X-ray Microscopy of Green Cements

Paulo J.M. Monteiro
University of California
Berkeley

Collaborators

- Prof. Rudy Wenk
- Prof. David Attwood
- Dr. Robert P. Winarski
- Dr. Steve Wang
- Dr. Anne E Sakdinawat
- Dr. Gerd Schneider
- Dr. Peter Guttmann
- Prof. Pierre Levitz
- Dr. Juyoung Ha
- Rosie Chae
- Seyoon Yoon
- Cagla Meral
- Pierre Itty
- Ju-hyuk Moon

Dr. Tony Warwick
Dr. Peter Fischer
Dr. Tolek Tyliszczak
Dr. David Kilcoyne

World demand/year

- 16 billion ton of concrete
- 1.5 billion ton of water
- 12.4 billion ton of aggregate
- 2.1 billion ton of cement

Consequences

2.1 billion ton of cement

Problem!

- **Generates 2.1 billion ton of CO_2**
- **Responsible for 5-7% CO_2 production in the world**

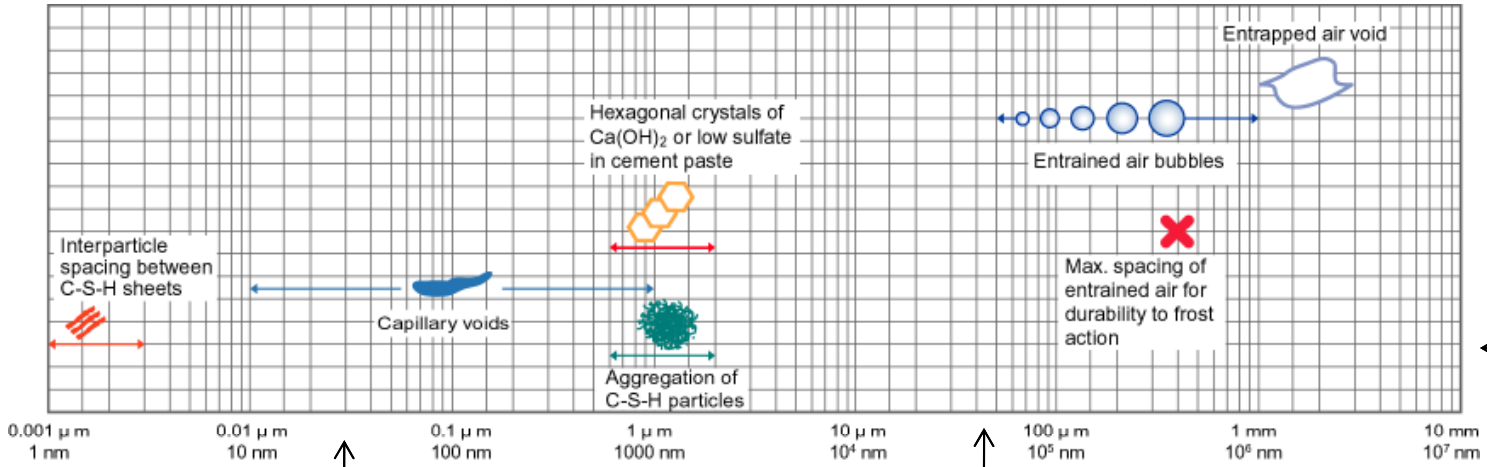
Challenges

Challenge II: Long-term durability

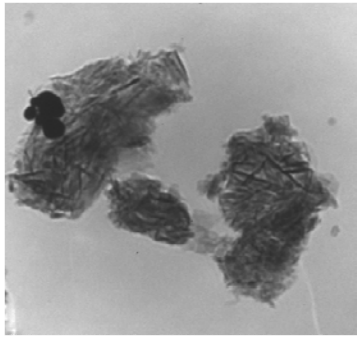
Civil Infrastructure quickly deteriorating

March 17, 2008, I-95 in Philadelphia

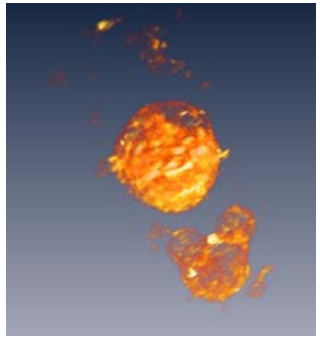
Multi-scale engineering



85 m (high)



Roman Concrete



Ice in air voids

Maua dam

Fundamental Question:

Exciting opportunities with disruptive impact have been identified. Why somebody has not taken advantage of them?

BECAUSE IT IS <u>REALLY</u> HARD:

120 years of development for portland cement.

It requires science, technology, and resources.

The 8-Million Dollar Question:

Can we develop a greener cement and create a sustainable infrastructure?

Integrated research

Advanced Light Source

Soft x-rays microscopy, Small Angle Scattering, High-Pressure, Microdiffraction, Microtomography, Ambient XPS.

Advanced Photon Source

Total scattering methods (pdf), Nanotomography, Small Angle Scattering.

