

Task 19: Reactive Flow- Through Experiments: A Look at Foamed and CO₂ Resistant Cements

Project Number FE1022403

Rick Spaulding

US DOE/ NETL

Team includes Thomas, R., Montross, S., Crandall, D., Moore, J., Goodman, A., Sanguinito, S.,
Brandi, M., Kutchko, B (TTC - PI)

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Technical Approach

Goal

- The objective of the effort is to investigate and evaluate the fracture opening or self-sealing of foamed cements and CO₂ resistant cements: Flow-through CO₂-saturated brine interactions at subsurface conditions typical in the Gulf of Mexico (GOM).

Research Questions

- Will foamed cements with a leak pathway (i.e. fracture) self-seal in a similar manner as ordinary Portland cement?
- When are CO₂-resistant cements needed?
 - Significantly more expensive than traditional Portland cements.
 - Not compatible with the traditional Portland cements used in other sections of the well.
- When can we use Portland cements and when should we use a specialized cement?
- These answers will improve safety, well integrity, and have significant economic benefits.

Approach

- It is unfeasible to run experiments on every single variable that exists in the subsurface. Therefore, the team needs to understand the fundamental mechanisms to make predictions.
- Flow-through experiments are being conducted on various cement formulations.

Current Research Scope

Samples

1. Generate foamed cement using API RP 10 B-4 procedures
 - Different foam qualities (20%, and 30% gas volume)
2. A commercially available CO₂-resistant cement
 - Fly Ash-modified Calcium Aluminate Phosphate Cement
3. Coal-derived Engineered Carbon
 - including graphene oxide, graphene flakes, coal-based graphene flakes, and coal-based carbon dots

Experiments

1. Cement cores fractured using the Brazilian method
2. Uniaxial Hasler cells with a confining pressure to create flow through the cement core
 - Predetermined flow rates for predetermined lengths of time.
 - Constant flow rate short core experiments
 - Constant pressure differential composite core experiments
3. CT- flow-through experiments



Current Research Scope

Analysis

1. Multi-scale computed tomography (CT) scanning*
 1. Resolution of 17 μm
2. Scanning electron microscopy with energy dispersive spectroscopy (SEM-EDS)
3. ATR-FT-IR (Attenuated Total Reflectance-Fourier Transform Infrared Spectroscopy)
4. Mechanical testing

Flow Through

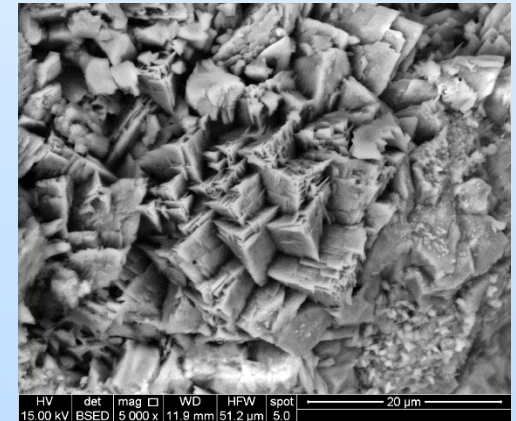
1. Confining pressure = 1200 PSI
2. Pore pressure = 800 PSI
3. DI water at equilibrium with CO_2 as injected fluid (room temperature)
4. Flow rate of 0.1 and 0.5 ml/min

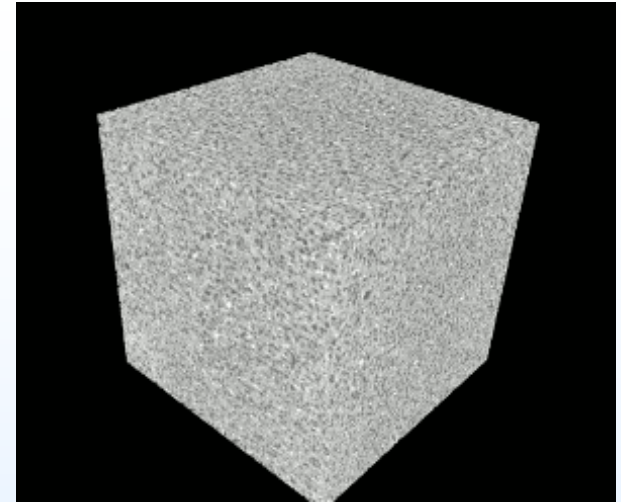
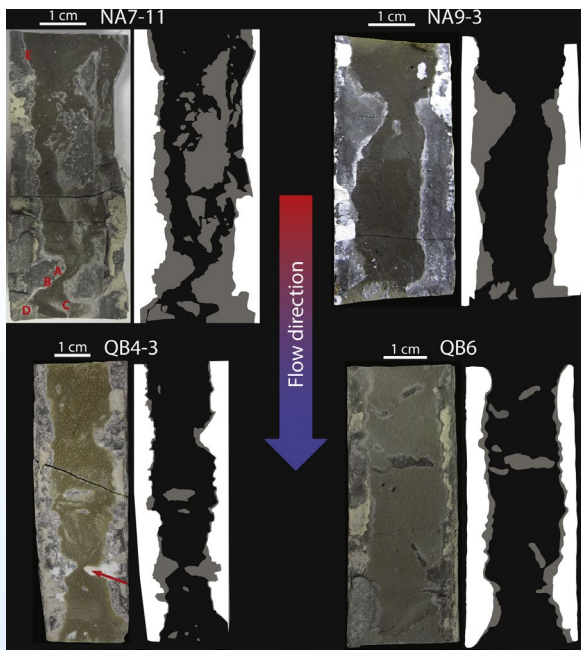
ImageJ Processing

1. Images were scaled by 50% (reduction to 0.5 in X/Y/Z)
 - a. Size management is critical to processing speed and efficiency
 - b. Images underwent bright outlier removal at 2-pixel radius
2. Images were then filtered using 2x2 mean (3-D)
 - a. Processes facilitated easier segmentation & feature isolation

