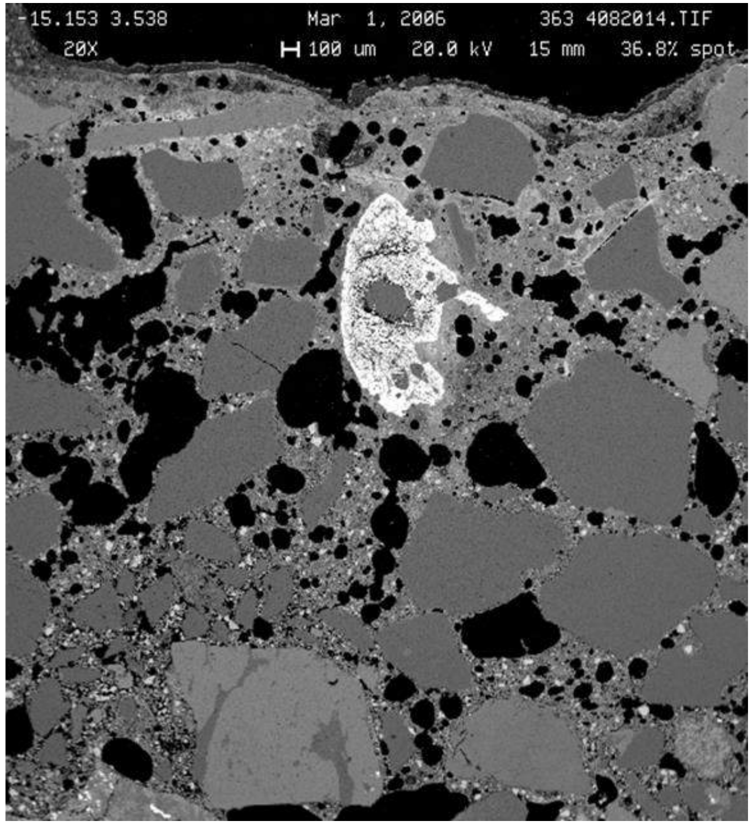


Stucco Affected by Pyrite Oxidation



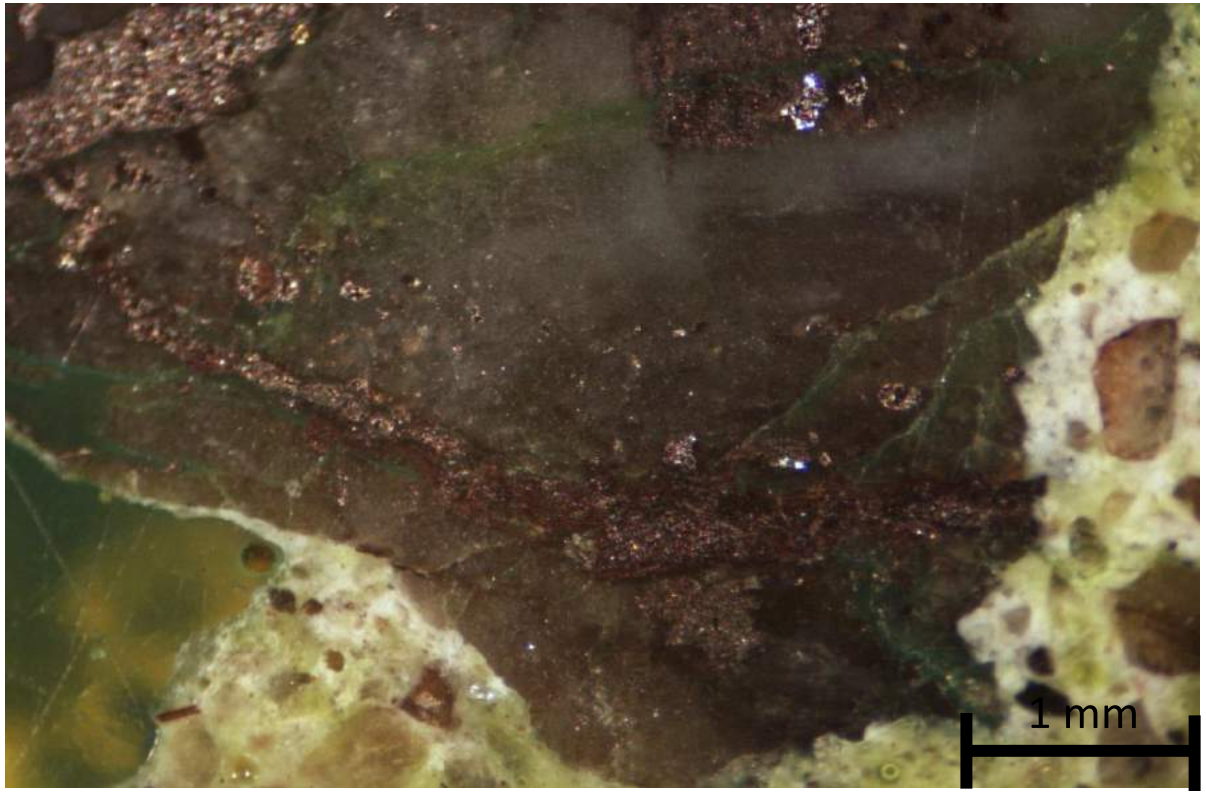


Stereo-optical microscope



SEM

Iron sulfide typically appears as minor inclusions within aggregate.

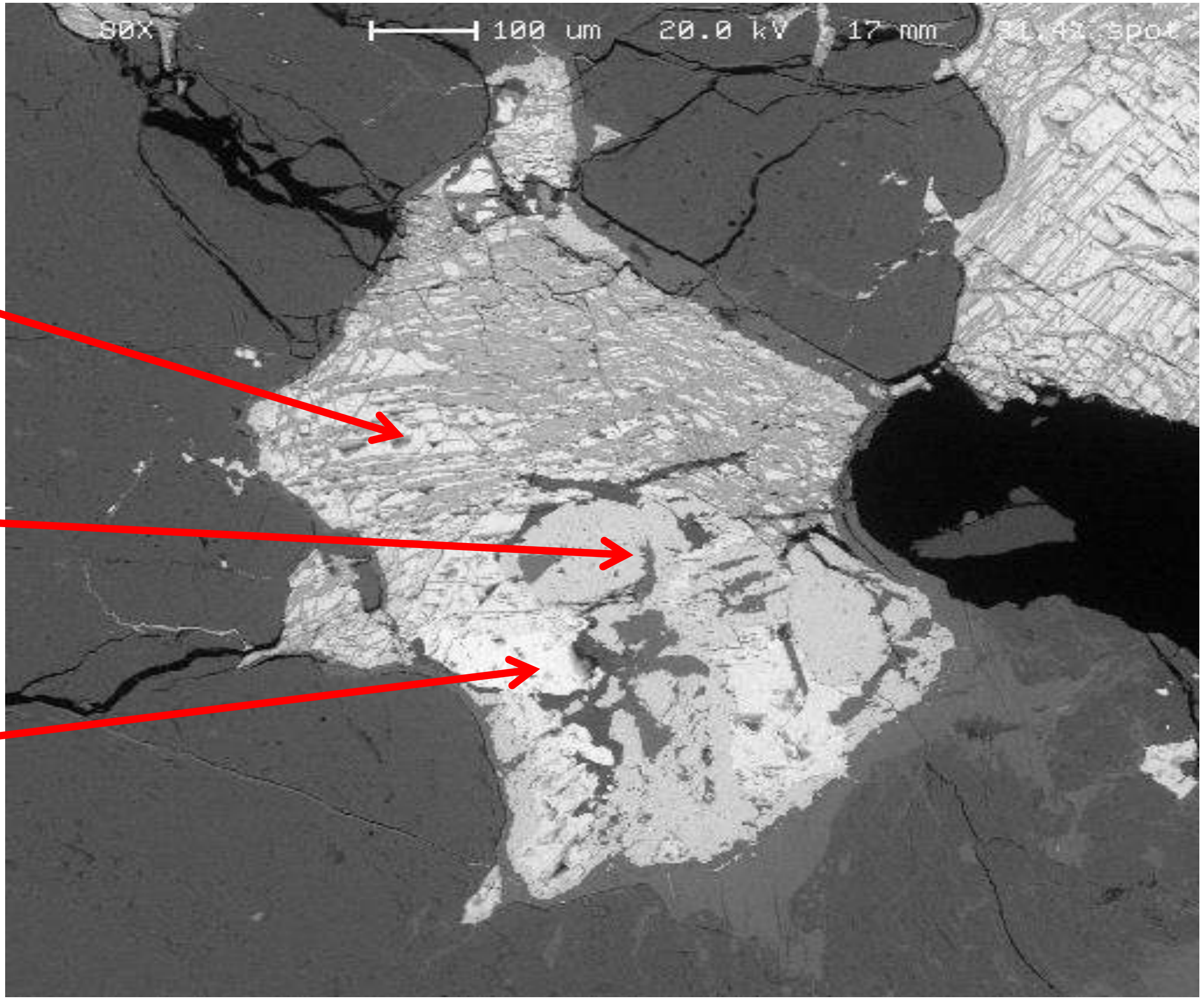


Iron Sulfide deposits often contain multiple mineral phases.

pyrrhotite

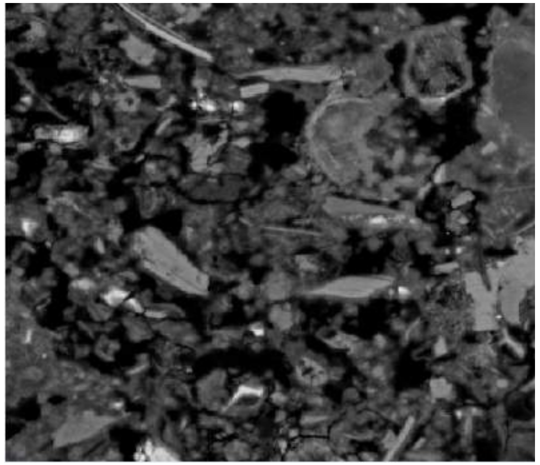
pyrite

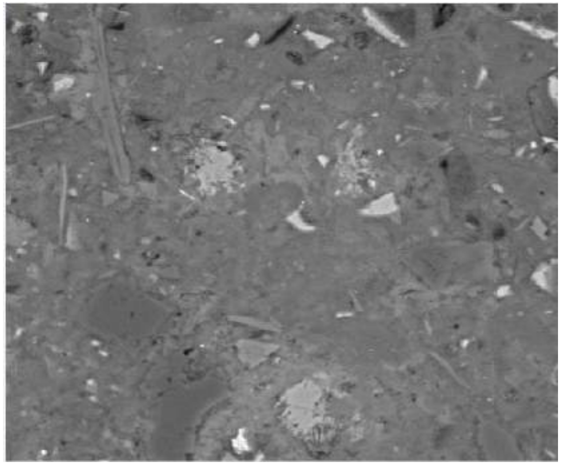
chalcopyrite



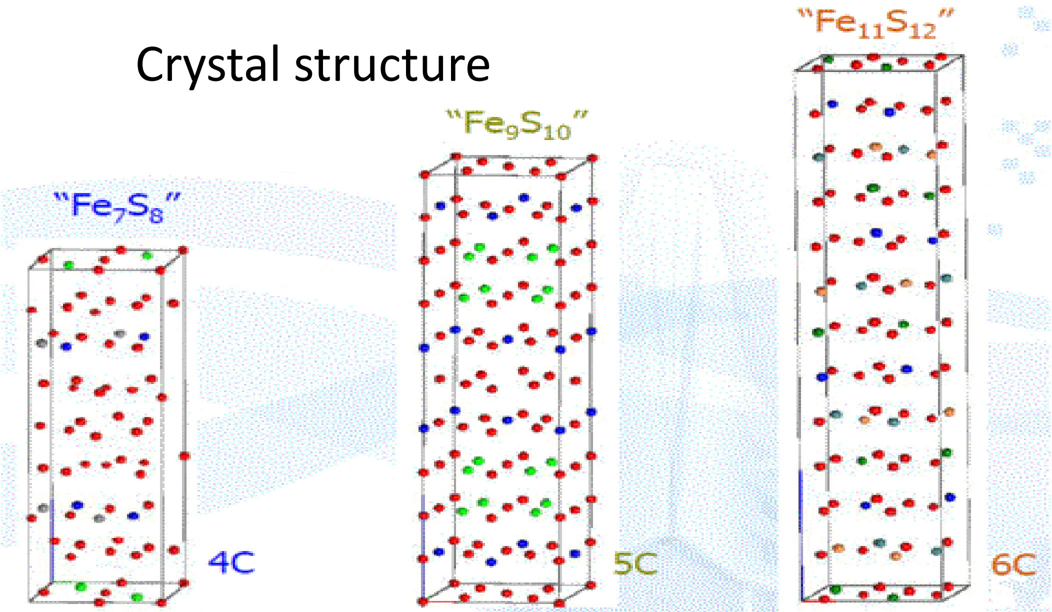
Multiple factors determine risk of internal sulfate attack. (not just amount of sulfides)

Concrete Porosity/ Permeability



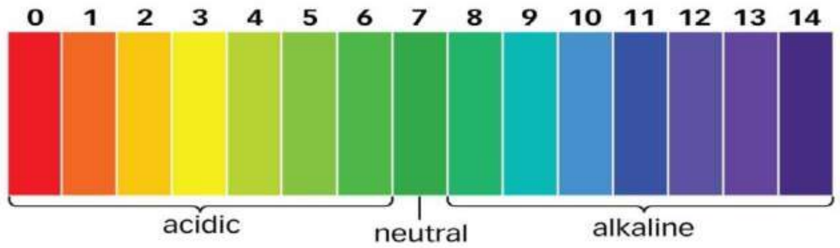


Crystal structure



Exposure

pH



Canadian Researchers early development of performance-based testing

1 – Chemical Approach
Total Sulfur by Mass %

2 - Oxygen Consumption

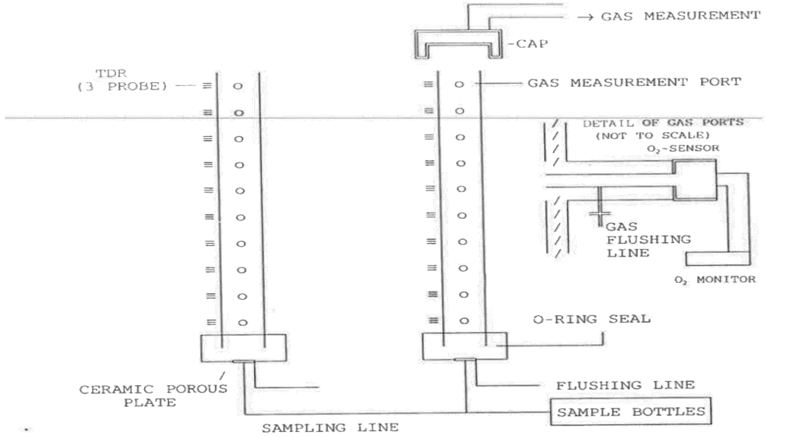
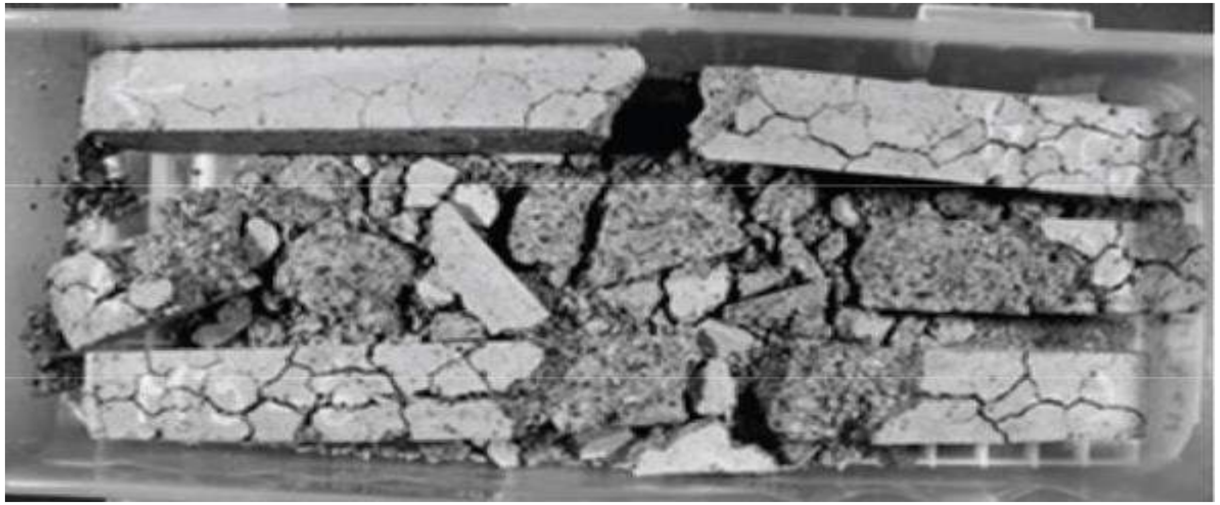


FIG. 3. Schematic configuration of the two columns used in the oxidation-rate experiments.