BESSY

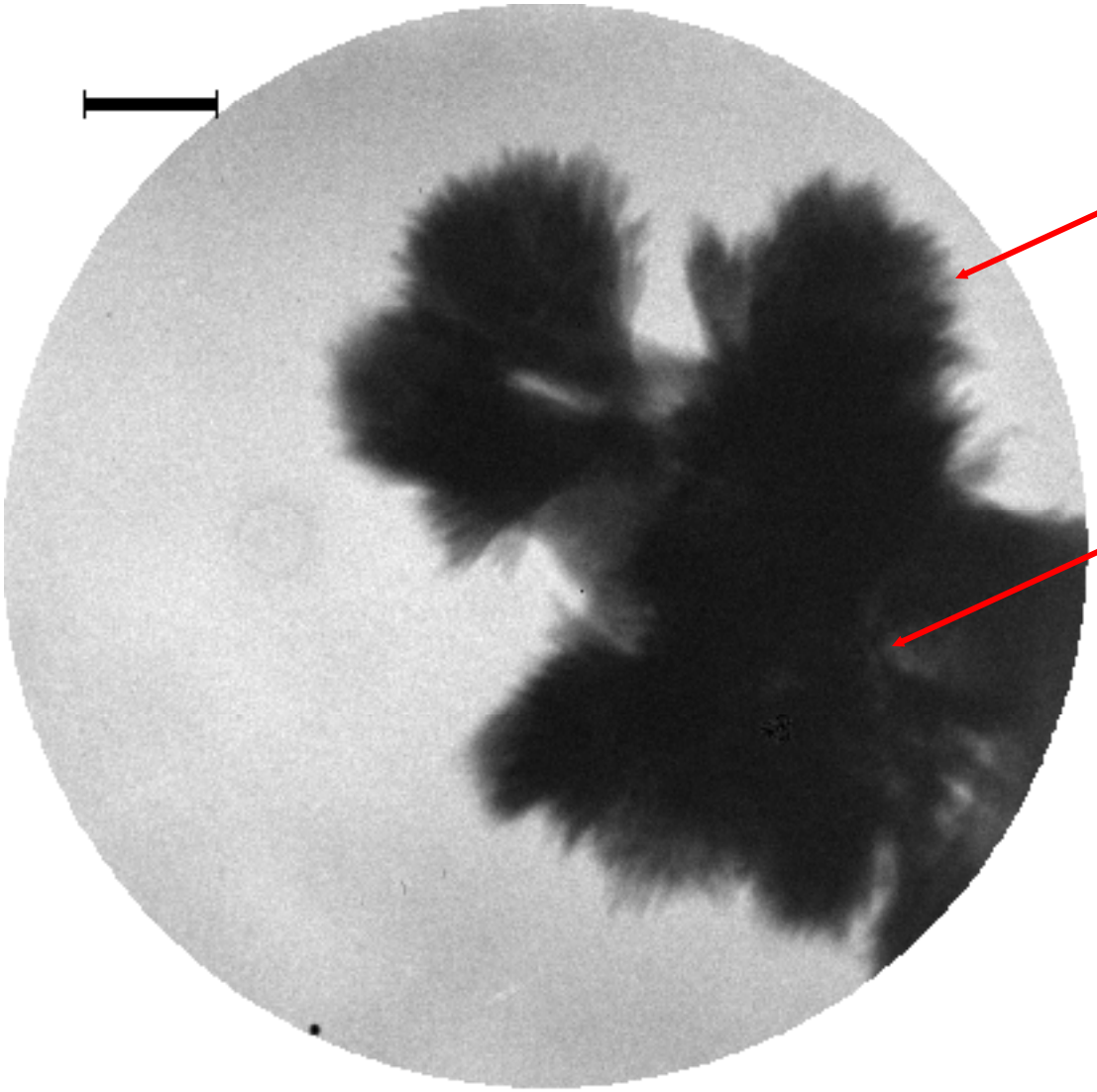
Nanotomography

… more at Los Alamos

Possibilities with soft x-ray microscopy

1. **Soft x-ray transmission microscopy allows the observation of wet samples under atmospheric pressure,**
2. **It is possible to image the cement grains as soon as 6 minutes after mixing.**
3. **Nucleation and growth of hydrates in solution can be observed over time.**