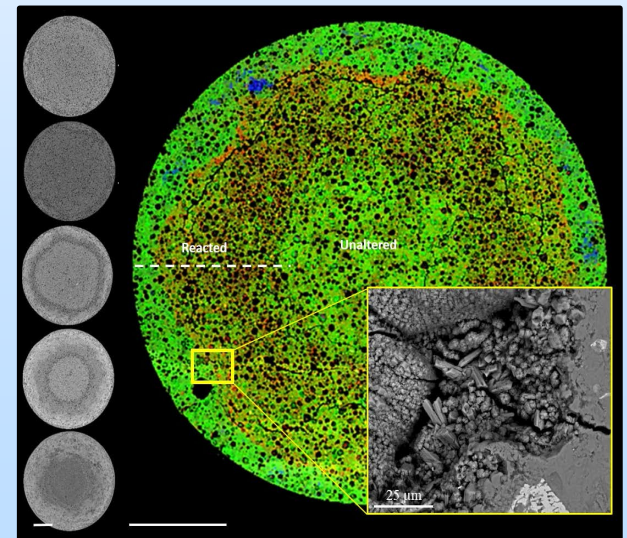
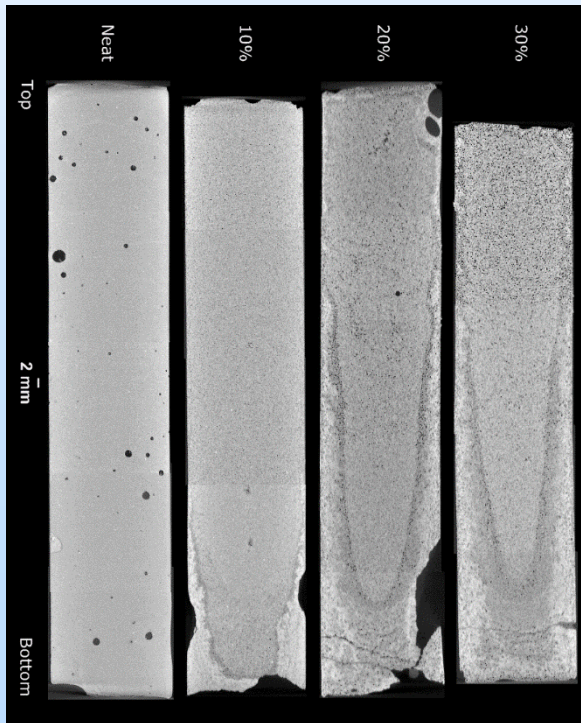
Project Schedule

- Milestone: Identify key mineral and chemical alterations of Class H foamed cement exposed to CO₂ and fluid. 12/2019 - Completed
- Milestone: Determine if fractured Class H foamed cement is capable of self-healing. 3/20/2020 Completed
- Milestone: Determine the effectiveness of CO₂-resistant cements versus traditional Portland cements (Class H). In Progress
- Milestone: Fluid Dynamics – numerical simulation of flow properties and cement behavior (see next slide)





RESULTS



Ordinary Portland Cement (OPC)

Sample A4

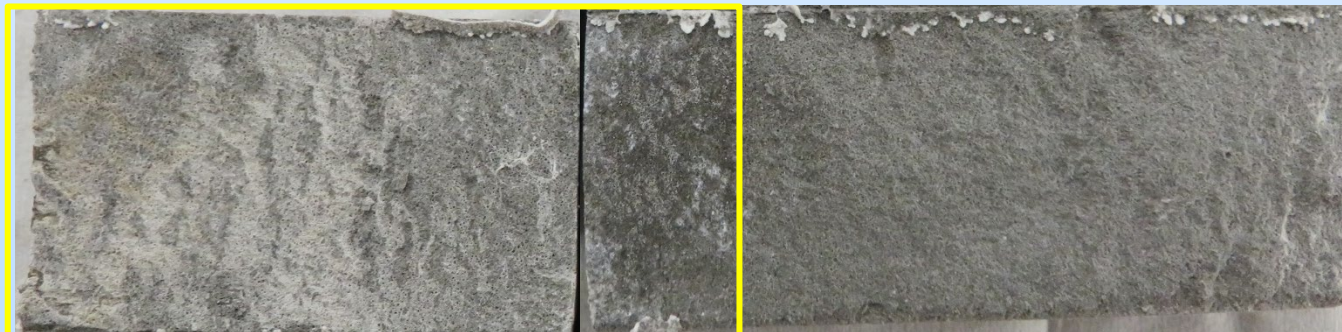


5.6cm,
0.03-0.2mL/min,
60mL fluid flowed,
0.18md to 0.03md

Most reaction happens within 5cm
Flow is channelized

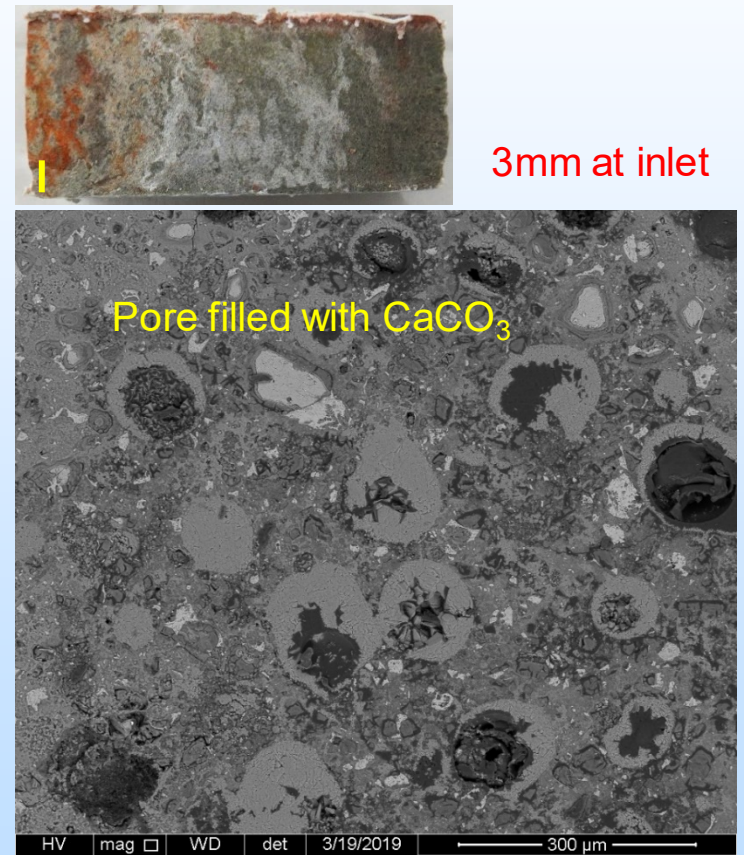
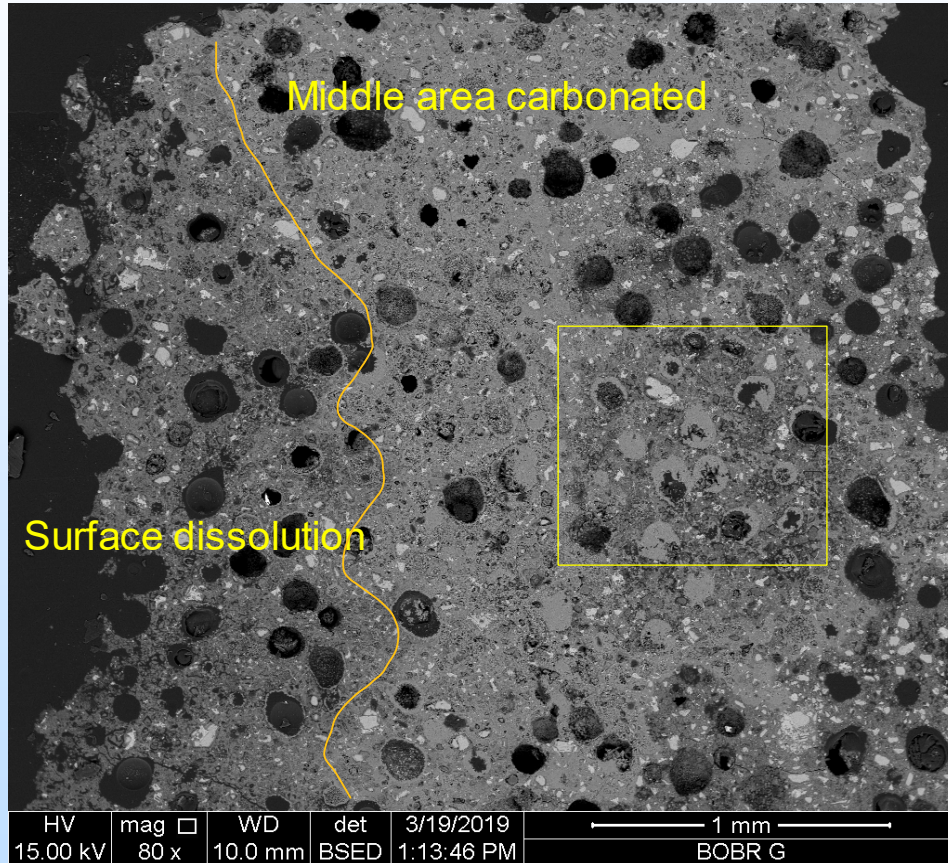
Sample A1