From Elberling, B., Nicholson, R. V., Reardon, E. J., & Tibble, R. (1994). Canadian Geotechnical Journal, 31(3), 375-383.

3 - Mortar Bar Expansion



From Rodrigues, A., Duchesne, J., Fournier, B., Durand, B., Shehata, M. H., & Rivard, P. (2016) *ACI Materials Journal*, *113*(3).

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

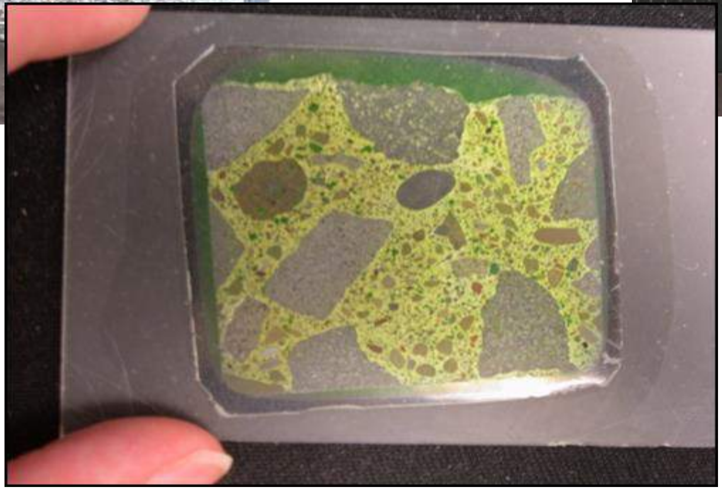
- Placement
- History?
- Needs of owner
- Performance expectations
- Physical Properties

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?



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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

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Process - Sample Photo-documentation

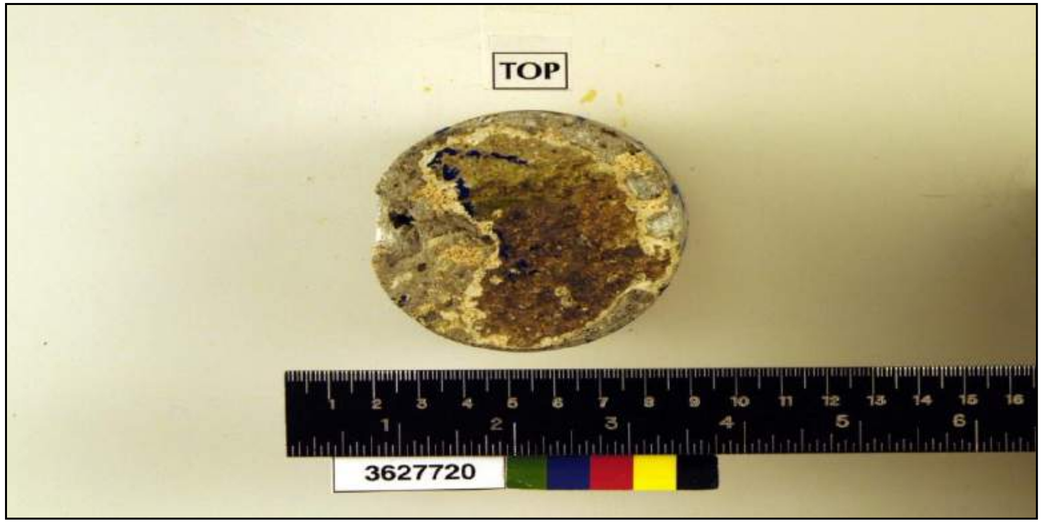
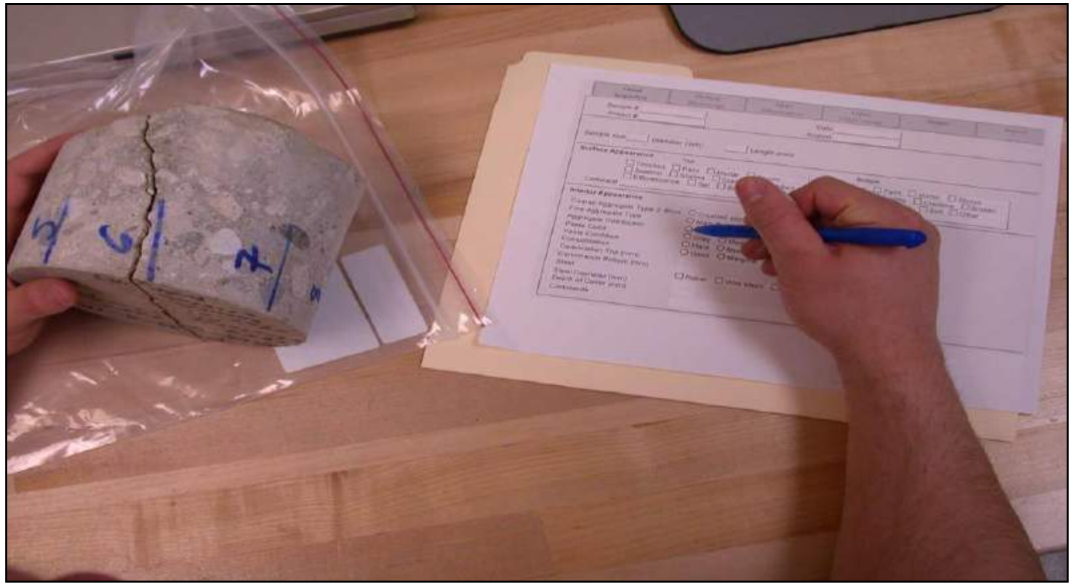
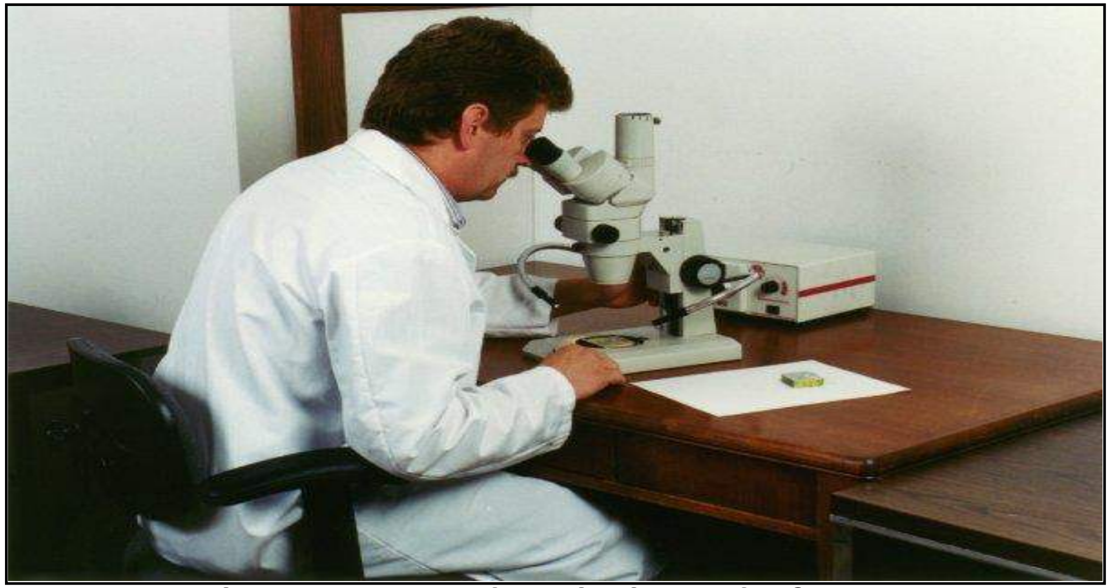


Photo Documentation: Each sample is documented in its ‘as received’ condition

Process – As-received Examination



Sample is visually inspected and observations are recorded



Sample is inspected with the aid of a stereo optical microscope

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Process - Sample Preparation

Core sample is cross sectioned with a diamond saw and polished for stereo-optical microscopy examination

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Process – Cross section Exam

Carbonation Depth using phenolphthalein pH indicator

Lightly polished cross section

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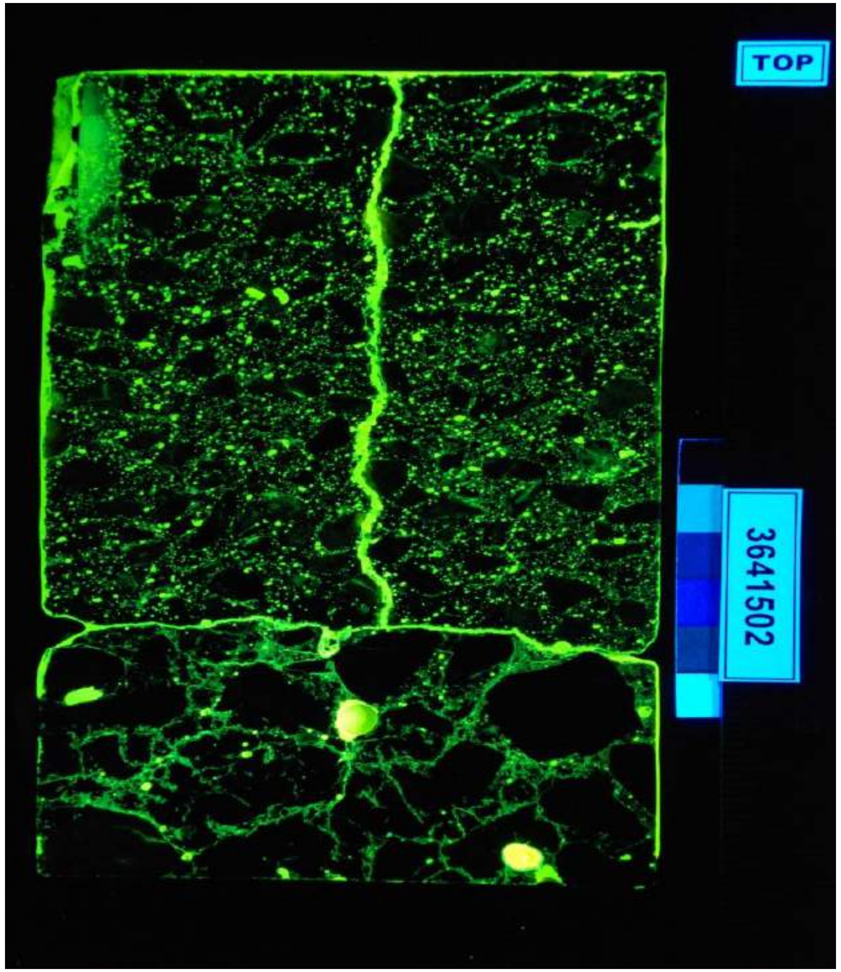
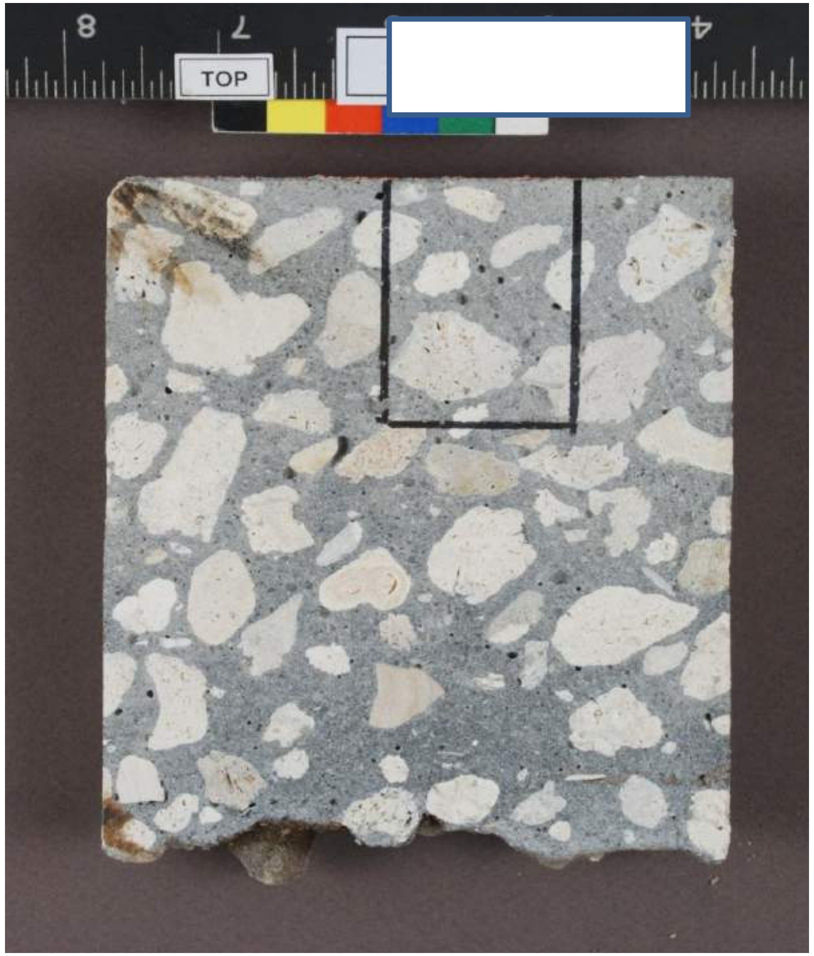
Process – Cross section Exam

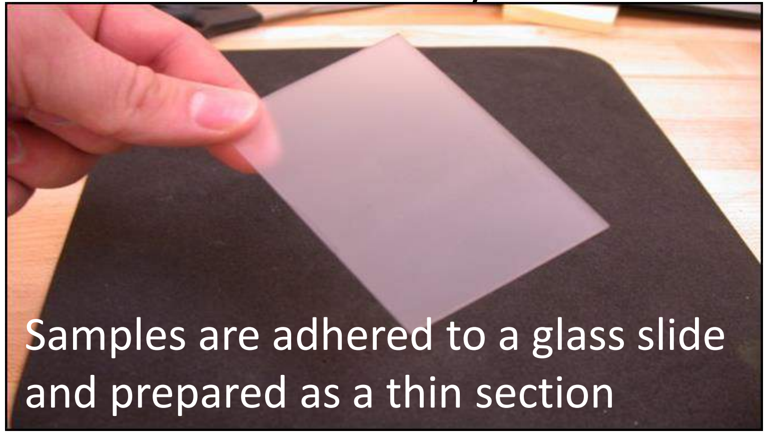




Process - Sample Preparation

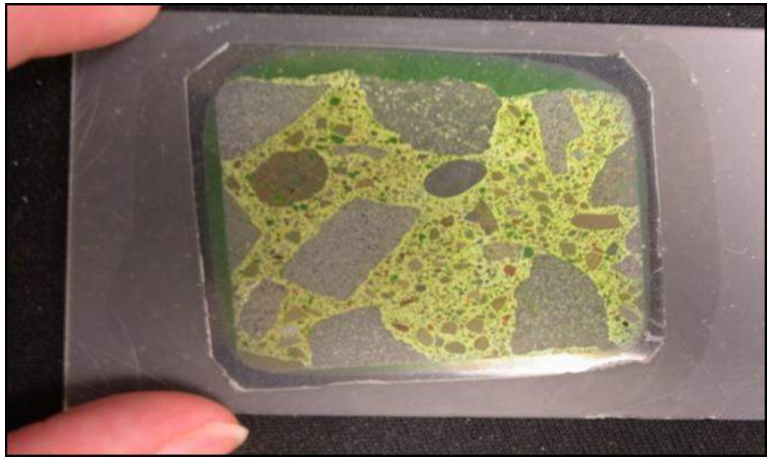
Vacuum impregnated with a fluorescent dye