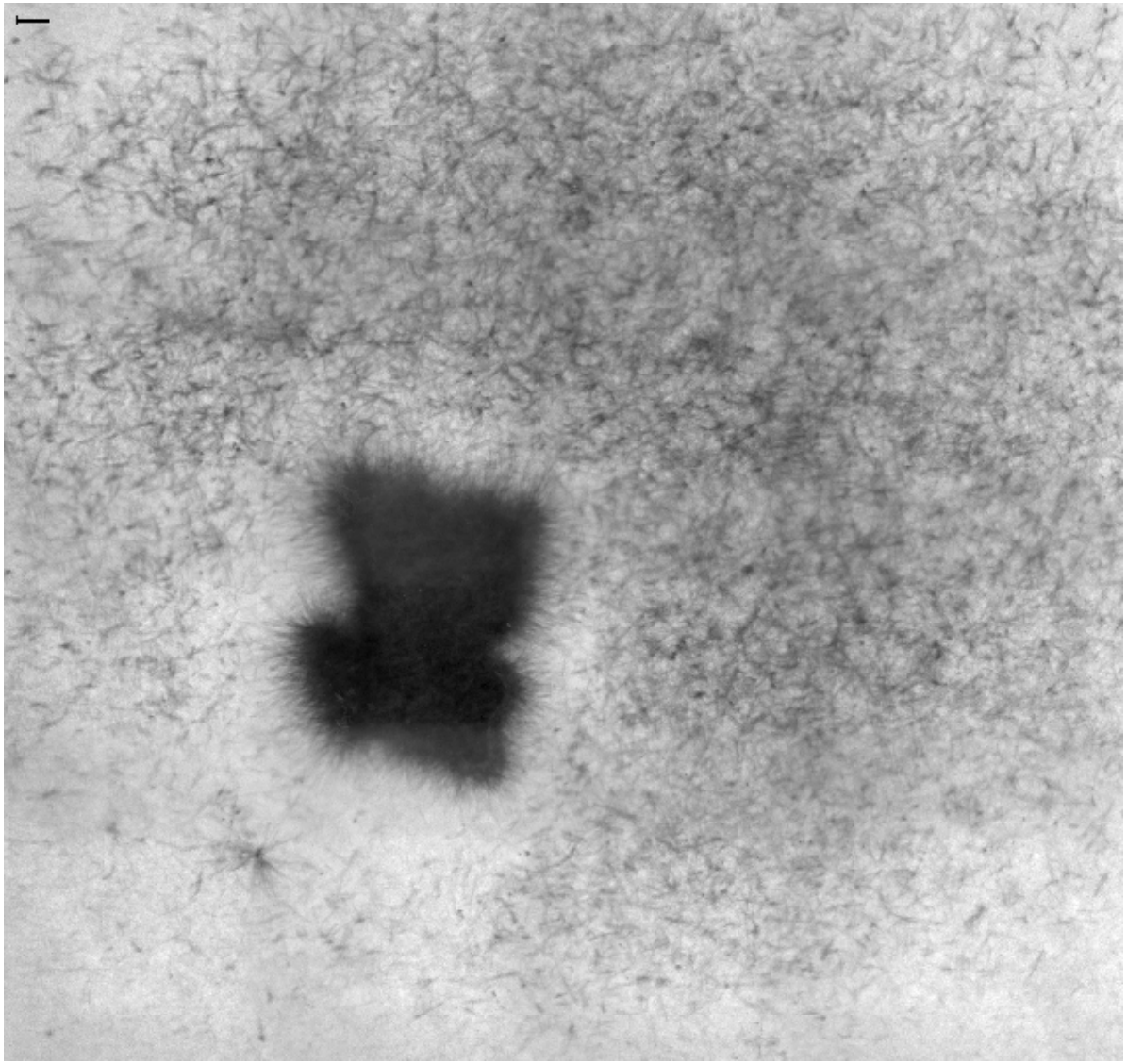
Early hydrates forming during the pre-induction period



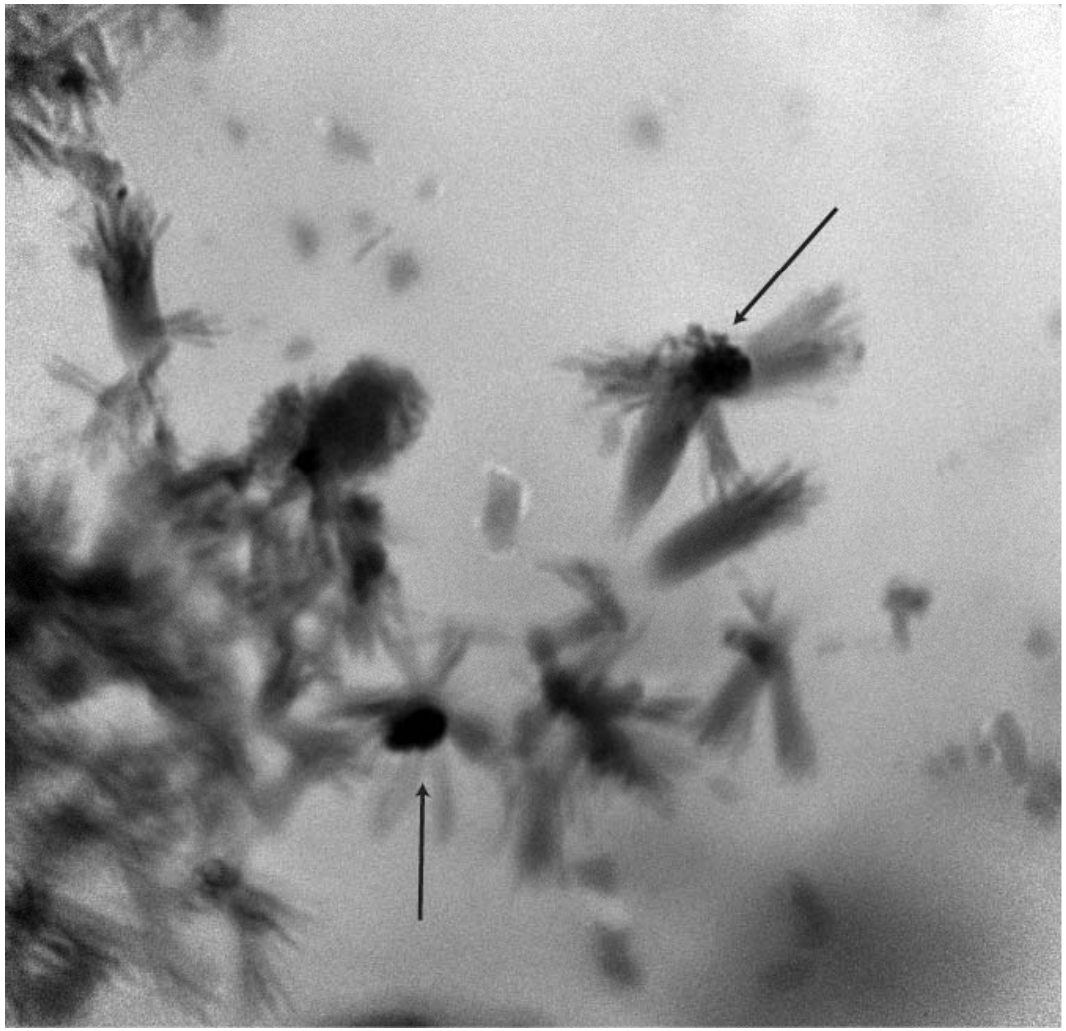
C3S hydrated for 34 min. in saturated lime and calcium sulfate at w/c=5, 1s exposure time, 516eV, scale bar 1μm.