Sample A3



10.3cm,
0.05mL/min,
50mL fluid flowed,
0.17md to 0.10md

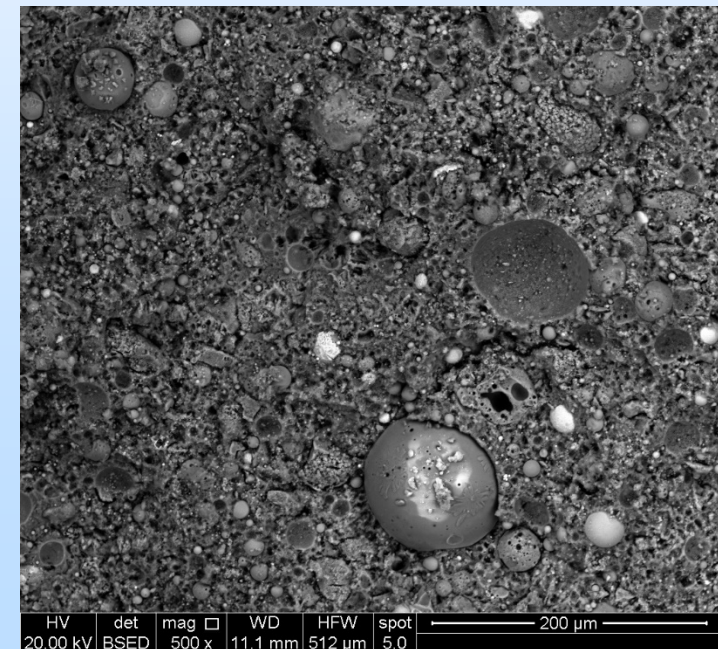
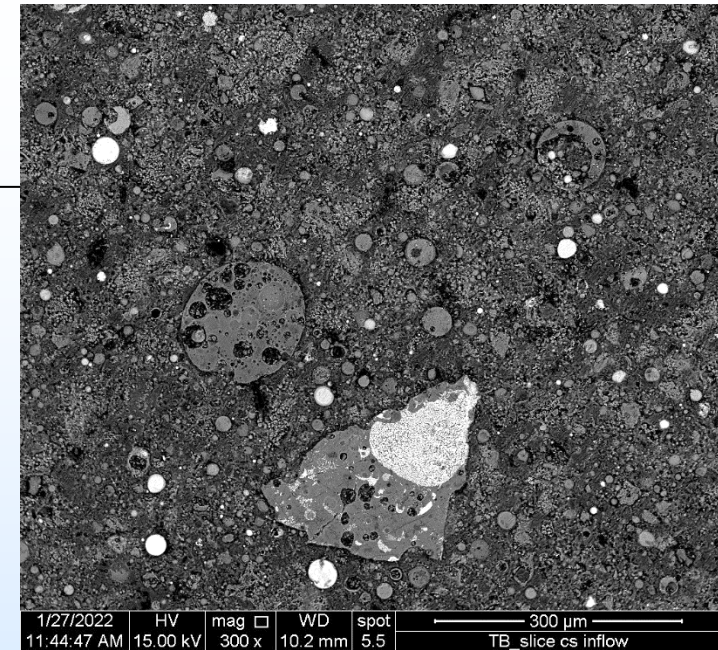
OPC cross section - Example