Cured



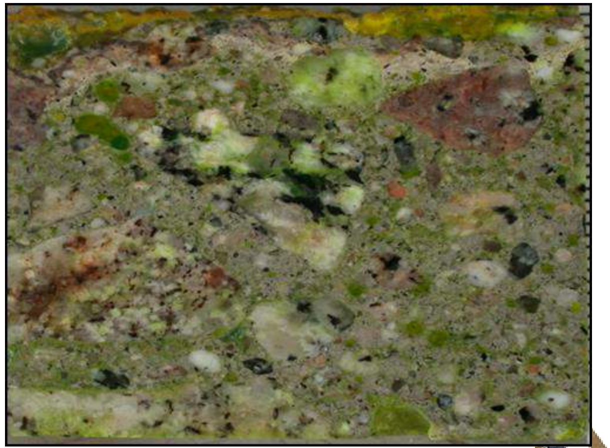
Samples are adhered to a glass slide and prepared as a thin section

And reduced

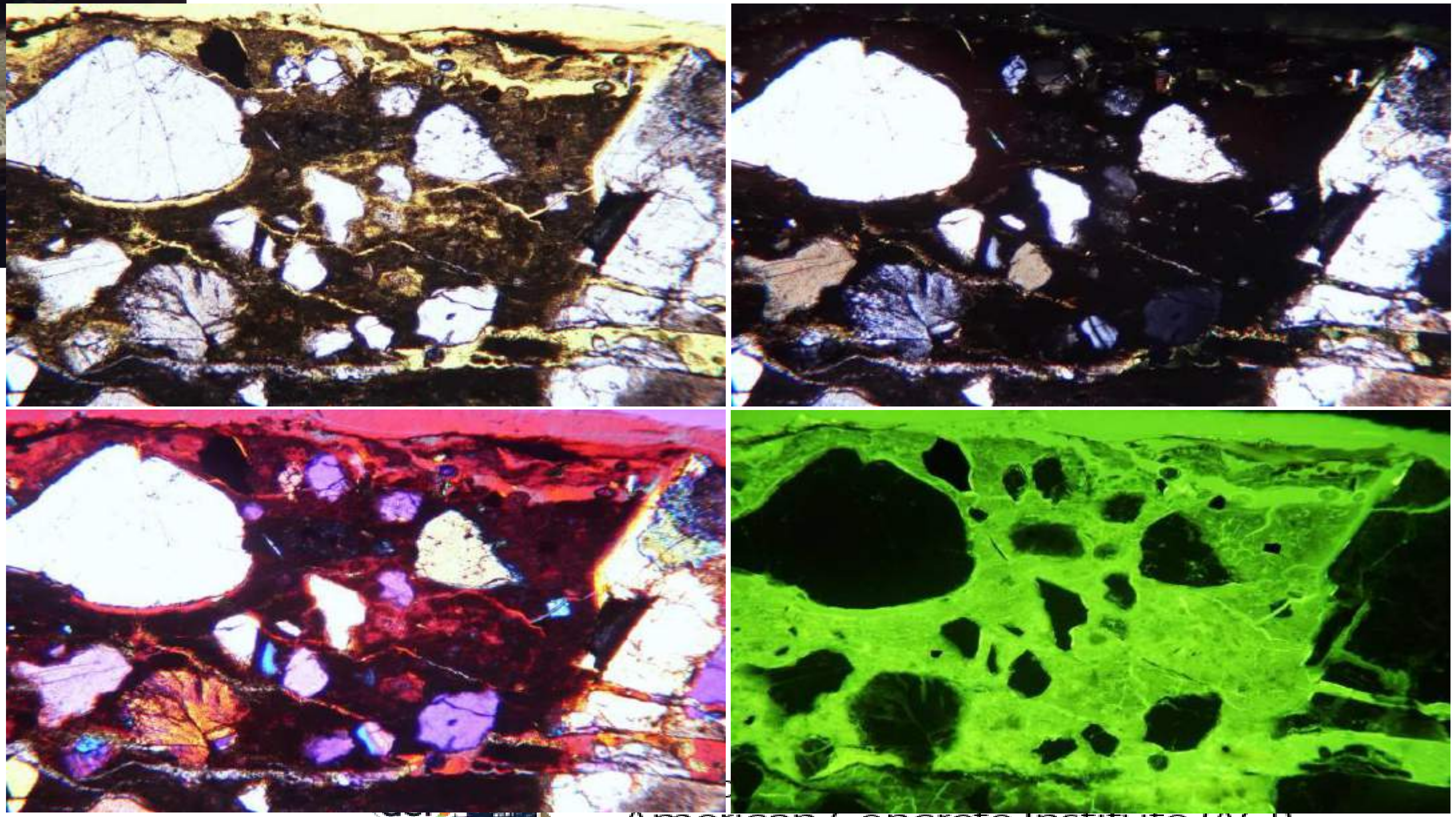


Sample Preparation

The impregnated sections are then polished with successively finer grit to produces a smooth surface



Polarized Light Microscopy



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

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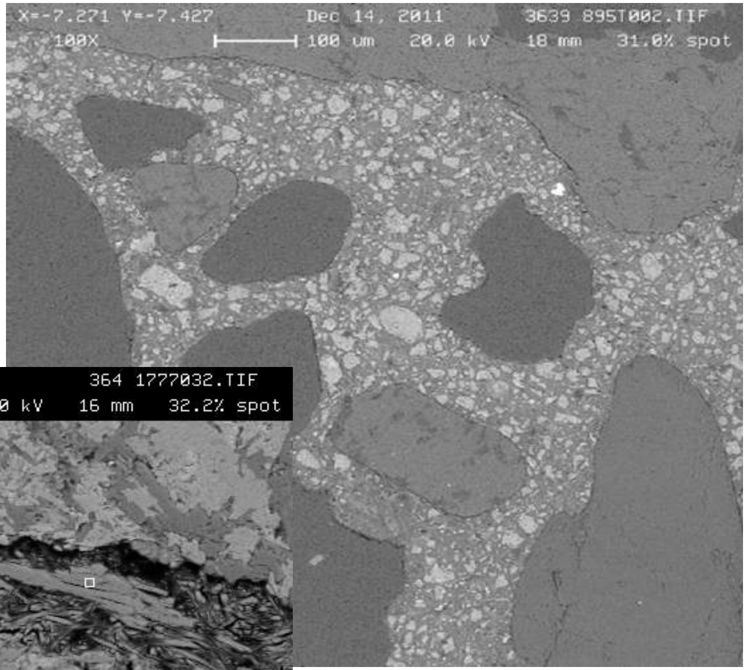
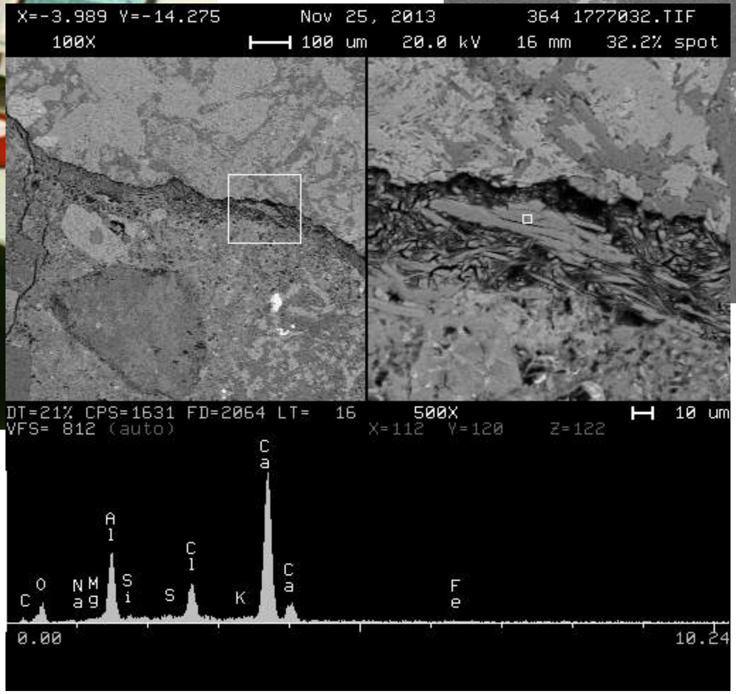
Scanning Electron Microscope with EDS





Supplements Optical Microscopy with Compositional Information

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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

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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

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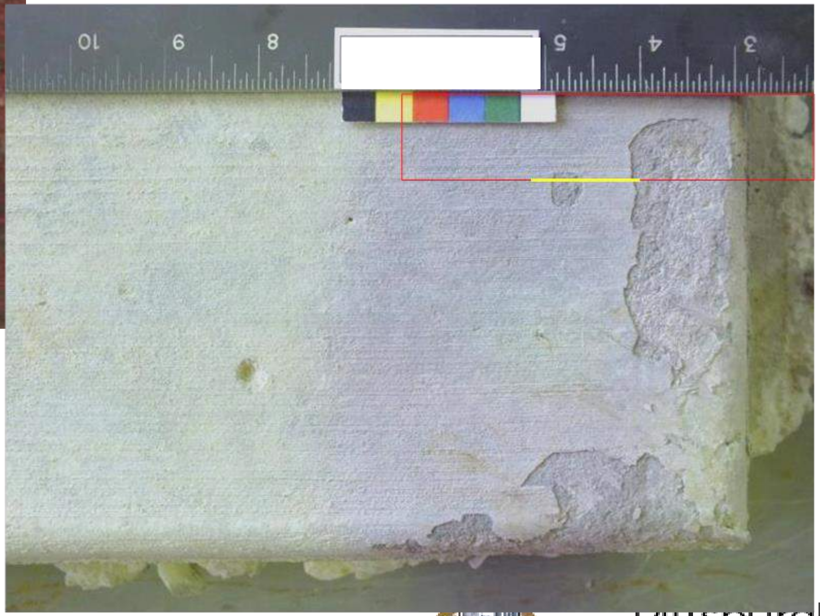
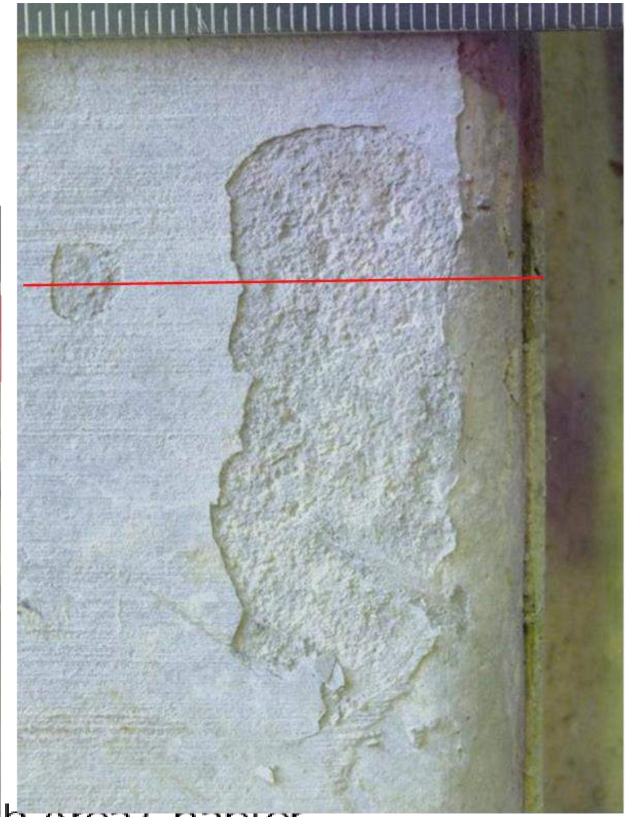
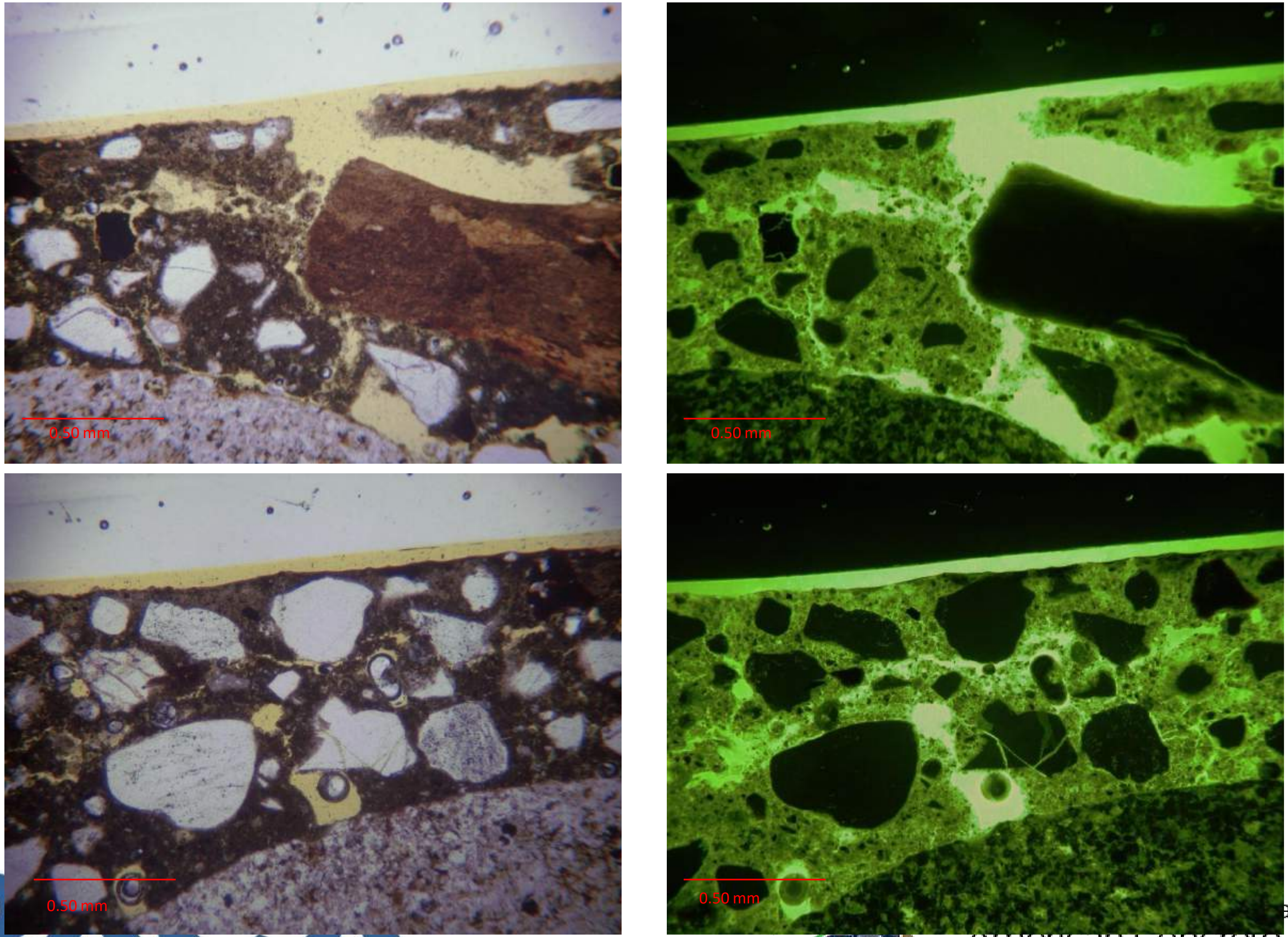
Common Causes of Poor Surface Durability Related to Finishing