In-situ Massive precipitation

8h 36min.



Why nanotomography?



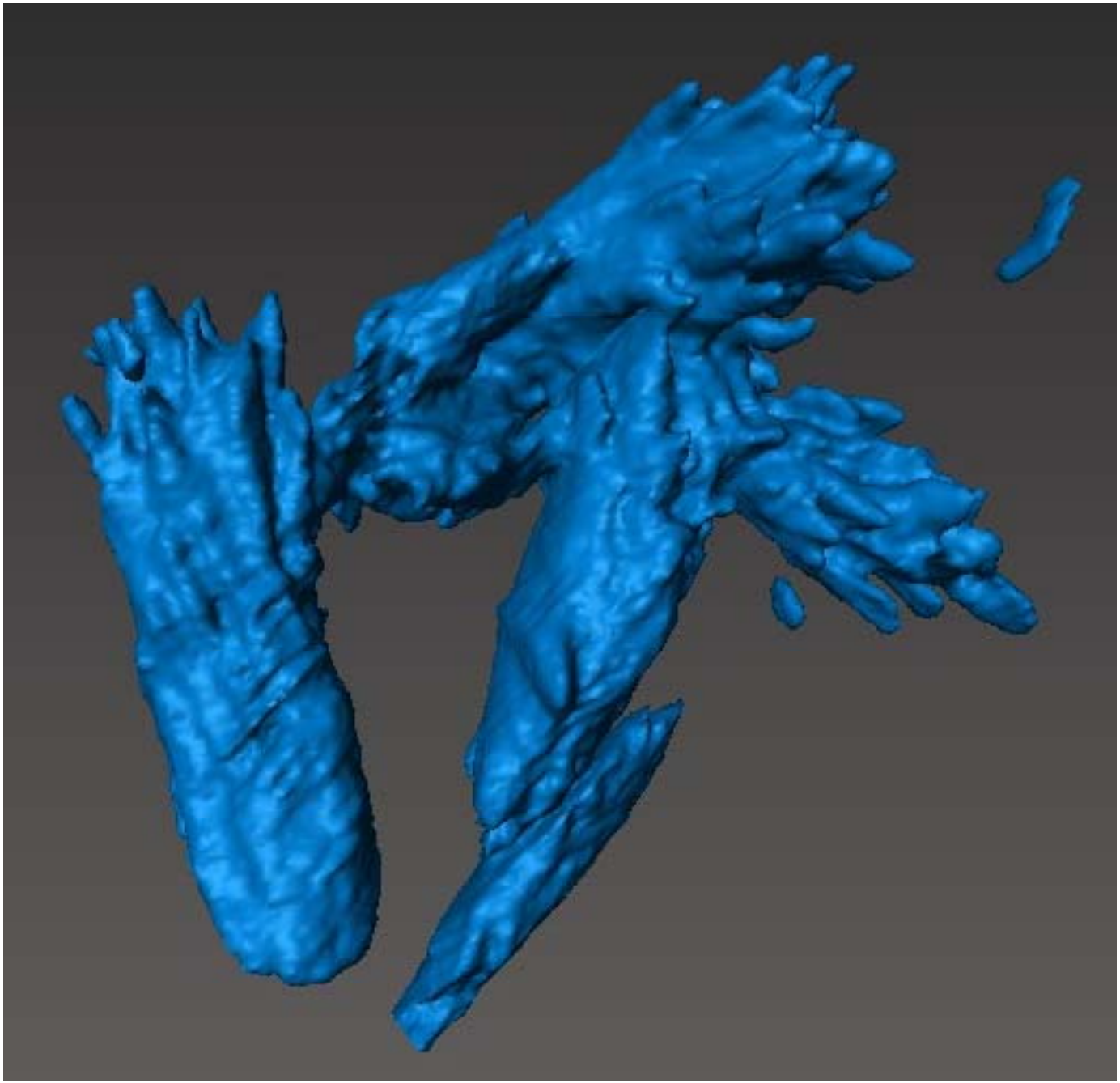
Work at BESSY

- This transmission image seems to show that the "sheet of wheat" (or "stars") have a core which acts as a nucleation point (see arrows)

Work with Brisard, Levitz and Chae

Wrong assumption!