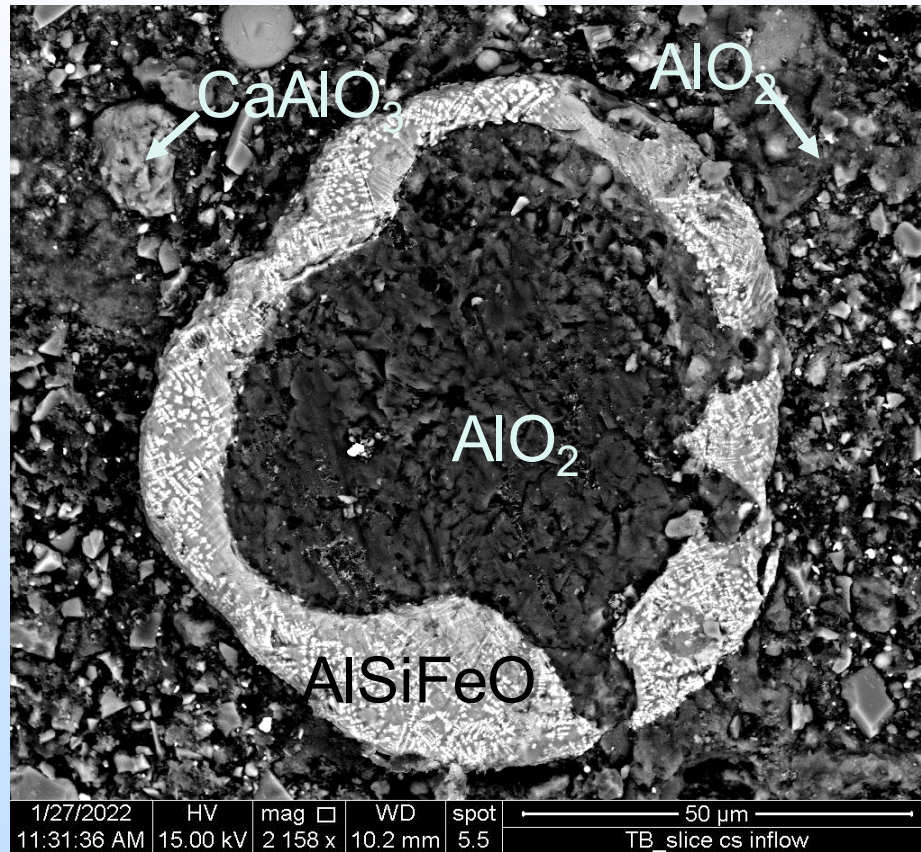
CO₂ Resistant Cement



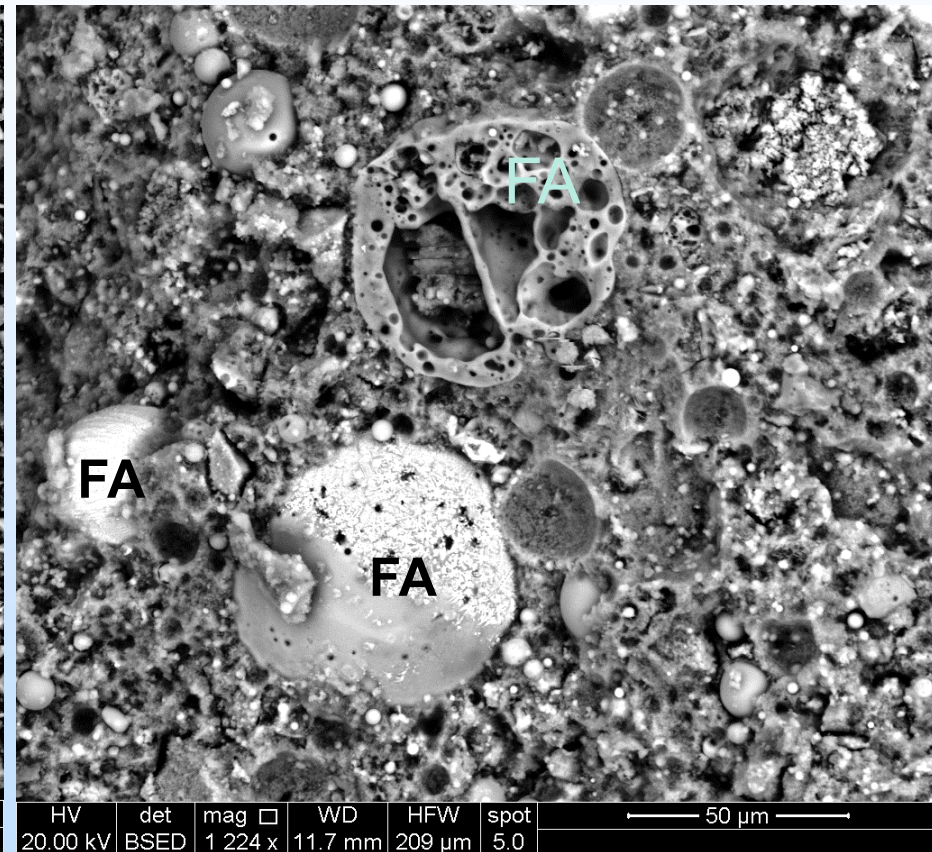
- Calcium aluminate
- Sodium polyphosphate
- Fly ash
- Non-crystalline additives



CO₂ Resistant Cement - unexposed



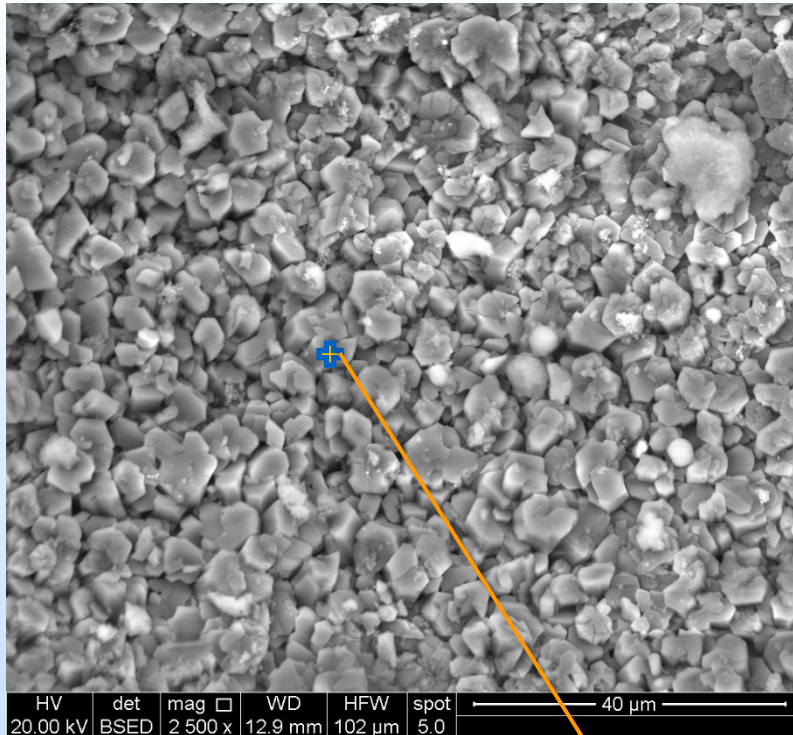
Polished cross-section



Fracture surface/unpolished

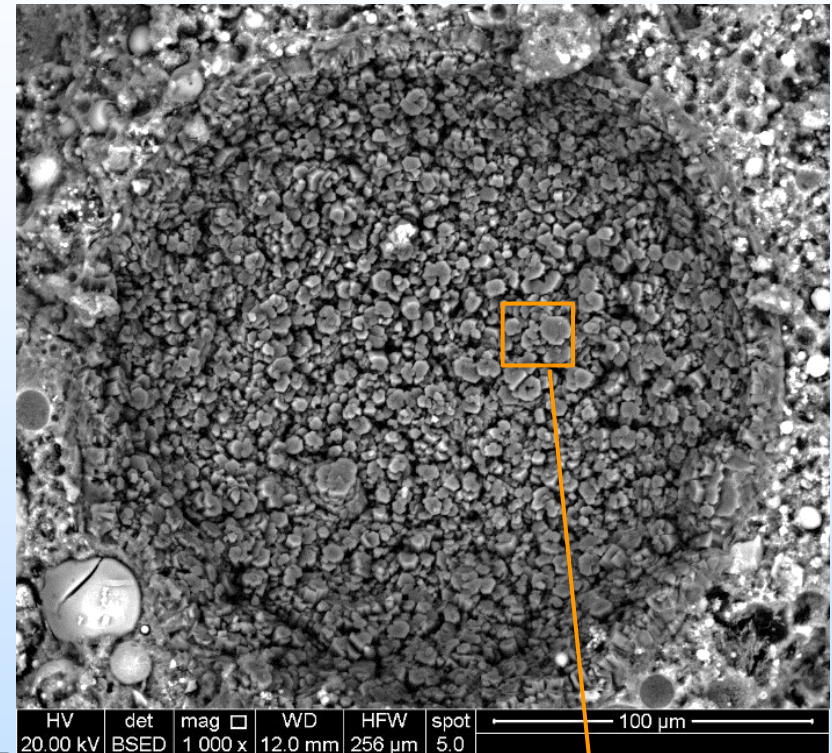
A lot of fly ash (Type F)