Premature Finishing Entrapped Bleed Water





Common Causes of Poor Surface Durability Related to Finishing



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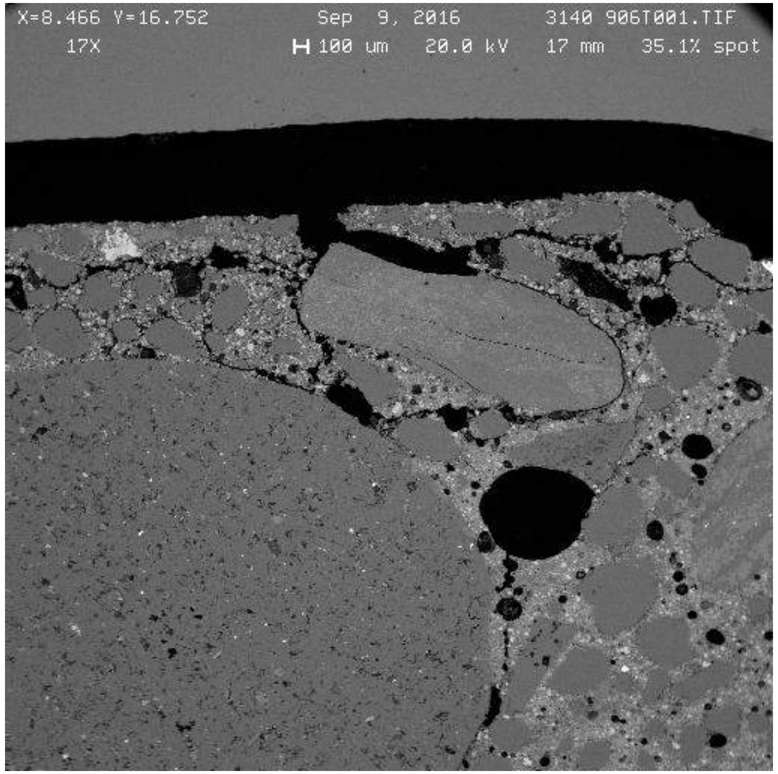
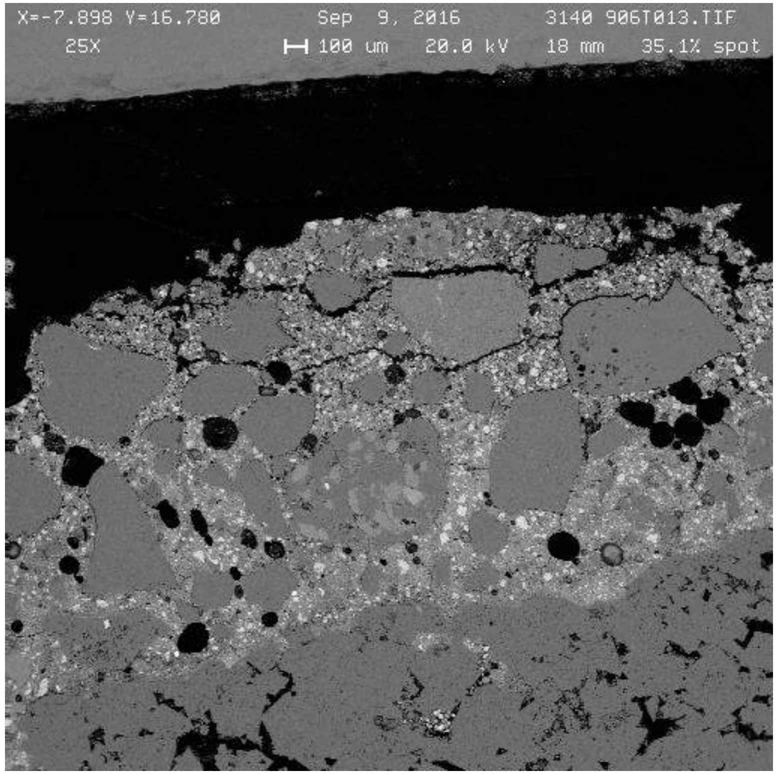
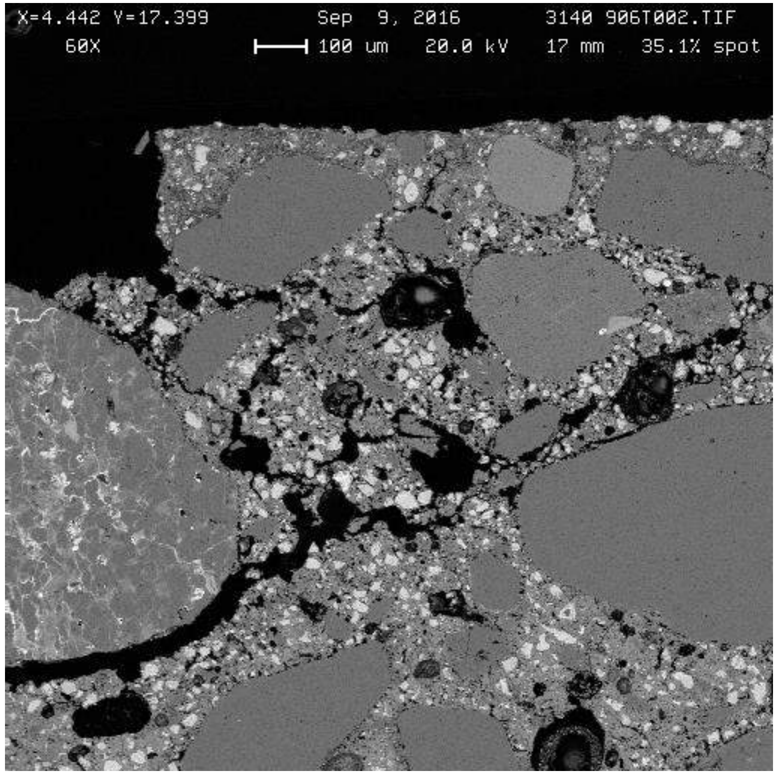
Common Causes of Poor Surface Durability Related to Finishing







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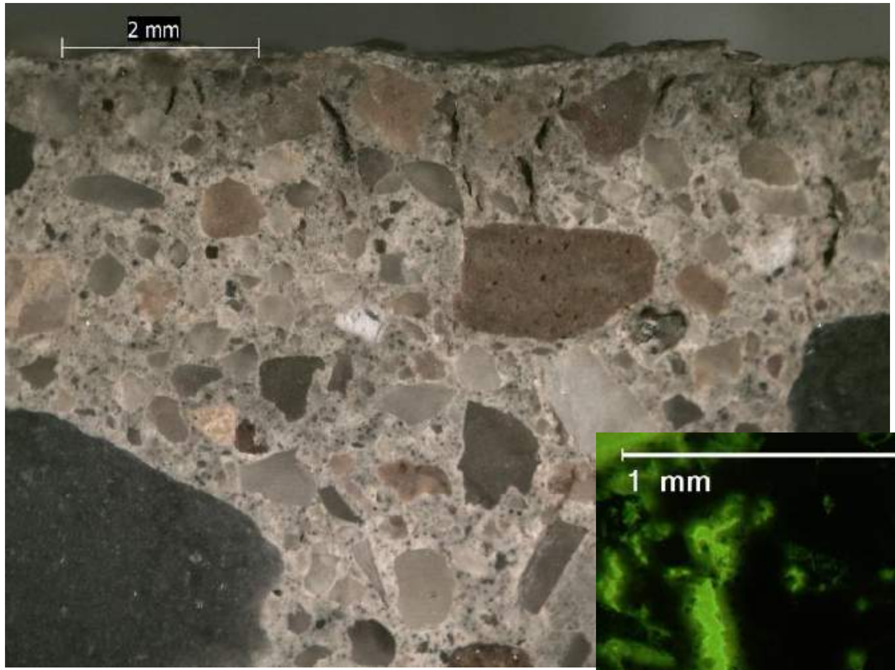
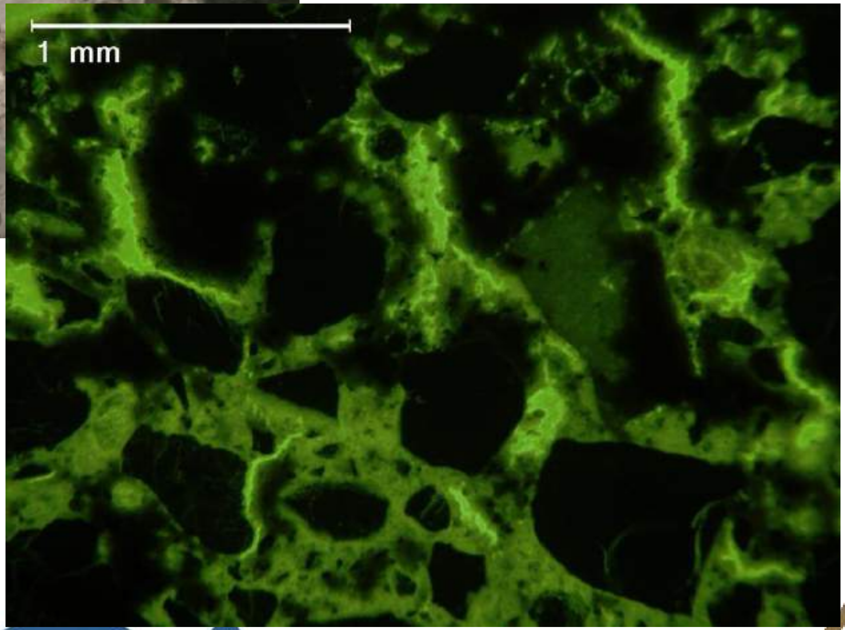
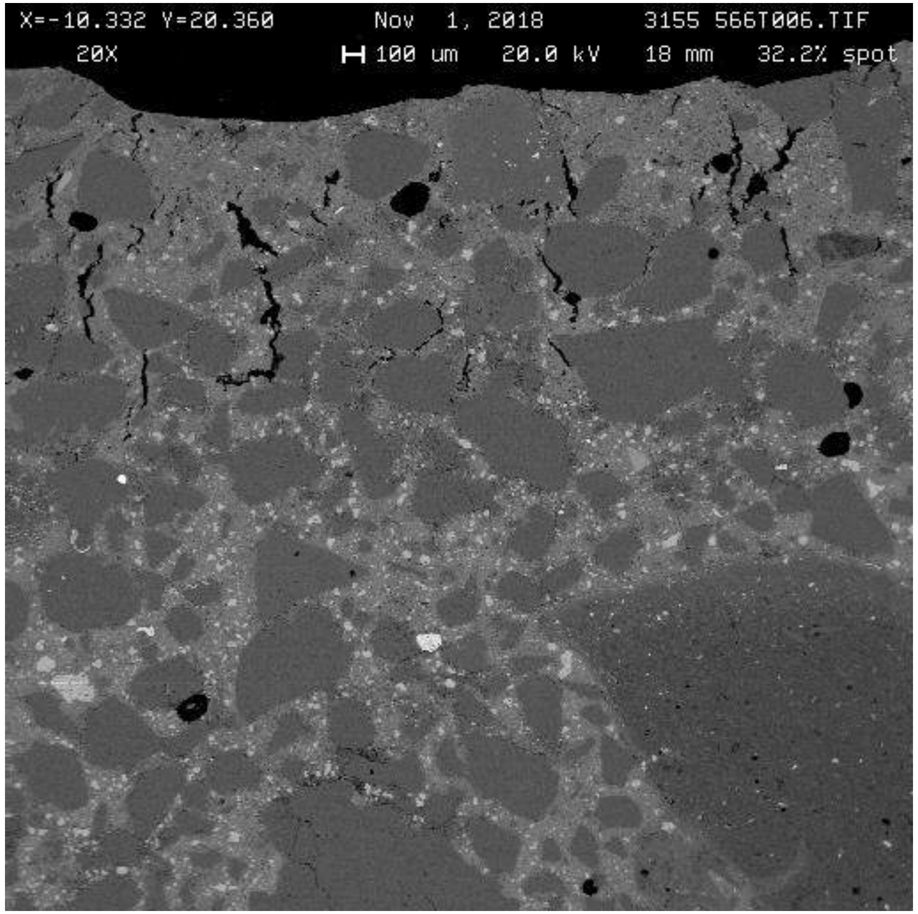
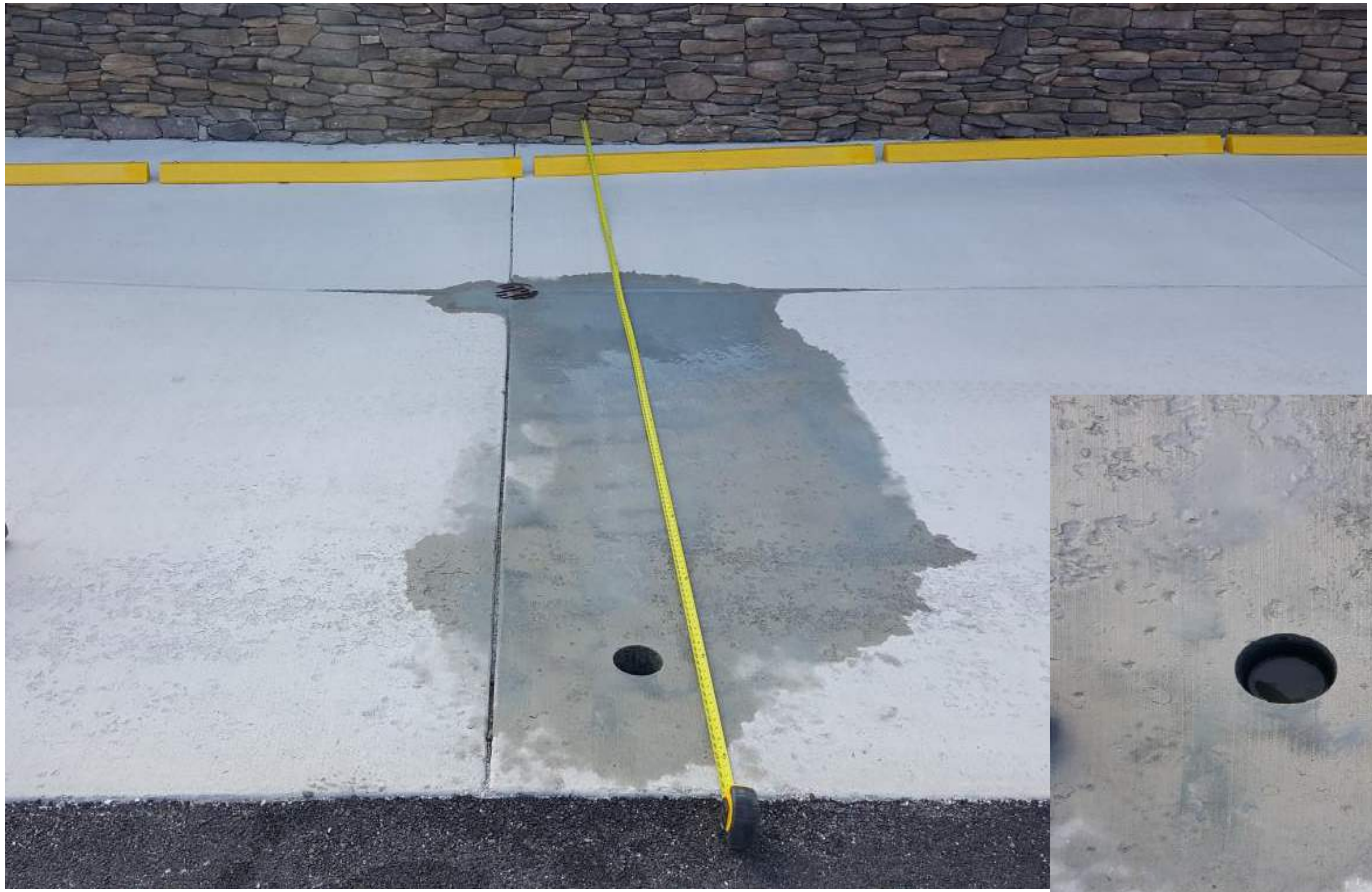
Common Causes of Poor Surface Durability Related to Finishing







Common Causes of Poor Surface Durability Related to Finishing
Increased W/Cm-ratio at surface