Work with Brisard, Levitz and Chae

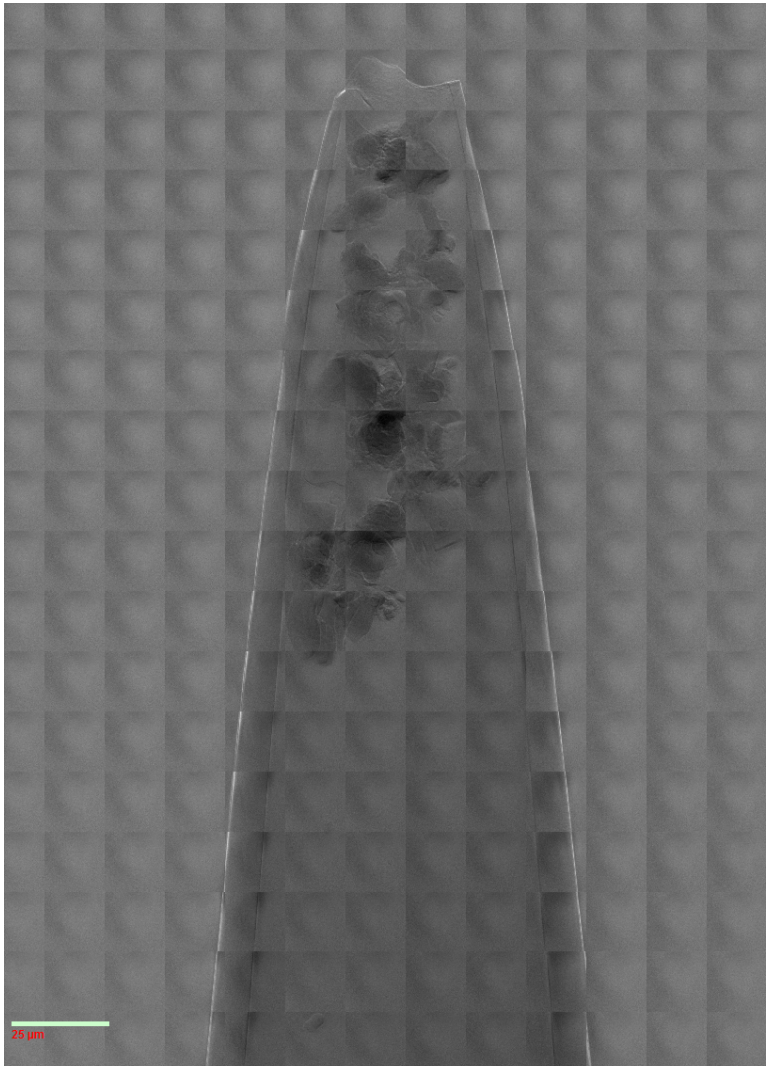


Work with Brisard, Levitz and Chae

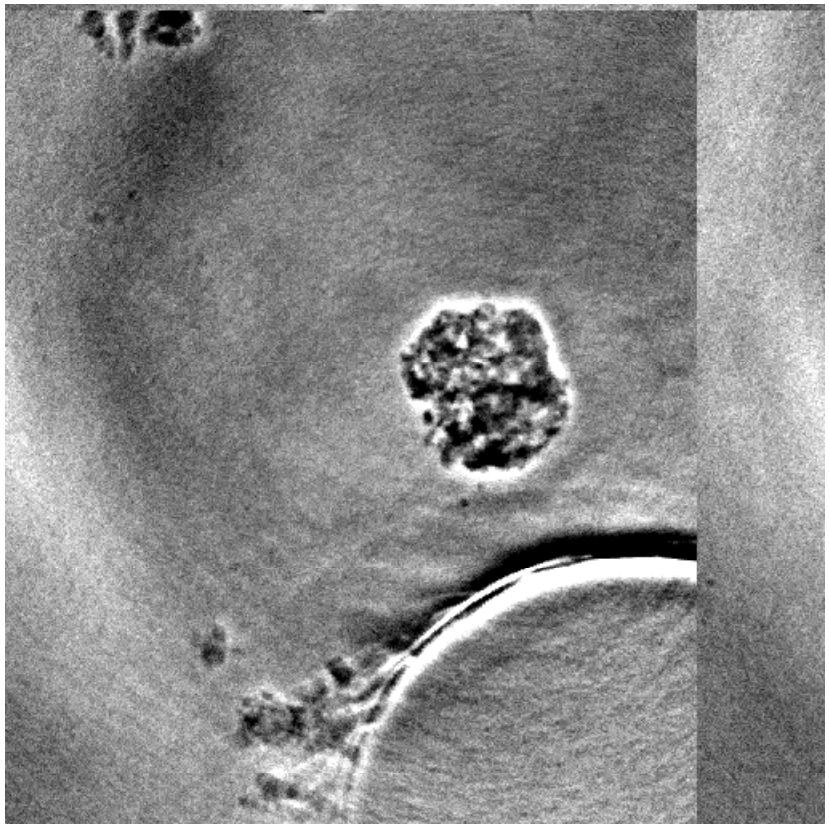