CO₂ Resistant Cement - unexposed



Spectrum 9

	Wt%	σ
O	63.0	0.5
Al	34.2	0.3
C	2.8	0.6

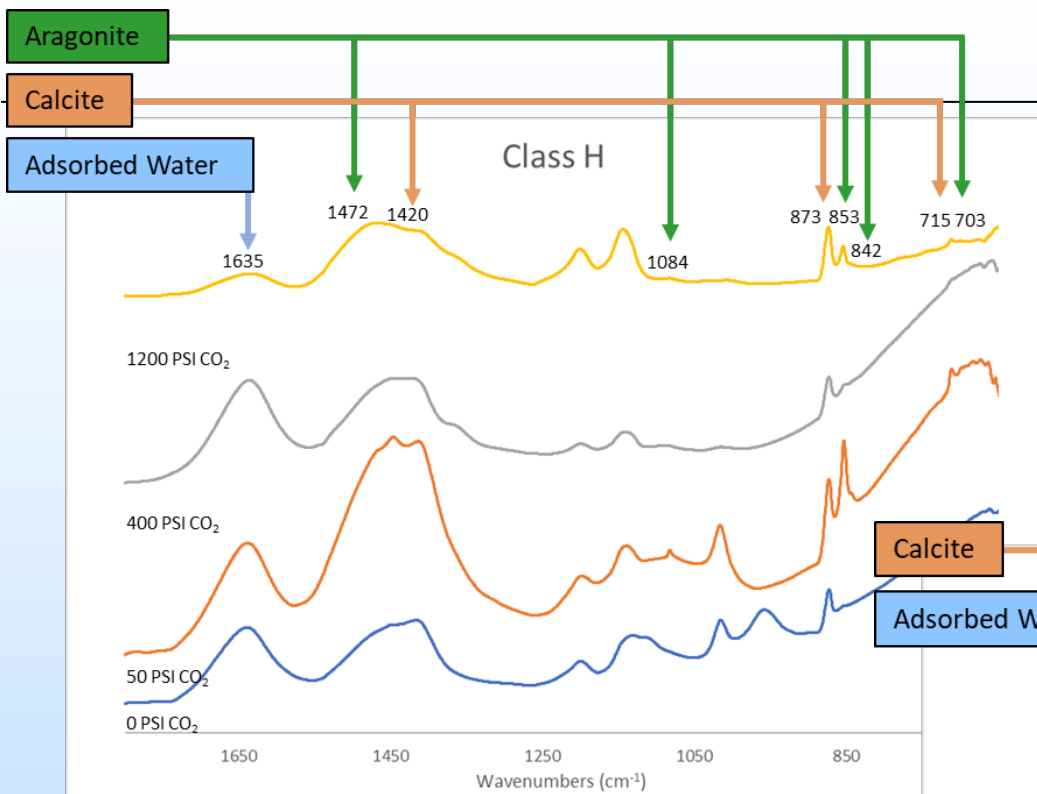


Spectrum 1

	Wt%	σ
O	60.6	0.6
Al	39.4	0.6

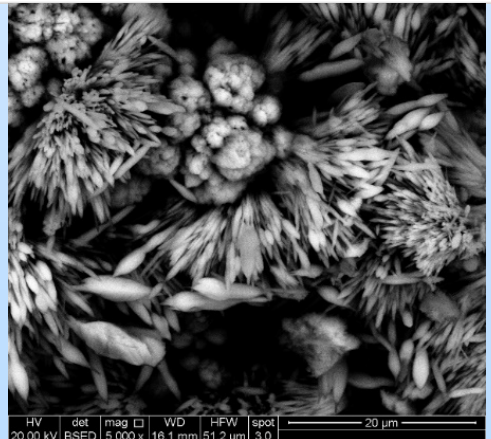
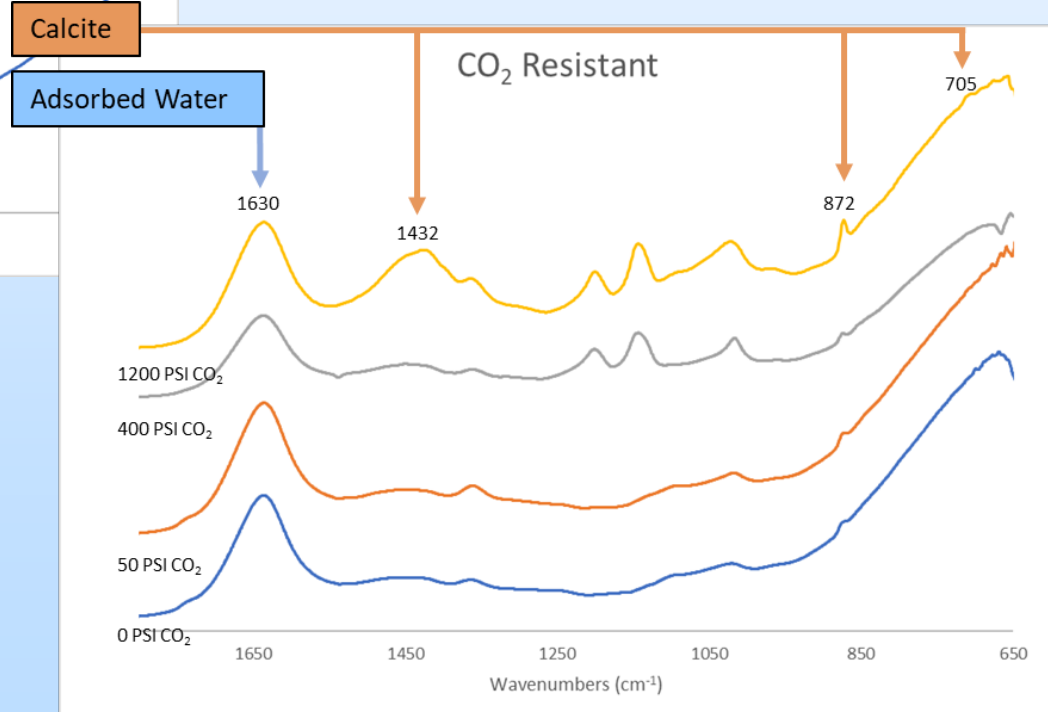
The voids are filled with an Aluminum-rich material – possibly Gibbsite

CO₂ Resistant Cement vs OPC: FT-IR



Class H cement shows pressure-dependent polymorphs of calcium carbonate

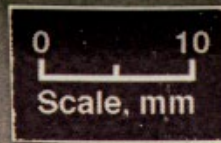
CO₂ resistant cement did not show changes in the calcite polymorph structure