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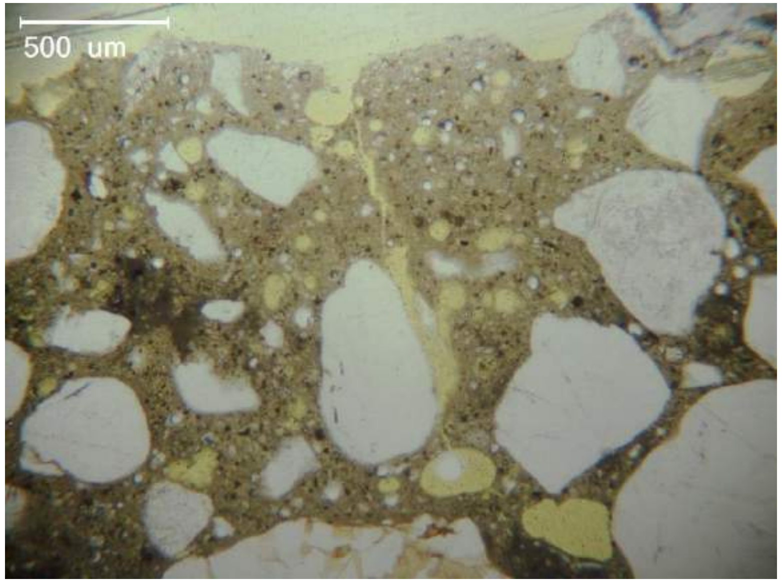
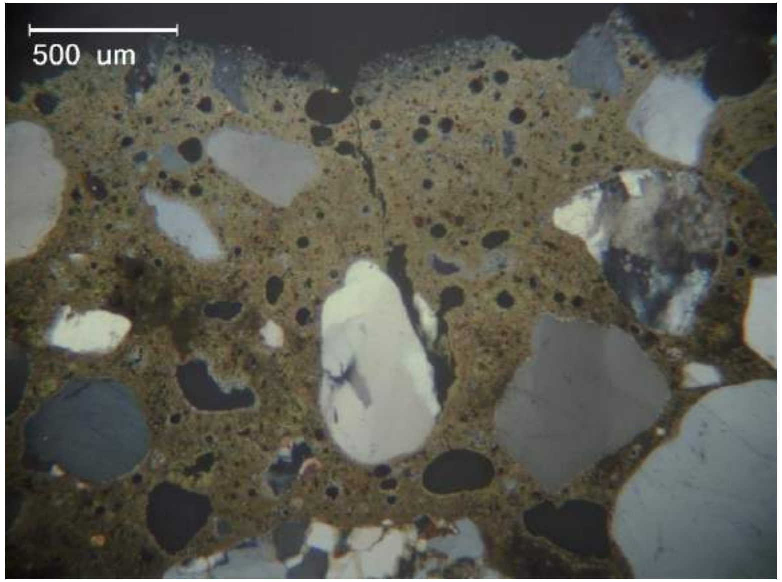
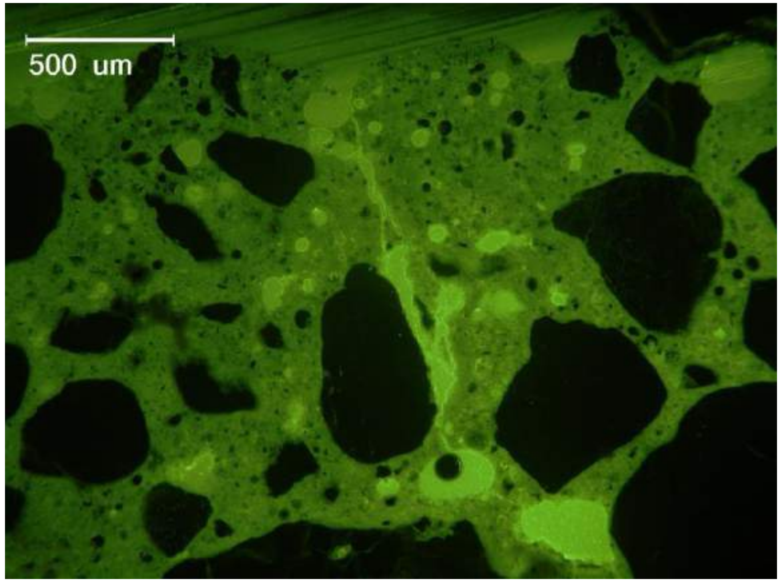
Common Causes of Poor Surface Durability Related to Finishing

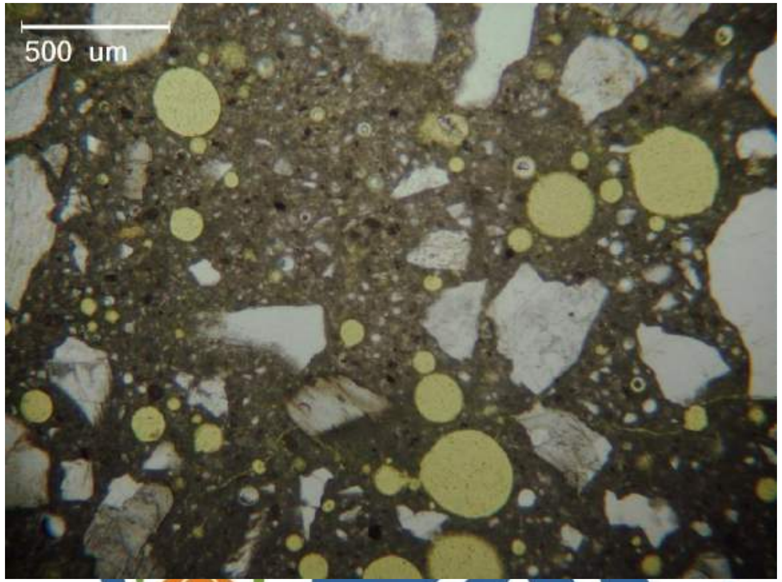
Surface

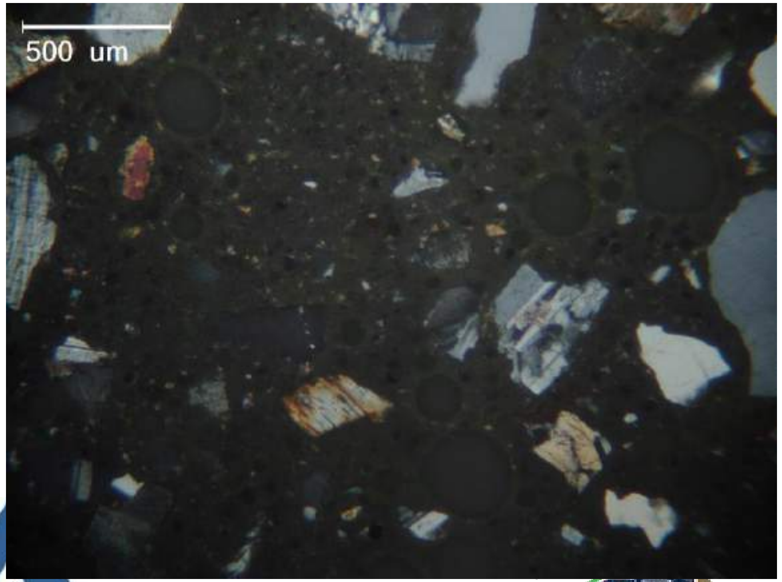


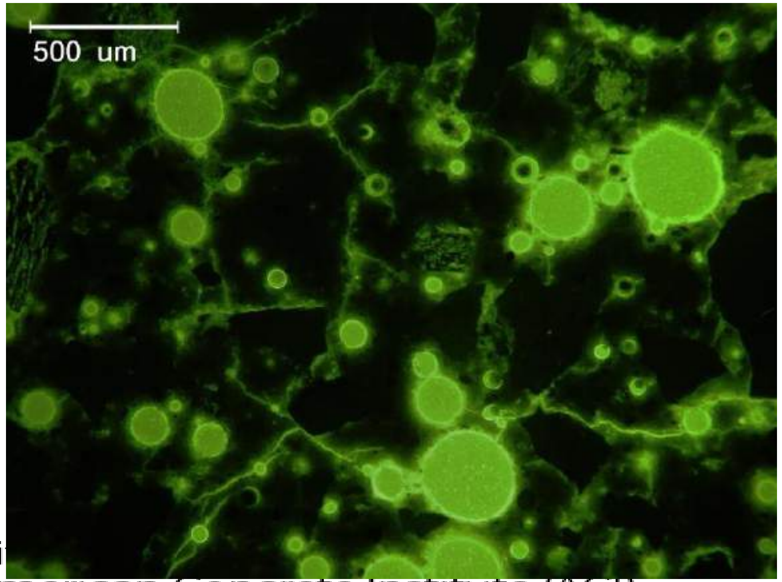




Depth of 1”







Plane Polarized Light

Cross Polarized Light

Fluorescent Light

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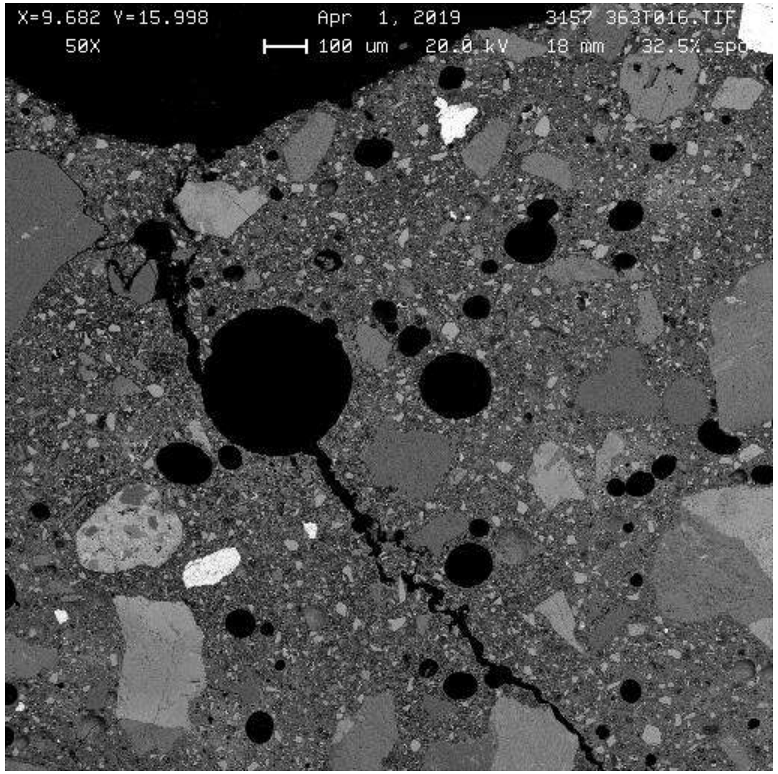
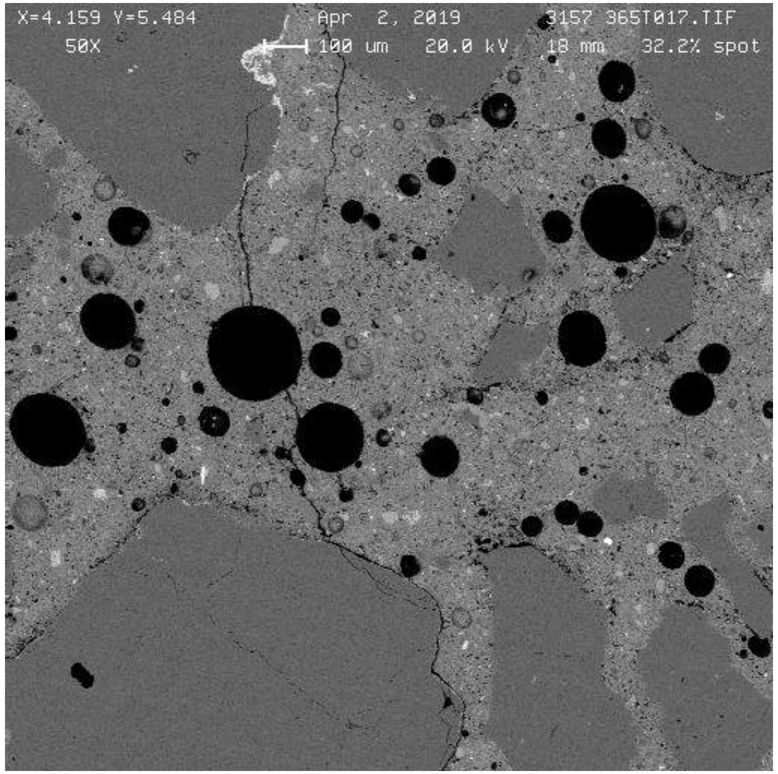
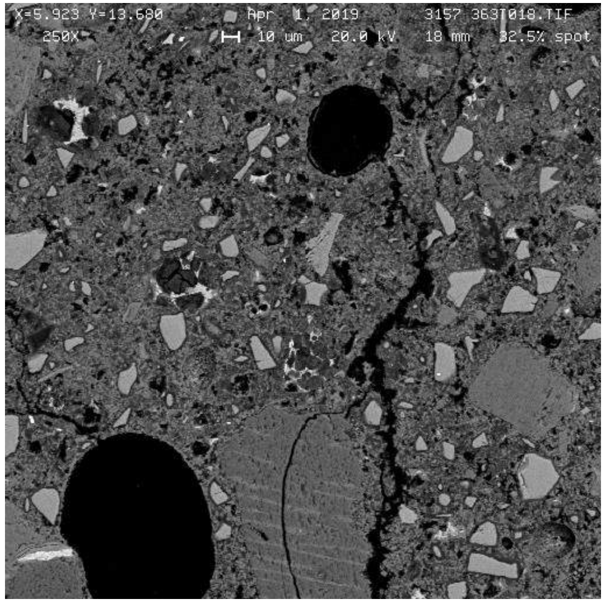
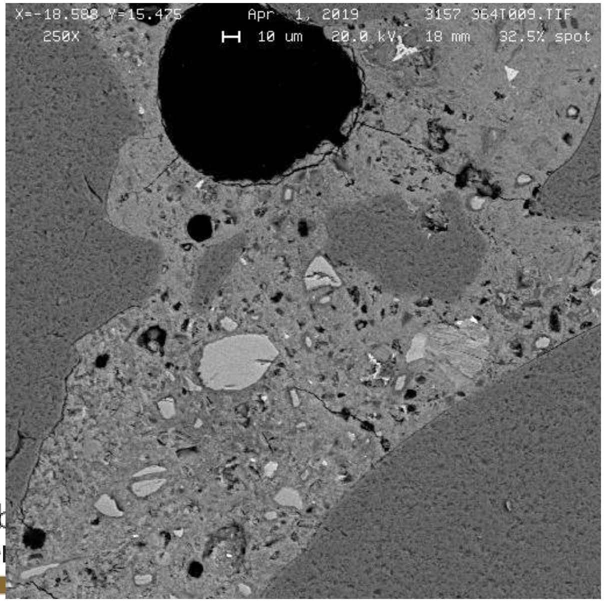
Common Causes of Poor Surface Durability Related to Finishing



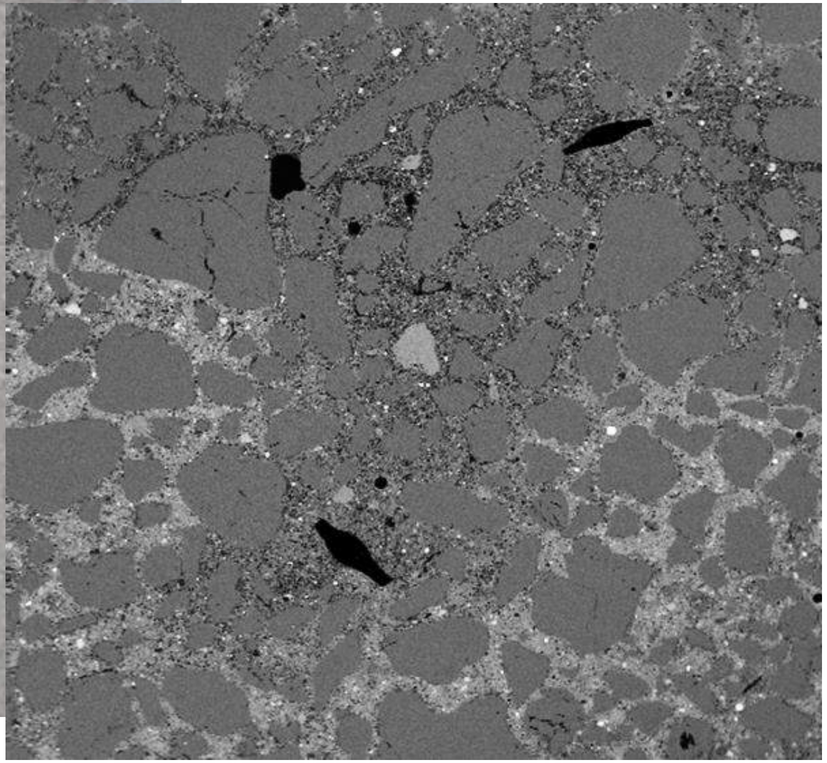






Surface Distress: Scaling

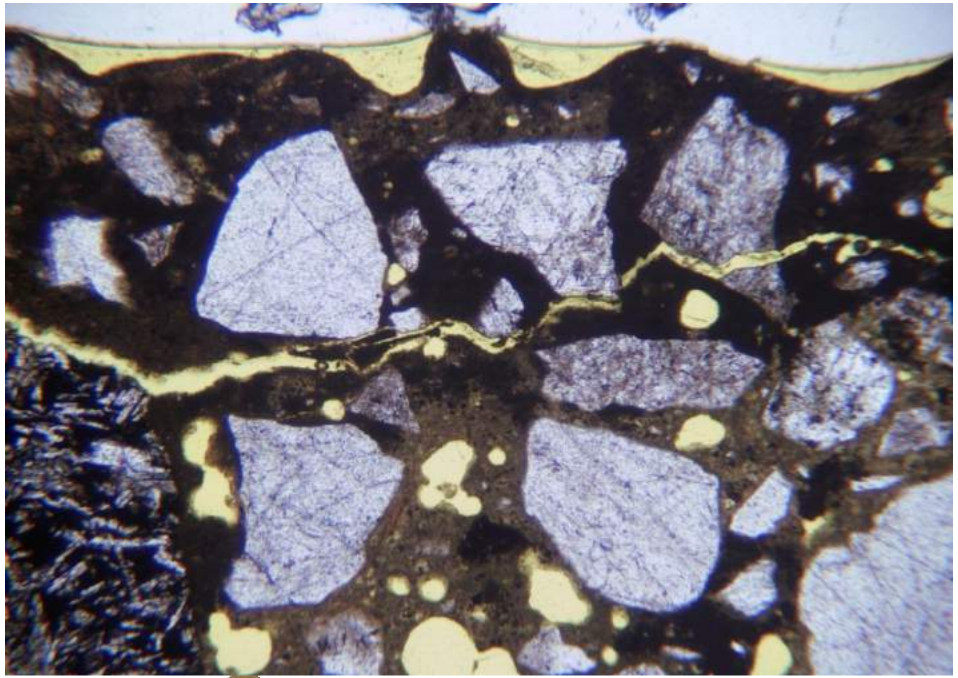
Finishing Issue: Re-tempering & addition of water



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.