Nanotomography at ALS

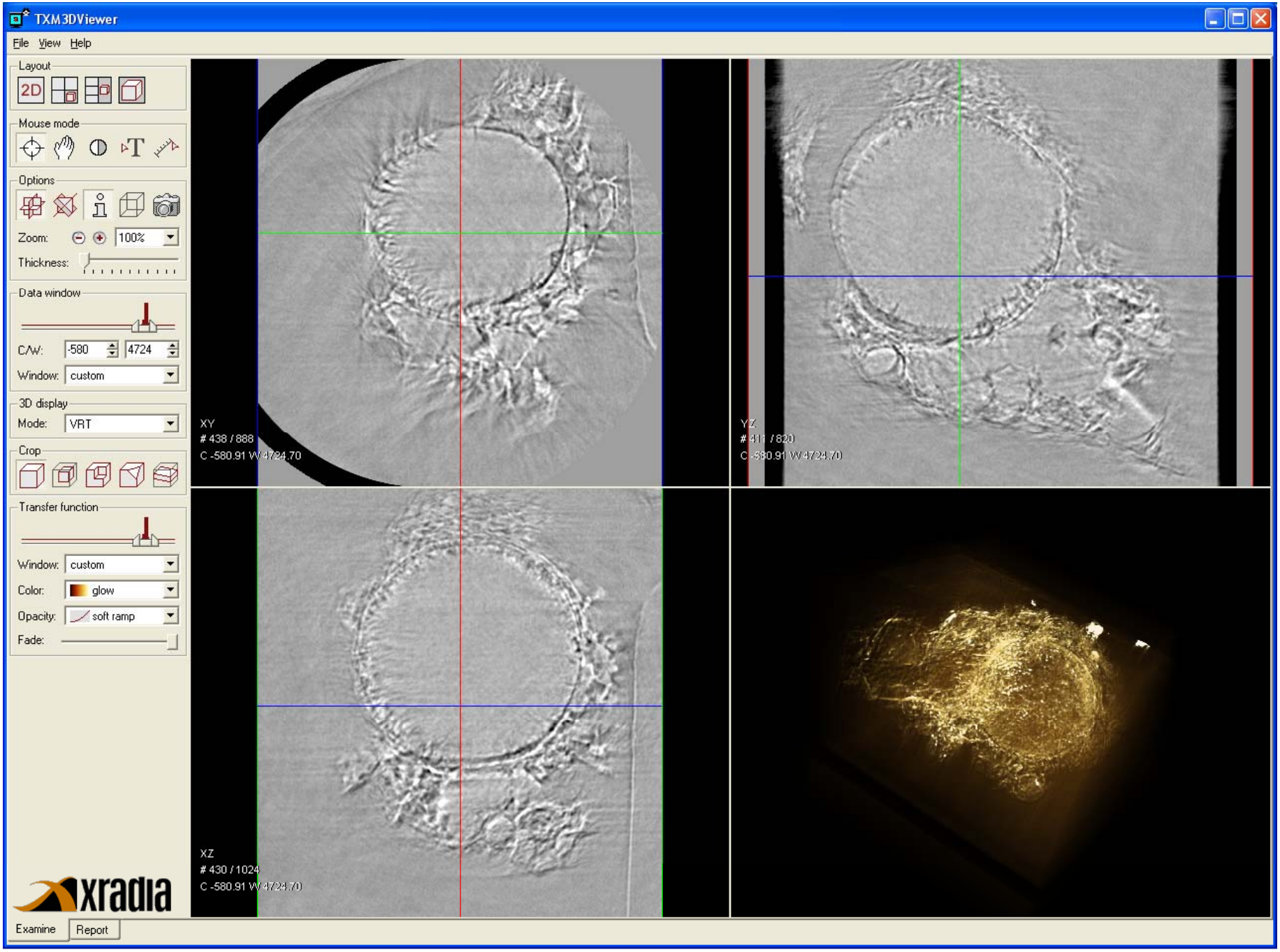
- Courtesy of Carolyn Larabell, Mark Le Gros, Dula Parkinson