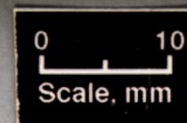
CO₂ Resistant Cement



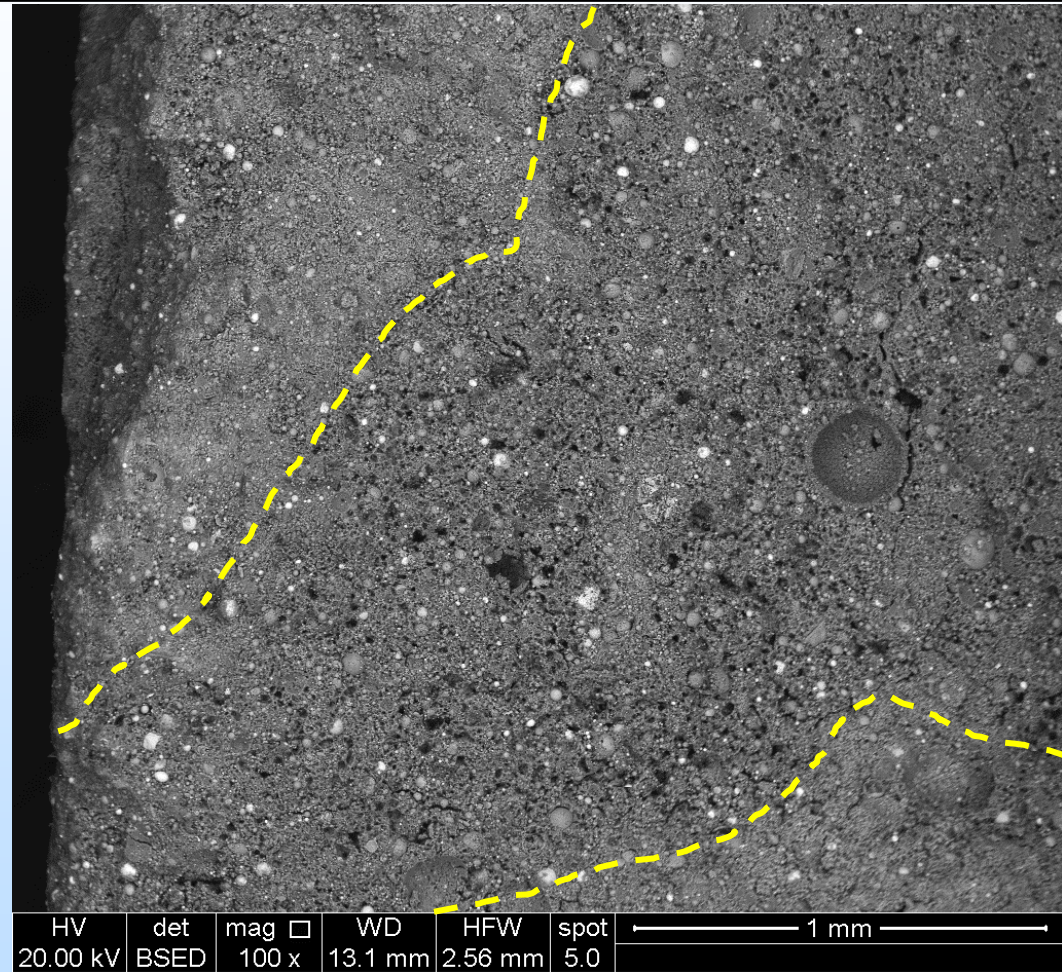
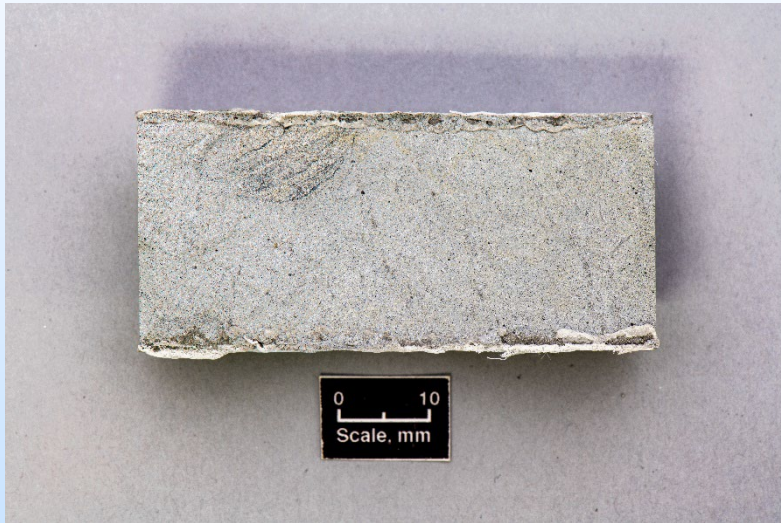
Unexposed



Exposed

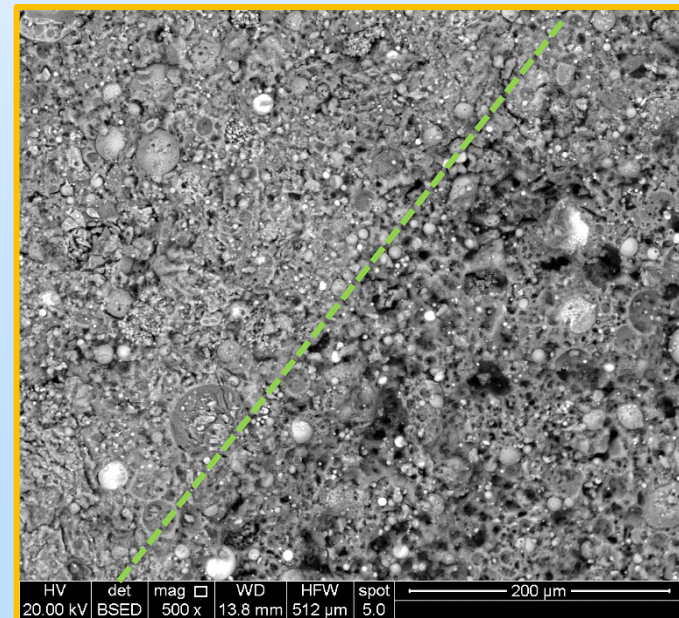
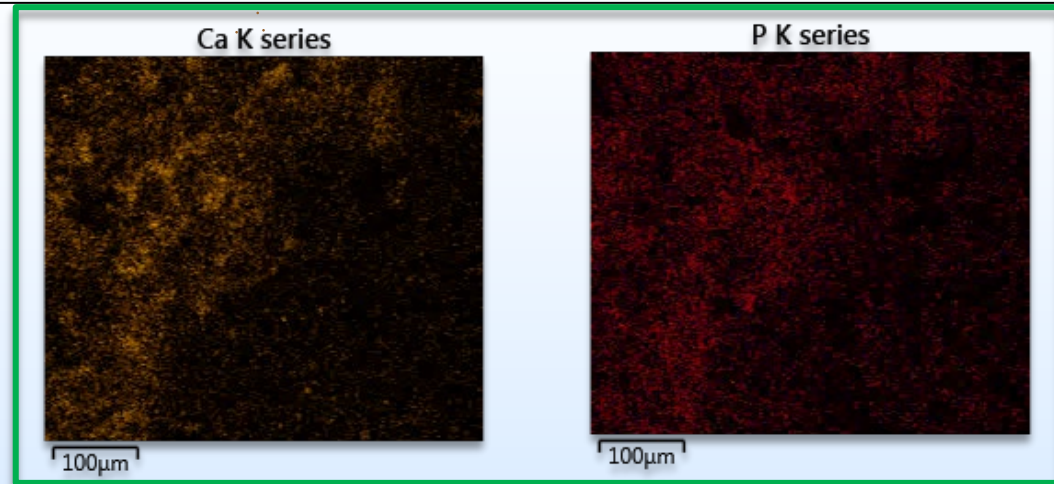
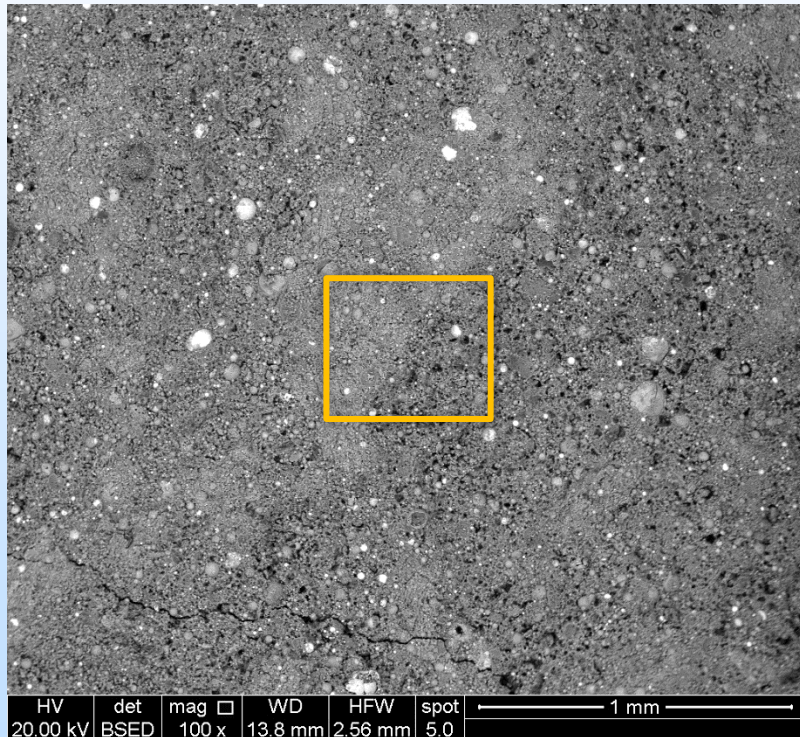