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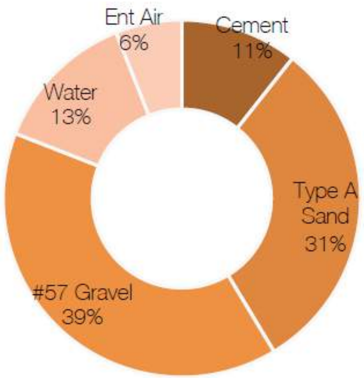
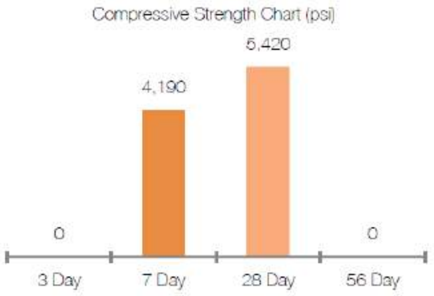
Surface Durability Study

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

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





Early Finish	On-time Finish with Cure	On-Time Finish without Cure	Late Finish
A	B	C	D

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Surface Durability Study

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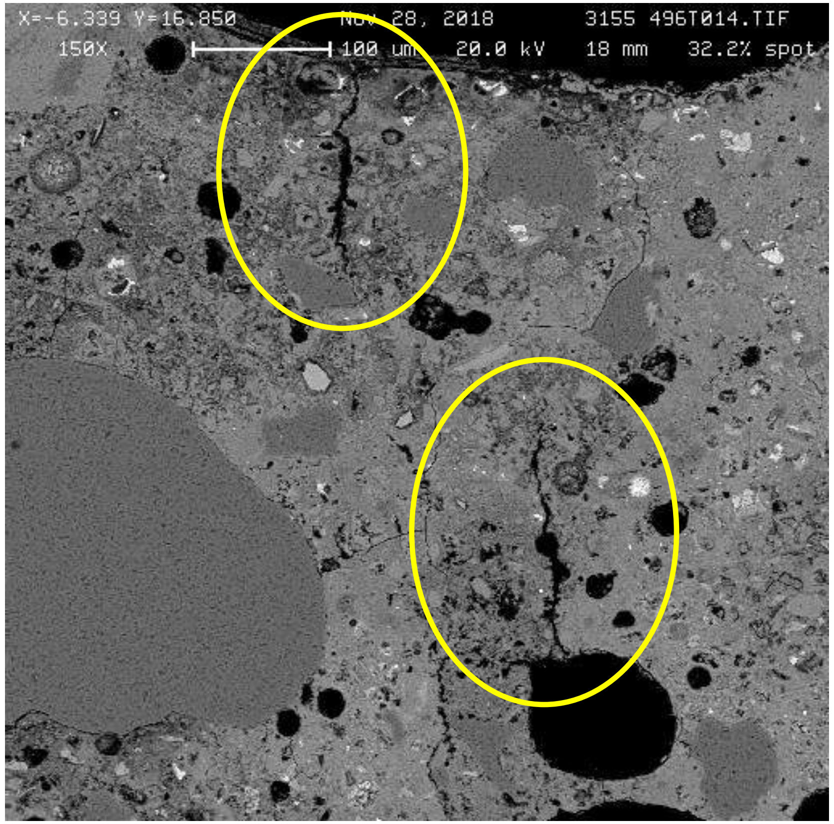
Surface Durability Study

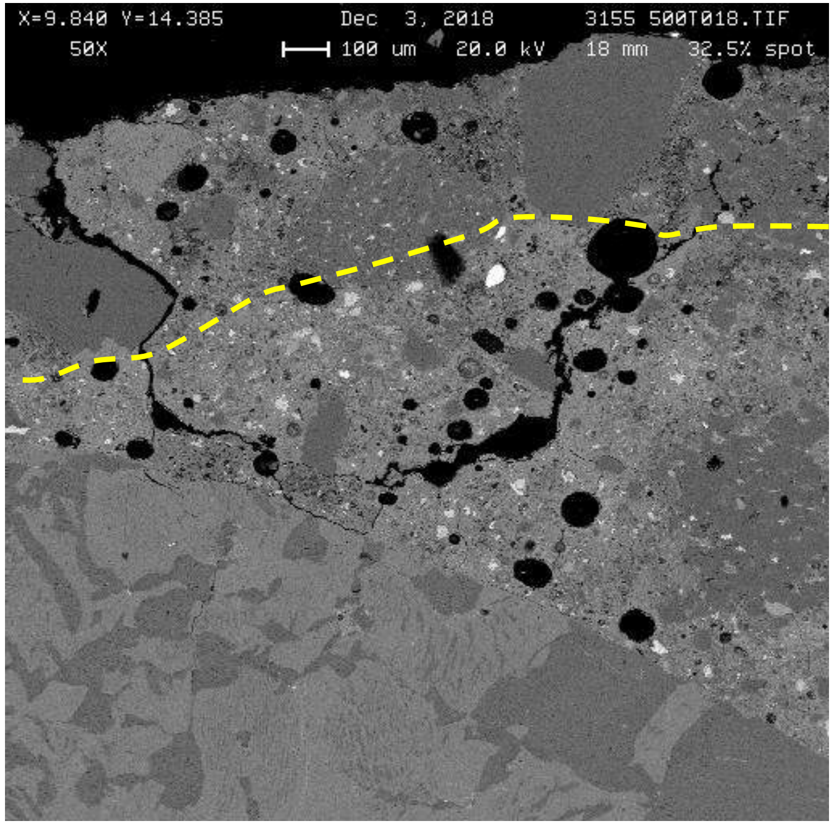
What Did We Do

- The slabs were placed in the fall and we extracted cores the following spring.
- Petrographic examinations were performed following ASTM C856 and C1723

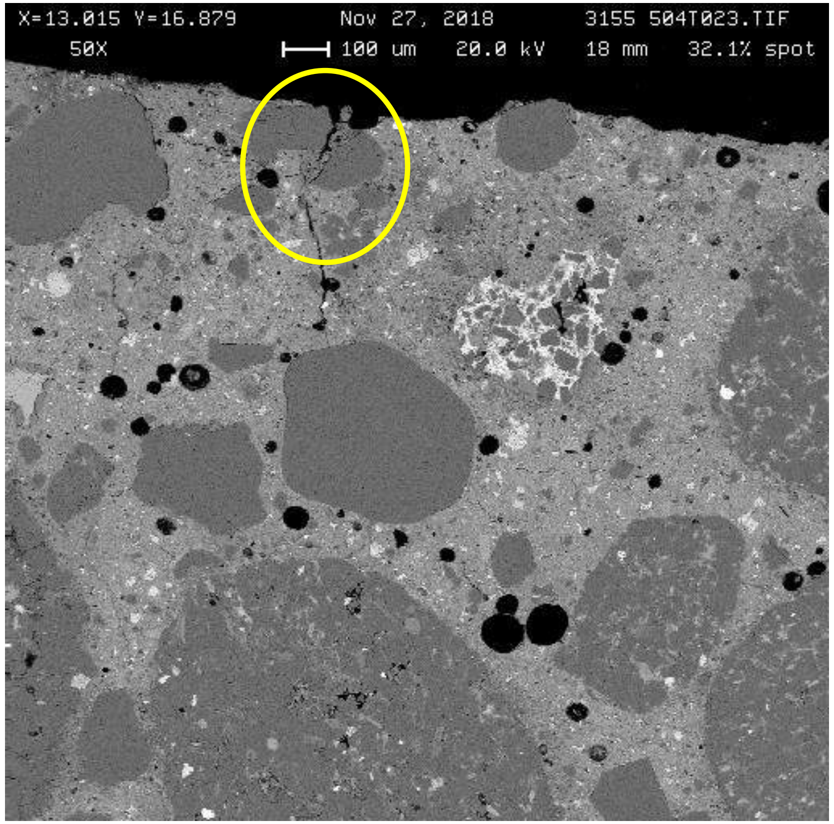
Affects of Early Finishing of Surface Durability Study