Tomography at APS Nanoprobe



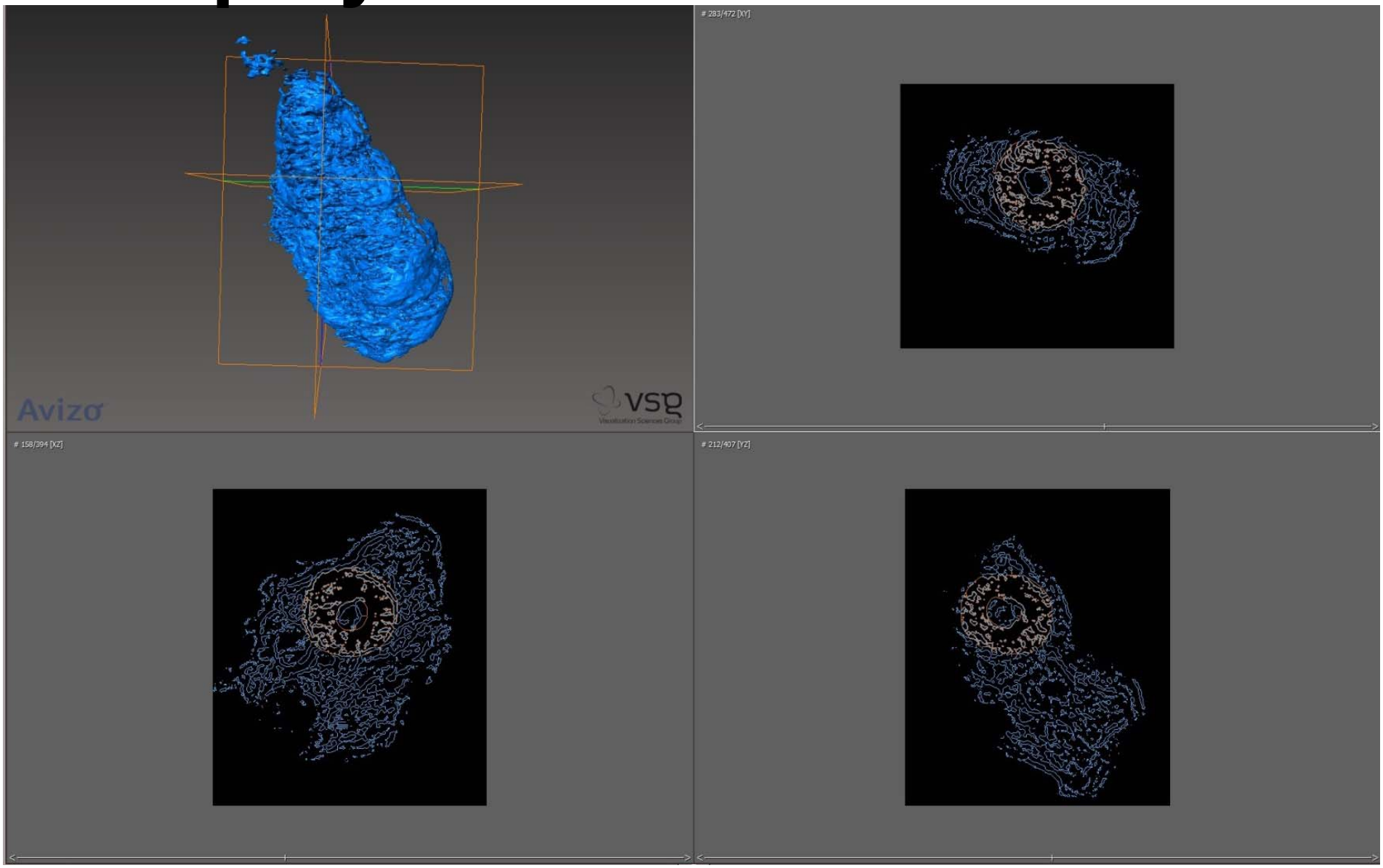
Cement paste inserted into extruded glass capillary tube