CO₂ Resistant Cement - exposed



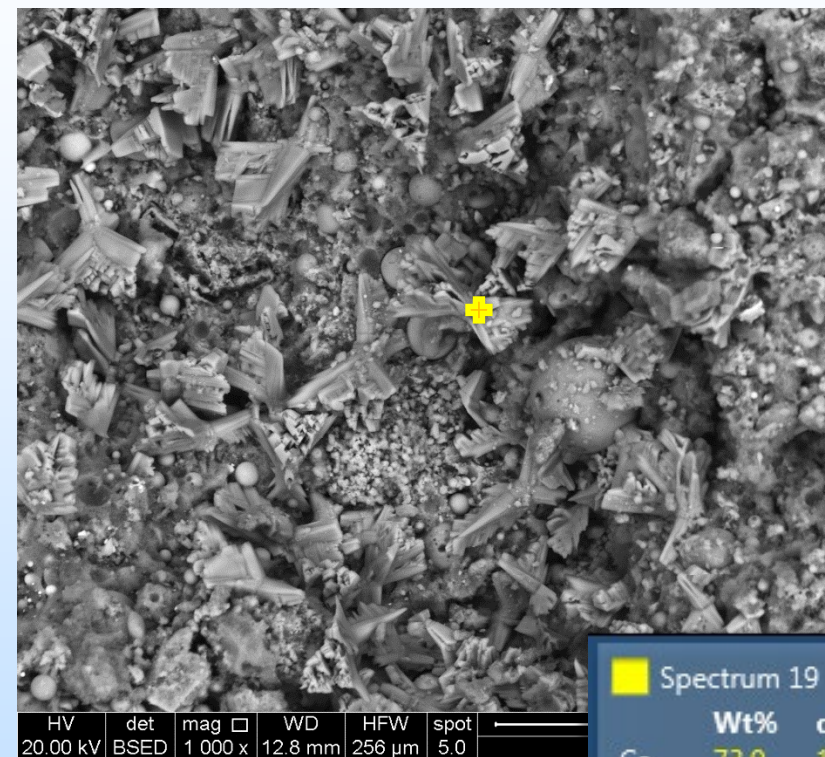
Slight alteration within yellow dotted lines

CO₂ Resistant Cement - exposed



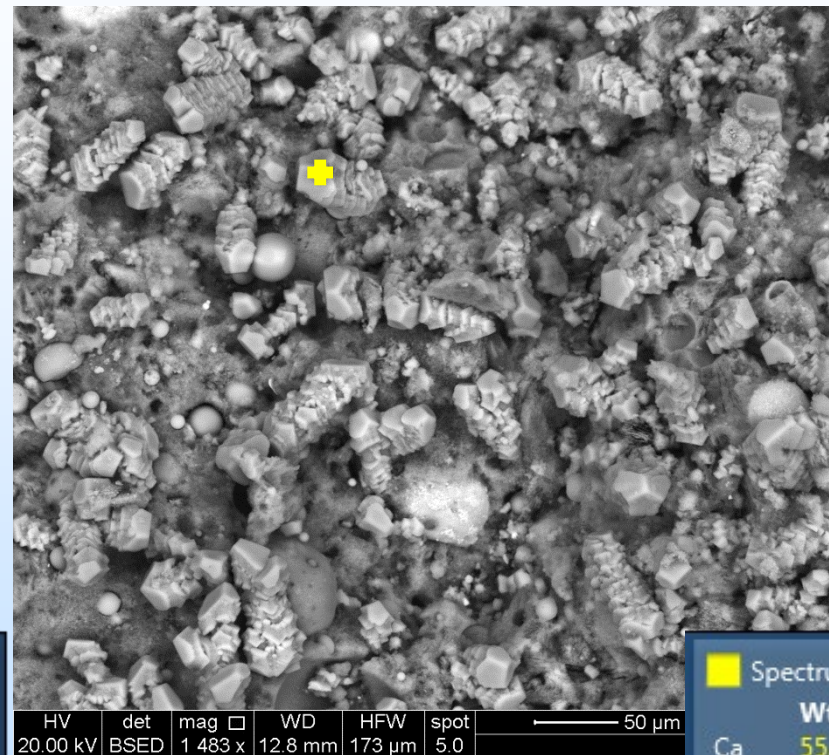
Calcium and Phosphorus show dissolution.