0.45 W/C ratio

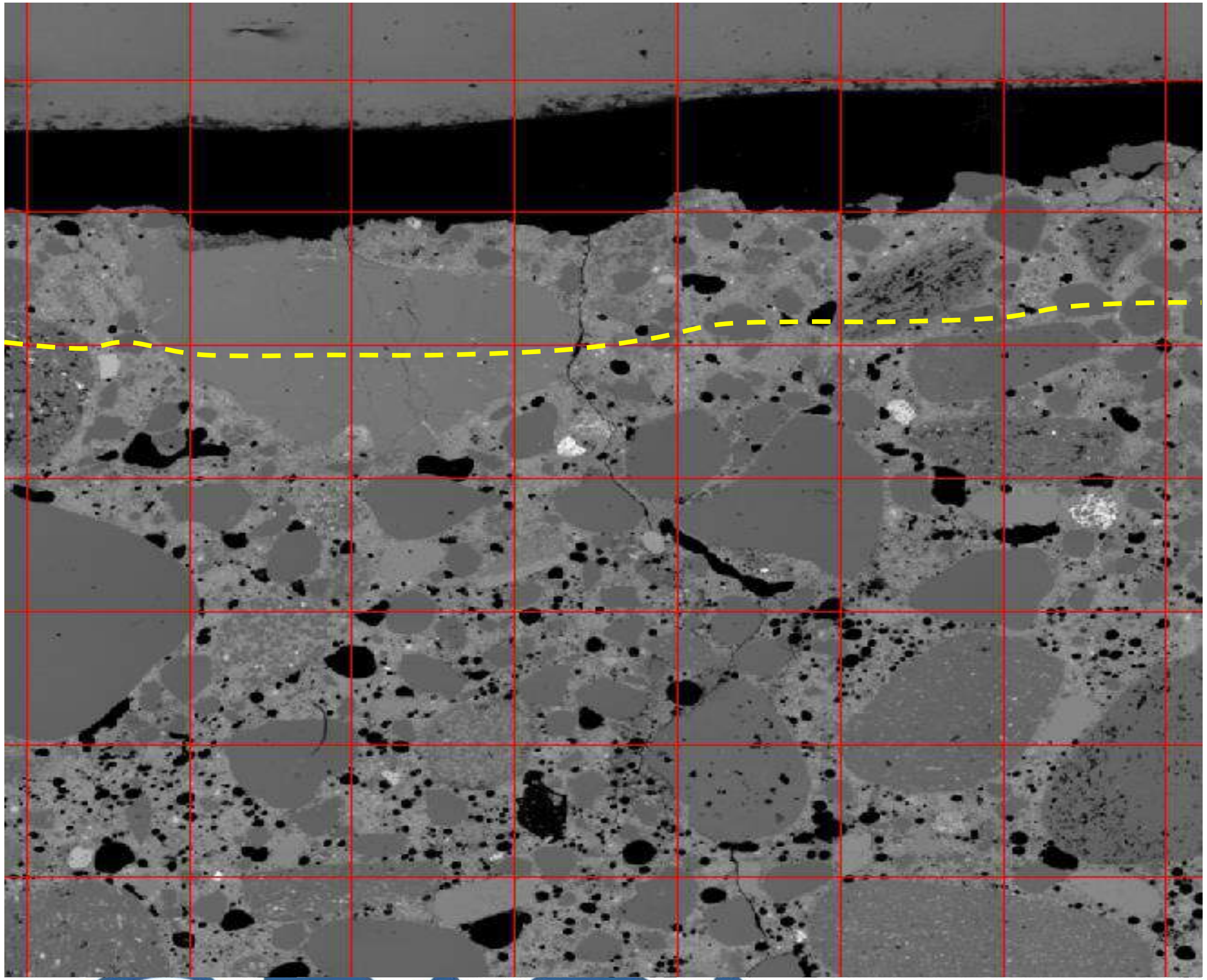


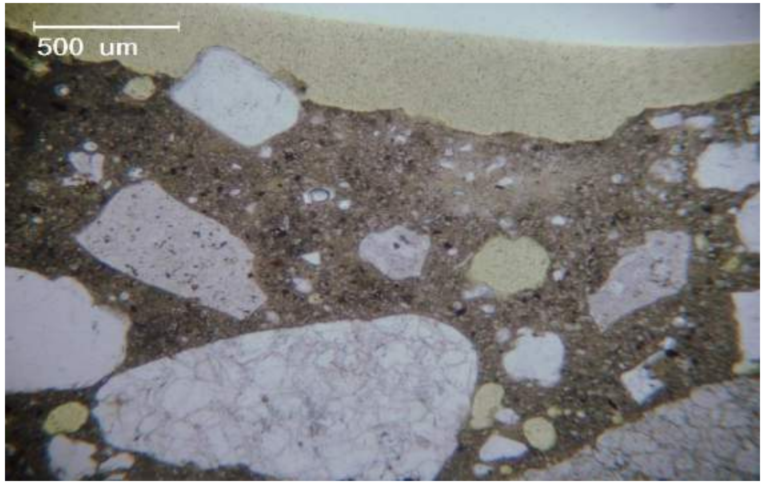
0.42 W/C ratio



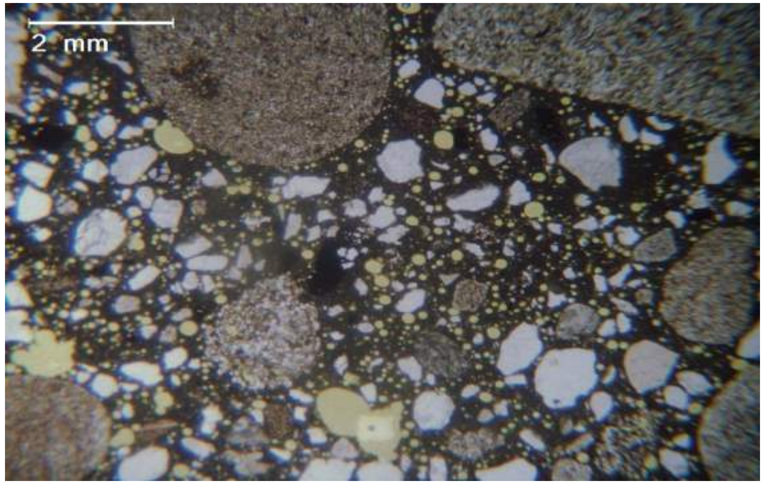
0.39 W/C ratio

Affects of Late Finishing of Surface Durability Study



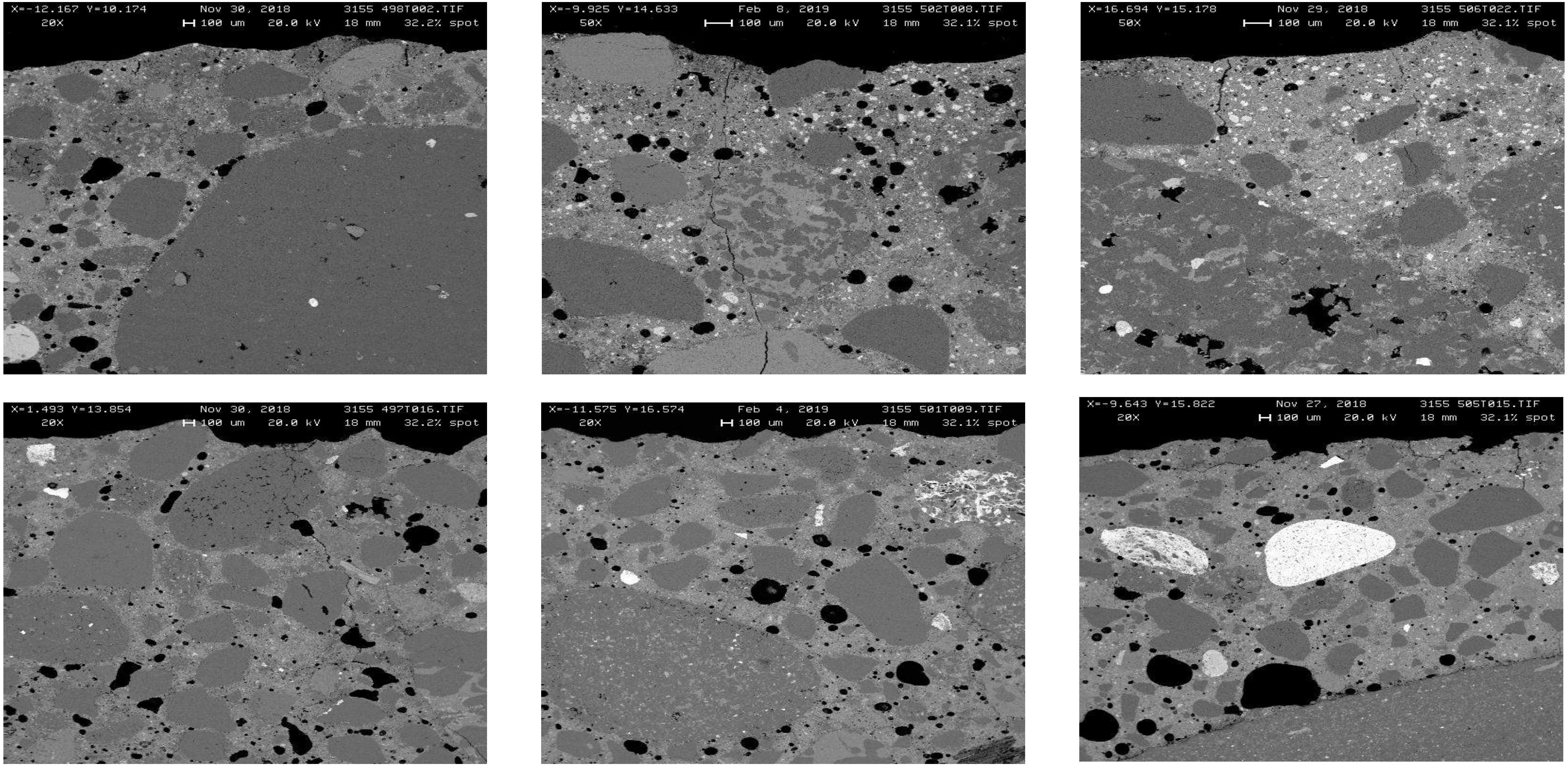


Field of View 2.6 mm



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

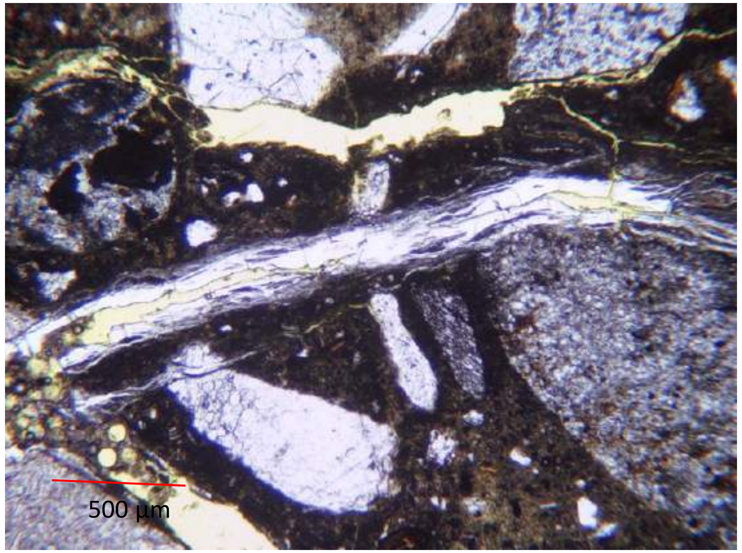
Conclusions Discussion

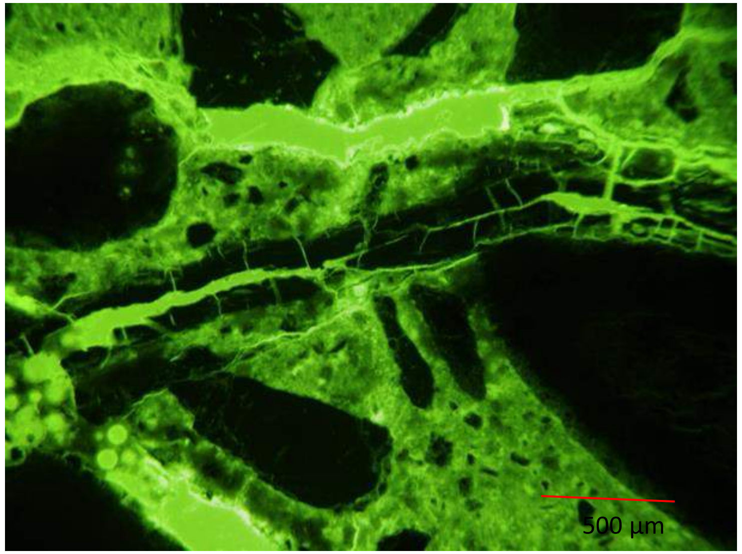
- Microstructural differences were slight.
  - We were not able to re-create the gross differences we wanted to express with the study.
  - They were so slight that we did not anticipate any significant difference in abrasion test results and so have not performed them as of yet.
- Factors:
  - Mix designs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





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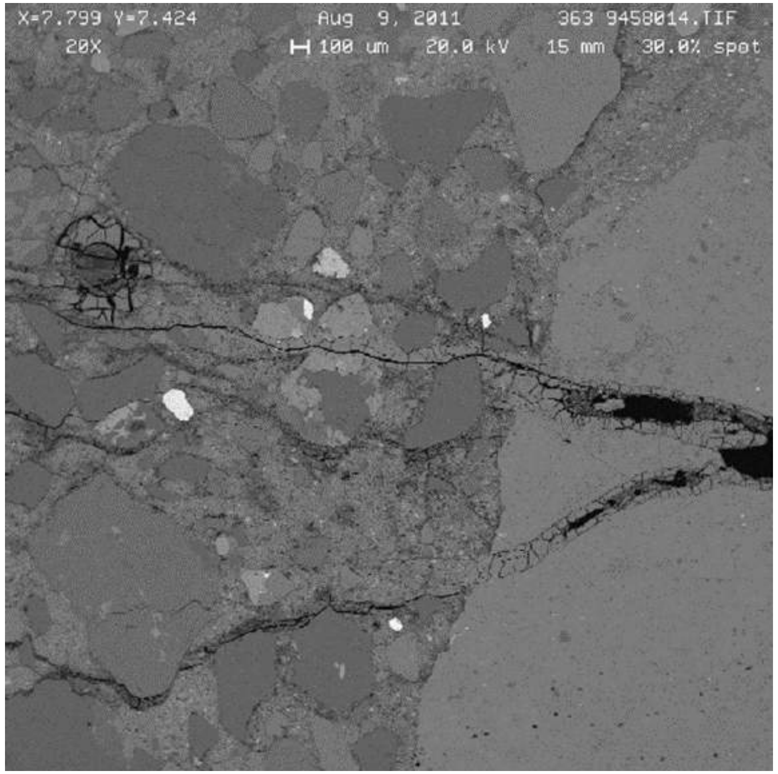
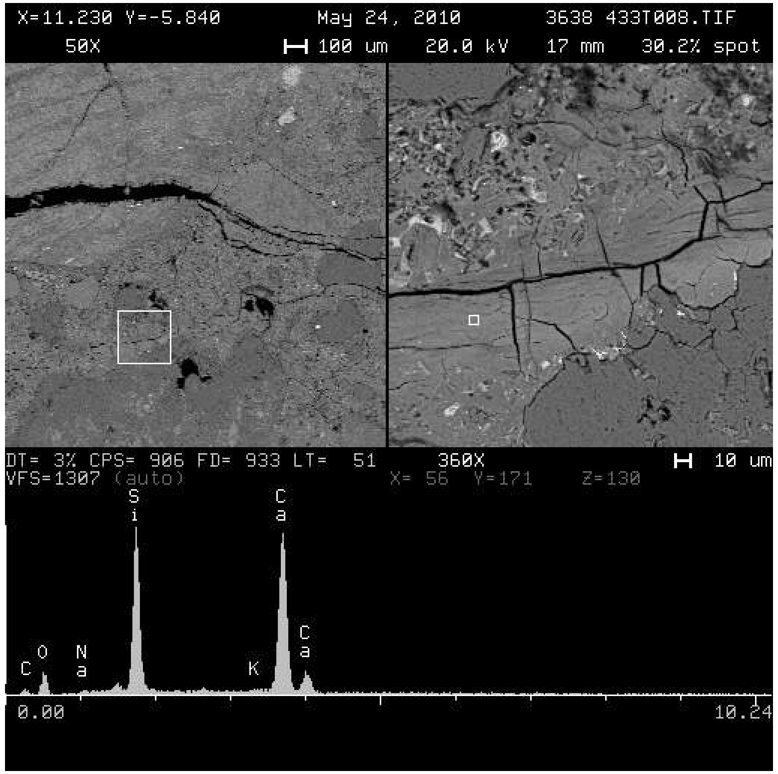
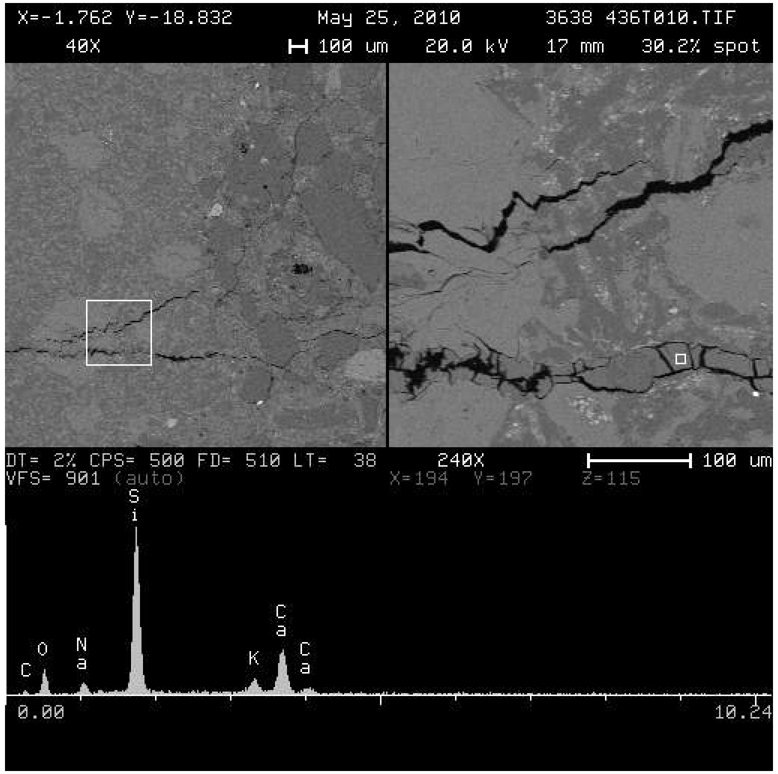
ASR – Diagnosis & Prognosis Info





Aged/Old Gel in crack in paste



Fresh gel within crack in aggregate

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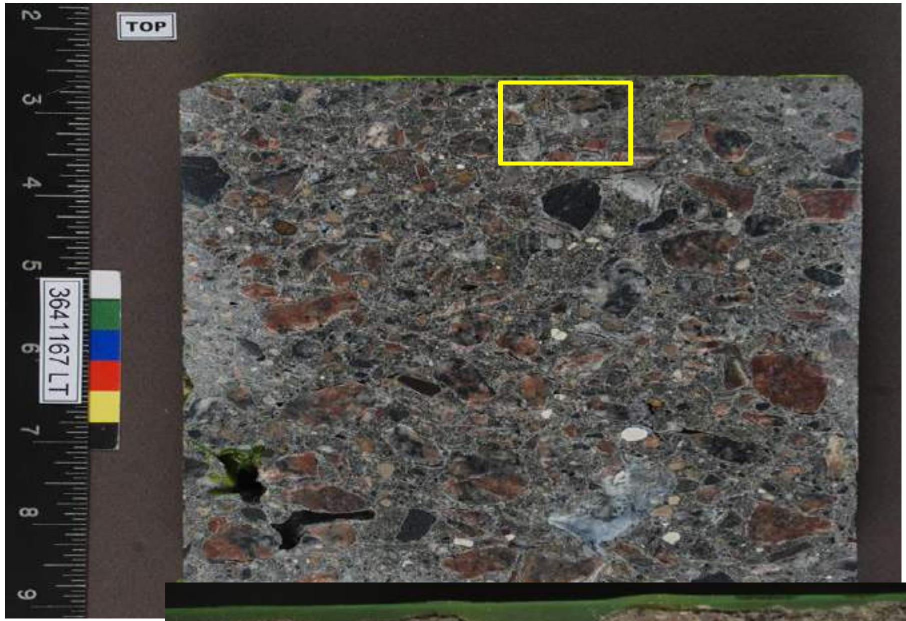
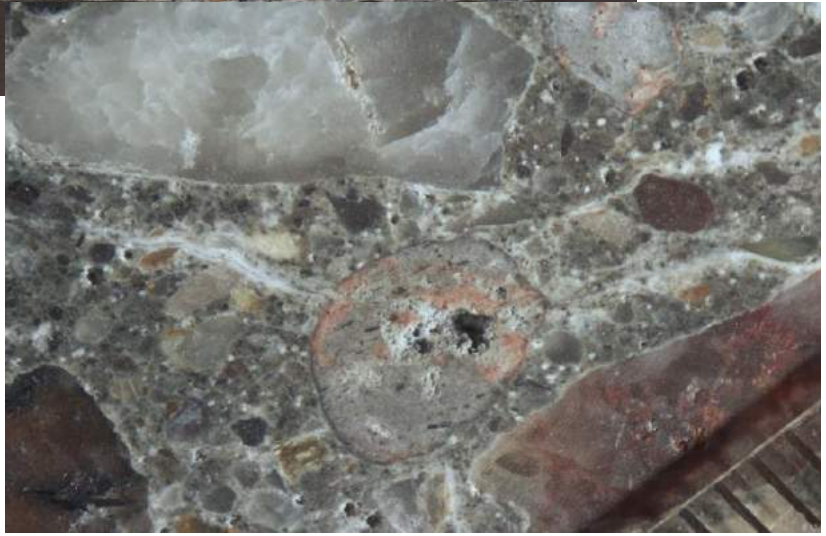
Alkali Silica Reaction Case Study