CO₂ Resistant Cement - exposed



■ Spectrum 19

	Wt%	σ
Ca	73.9	1.3
O	21.5	1.2
C	2.7	0.8
S	0.9	0.1
Al	0.6	0.1
Si	0.2	0.1
K	0.1	0.1



■ Spectrum 21

	Wt%	σ
Ca	55.0	1.2
O	37.4	1.2
C	5.8	1.3
S	0.9	0.1
Sr	0.4	0.3
Al	0.3	0.1
Si	0.1	0.1
K	0.1	0.1



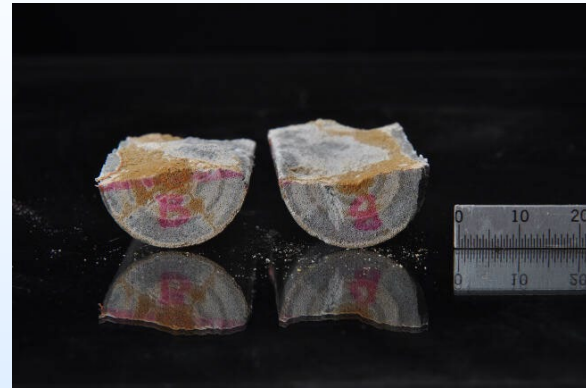
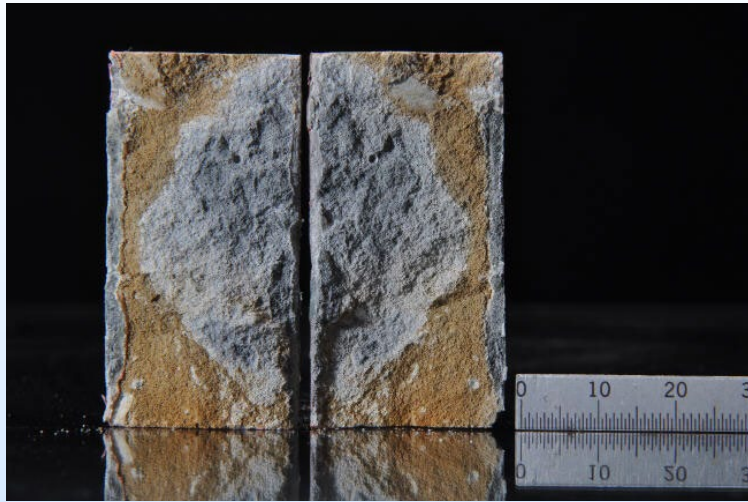
Summary

- Results showed that Portland Cements (both foamed cements and neat unfoamed cement) can self-heal with certain aperture sizes (milestones #1 and #2)
 - The extent of self-healing in foamed cements is comparable to that of neat cement.
 - This observation supports the hypothesis that applying foamed cement in deep water wells will not increase the risks of CO₂ leakage.
- CO₂-resistant cements do not show significant chemical alteration
 - We don't see the traditional dissolution and precipitation that we see in Portland Cements
 - Good for intact cement but might not be good for fractured cement where the chemical alteration is needed to ensure self-healing
- Waiting for flow data and mechanical measurements

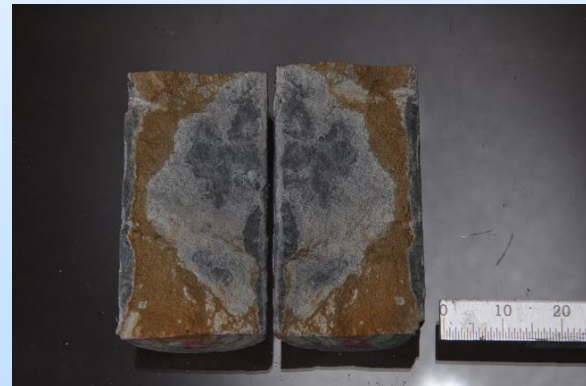
Questions?

Additional Information

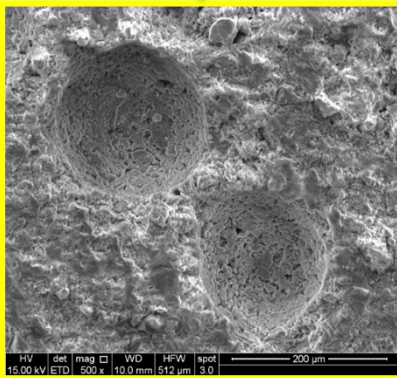
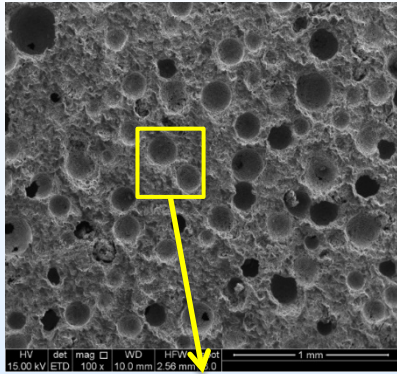
Ordinary Portland Cement



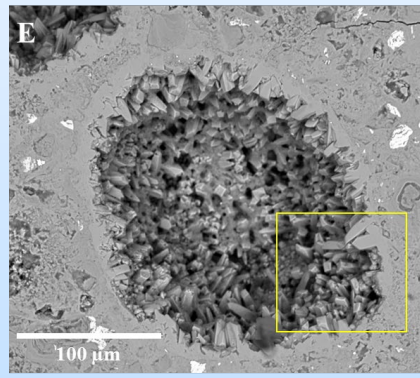
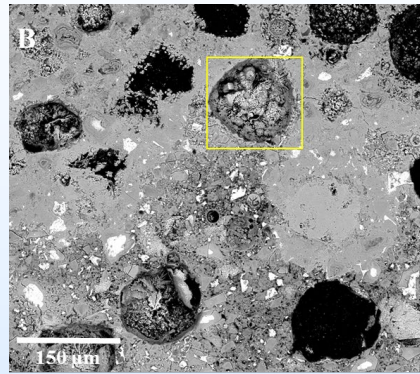
Photos of foamed cement sample (20% foam quality) exposed to variable flow of saturated CO₂ in the medical CT scanner



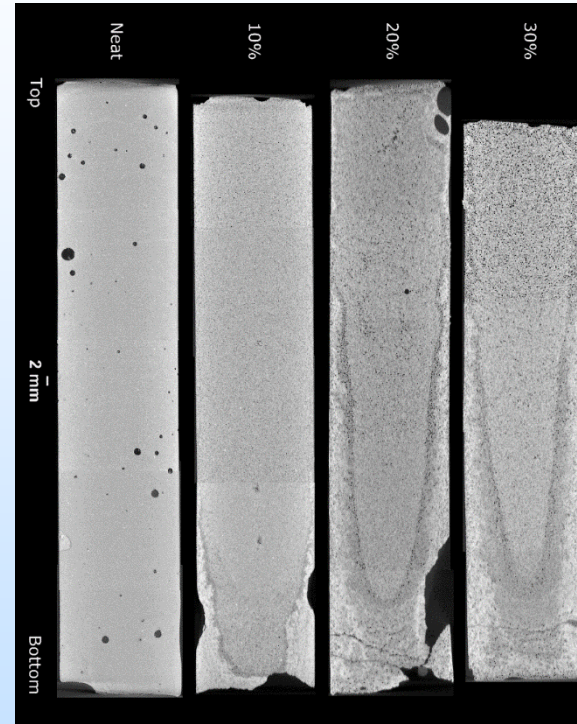
Previous Research



Unexposed foamed cement



Foamed cement exposed to SCCO_2 under static conditions (56 days)



Stitched CT Core montage on the XZ direction for neat, 10%, 20% and 30% cores exposed for 6 months. Stitched from approximately 9,000 2D images associated with the full scan of the core

The bubbles in the alteration zone are filled with calcium carbonate crystals

Illustrates how carbonation alters pore space by precipitation