- 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

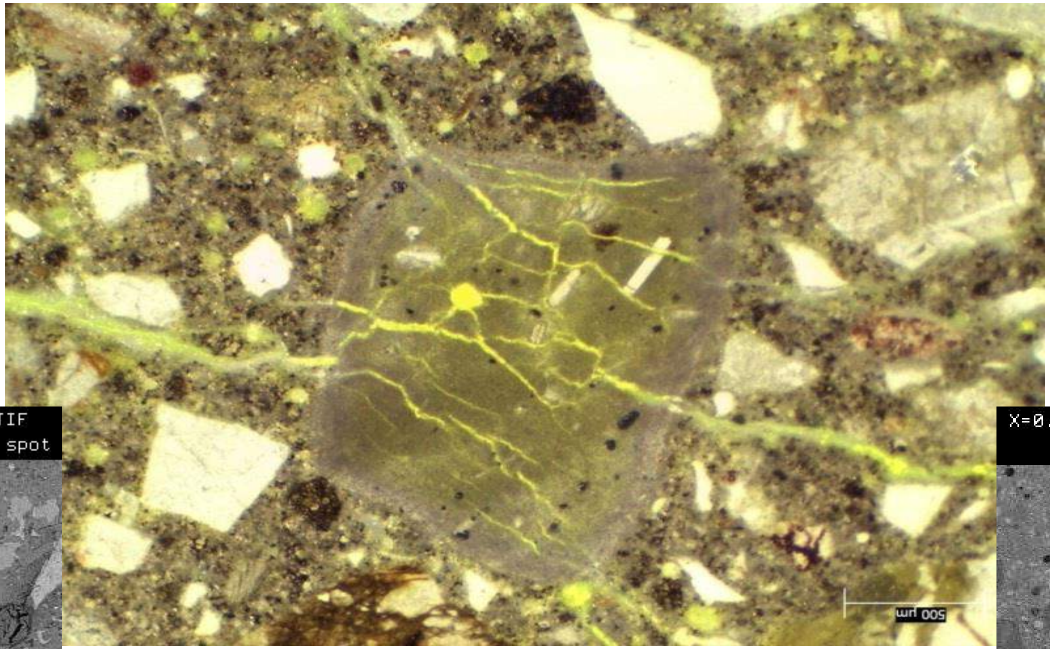
Alkali Silica Reaction Case Study

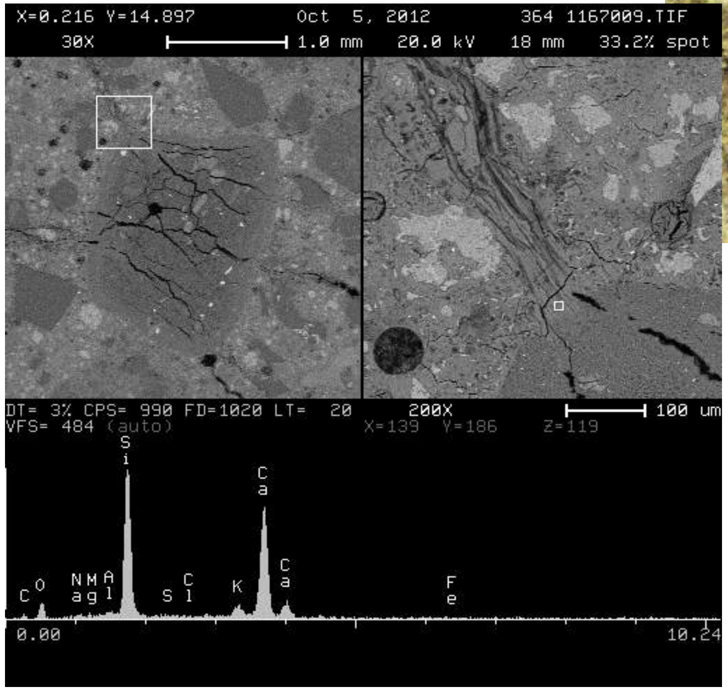


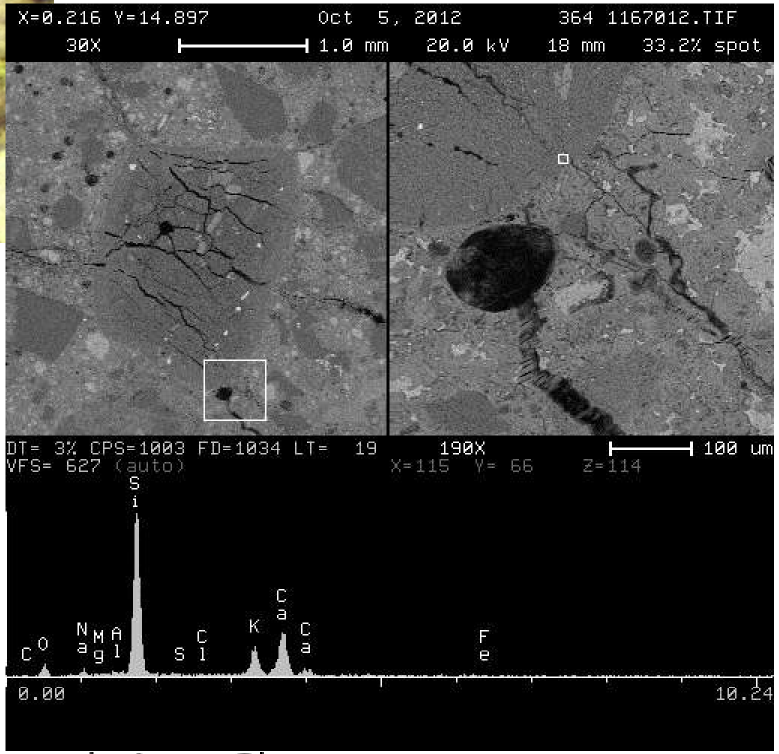


Alkali Silica Reaction – Case study

Andesite Fine Aggregate
in thin section







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ASR – Repair Evaluation

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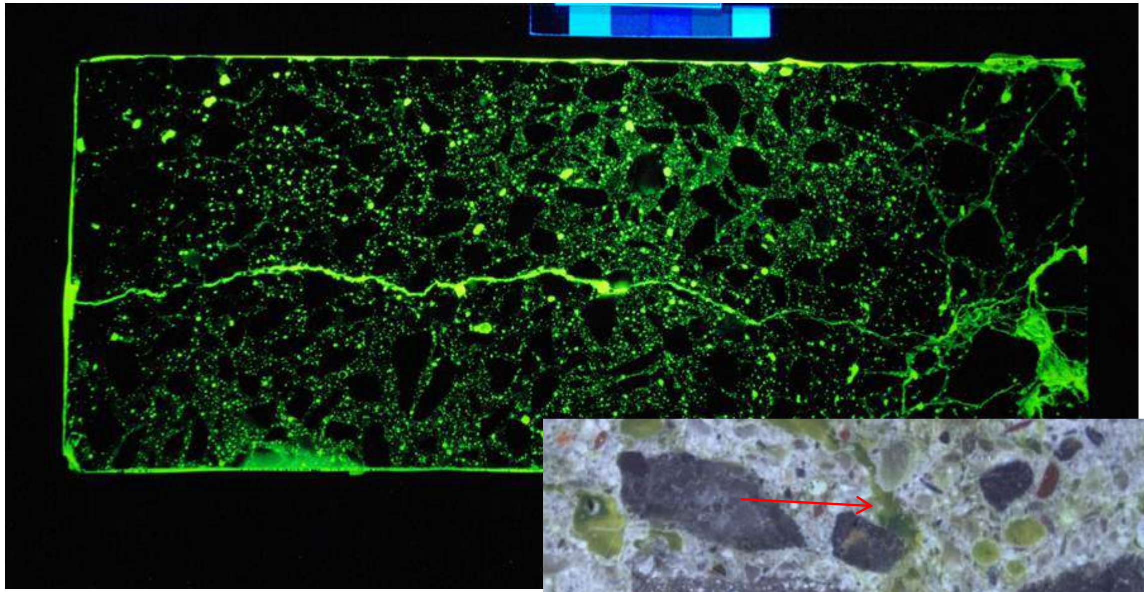
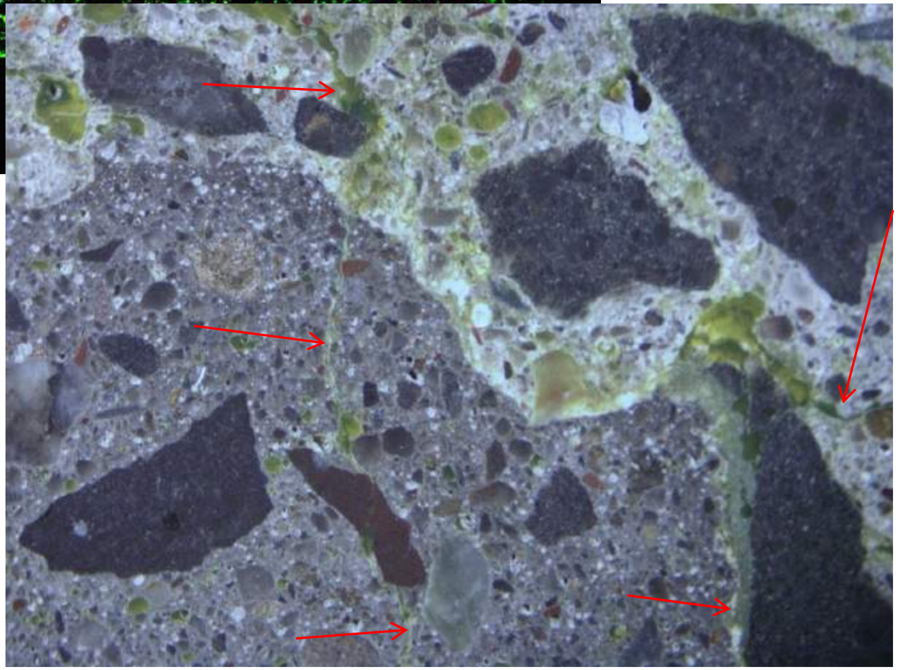
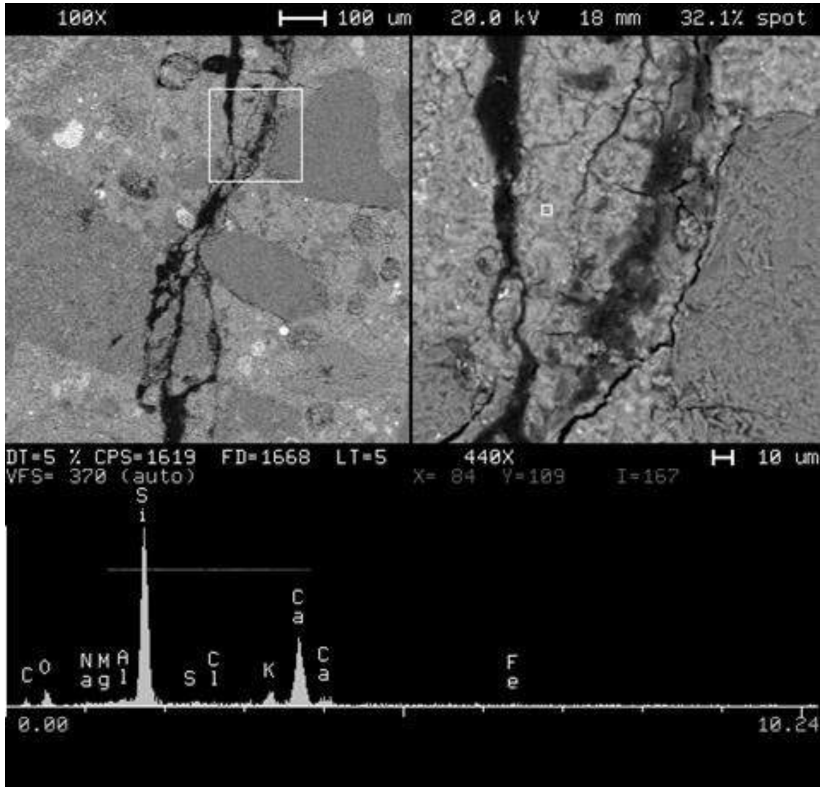
ASR – Repair Evaluation



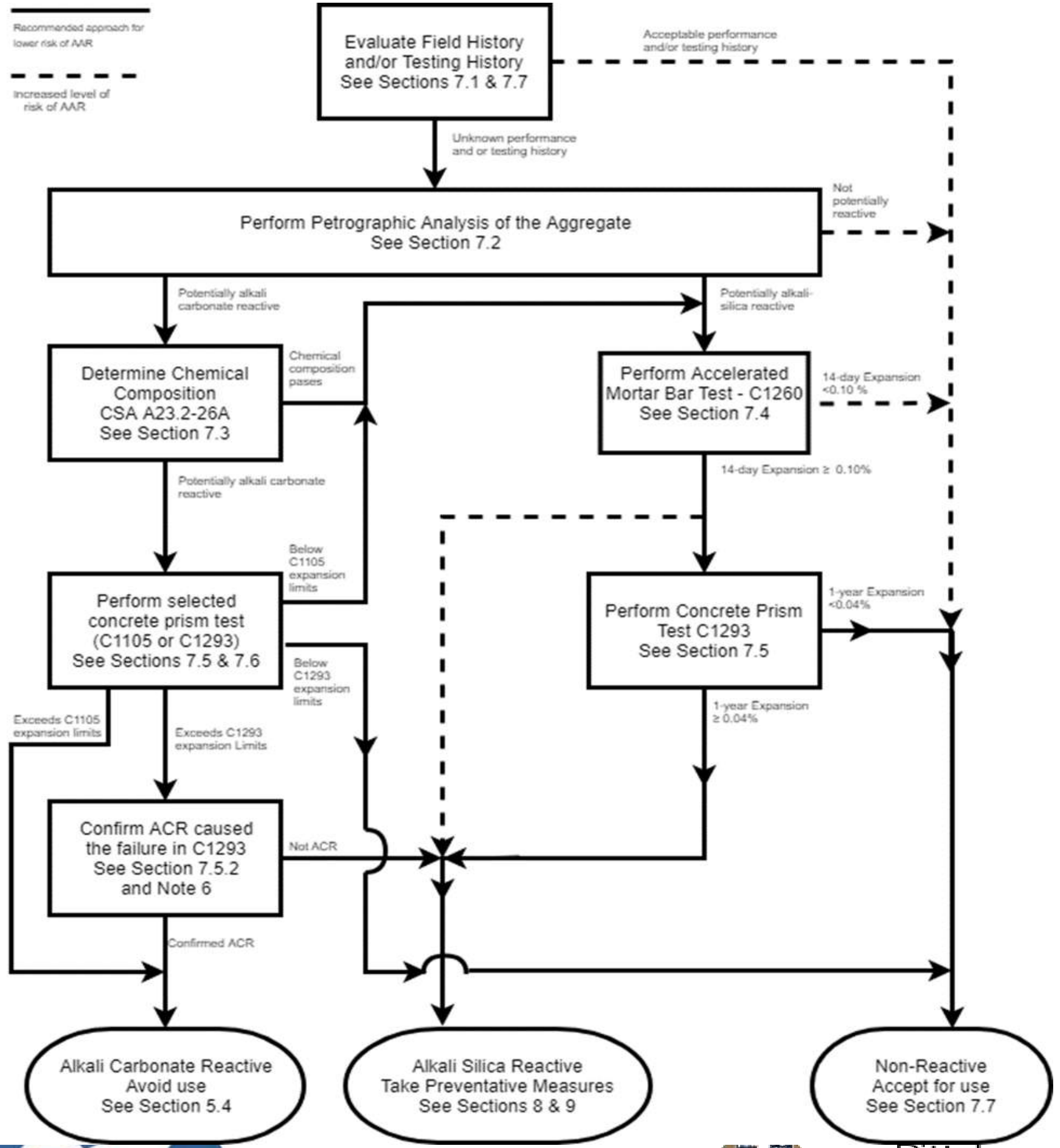




ASR Practice Document(s)

- 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



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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

Aggregate and Concrete Petrography

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

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