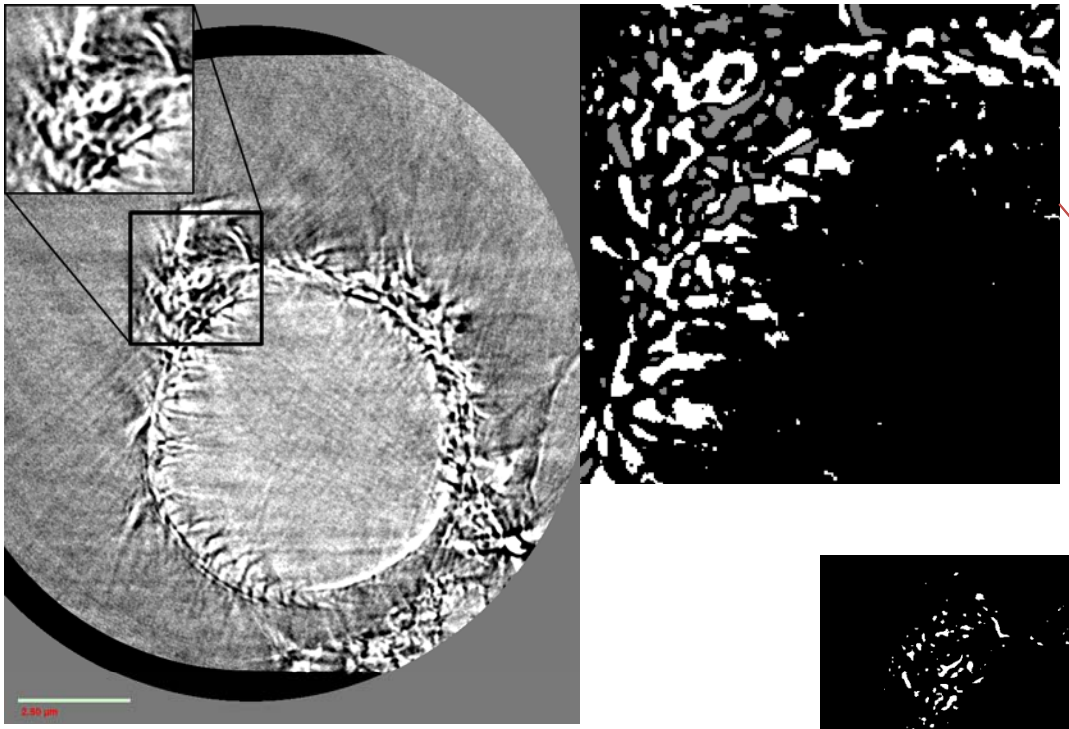


3/11/2011 Scan #27 Geop NaOH high T ambient

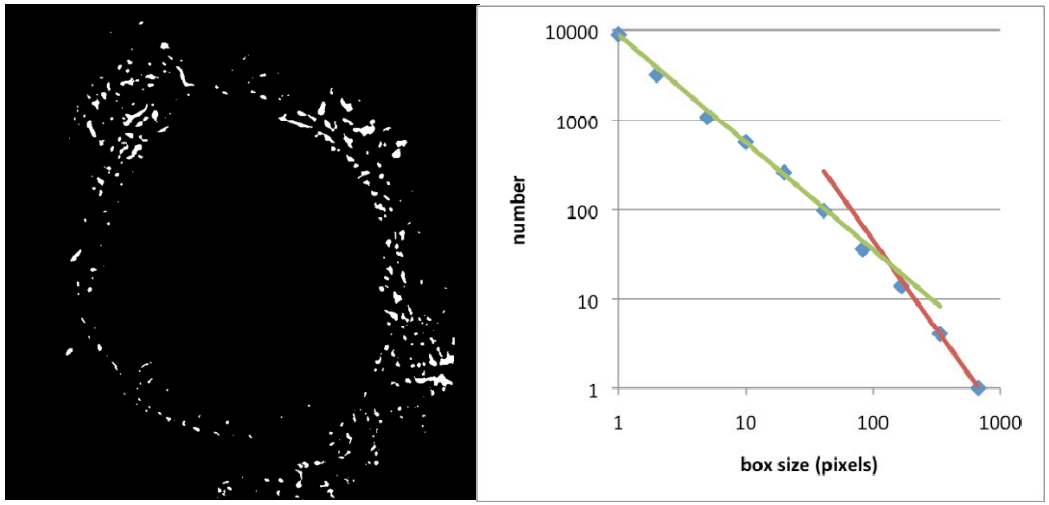
Geopolymers



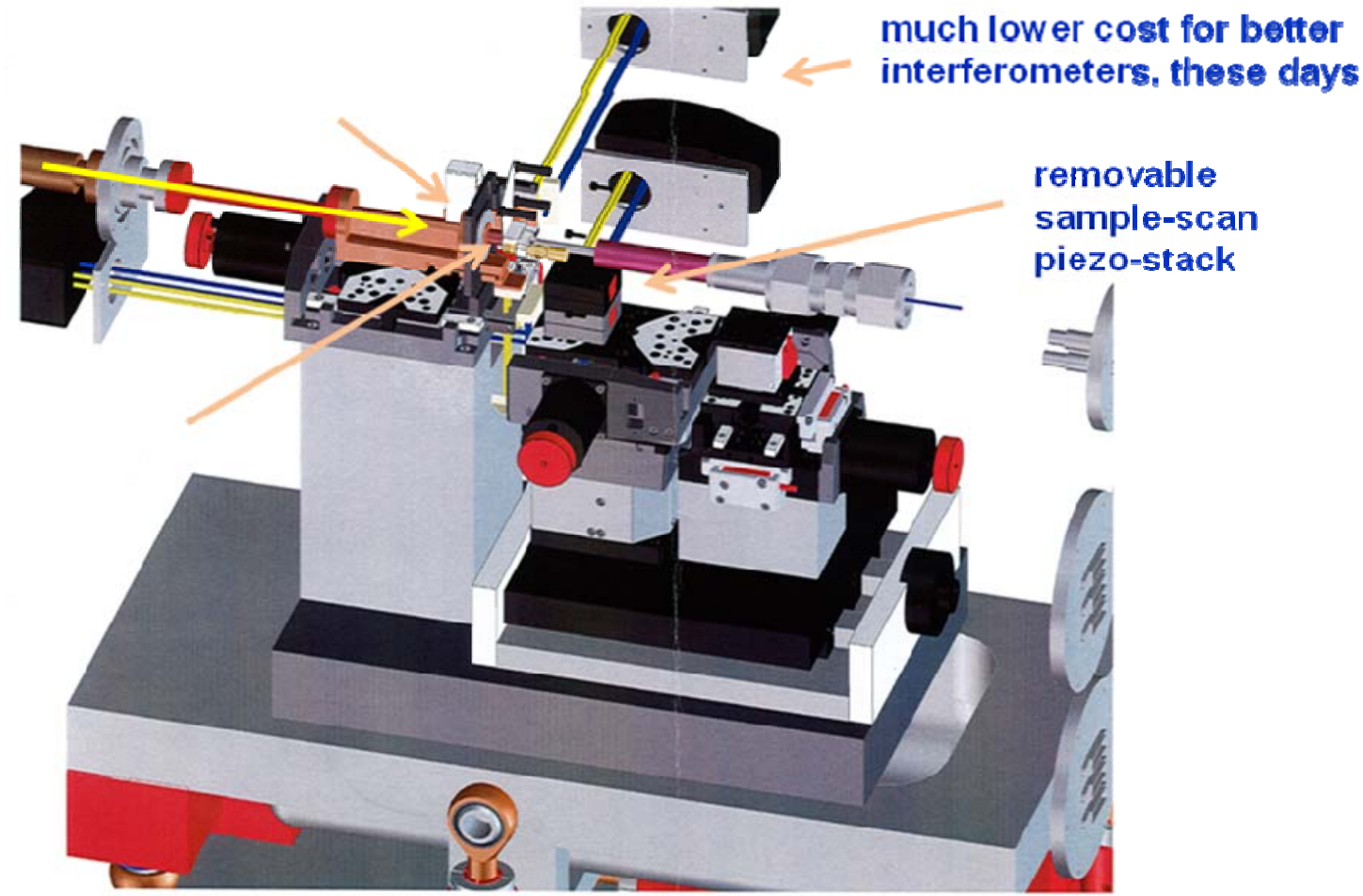
Study of dissolution



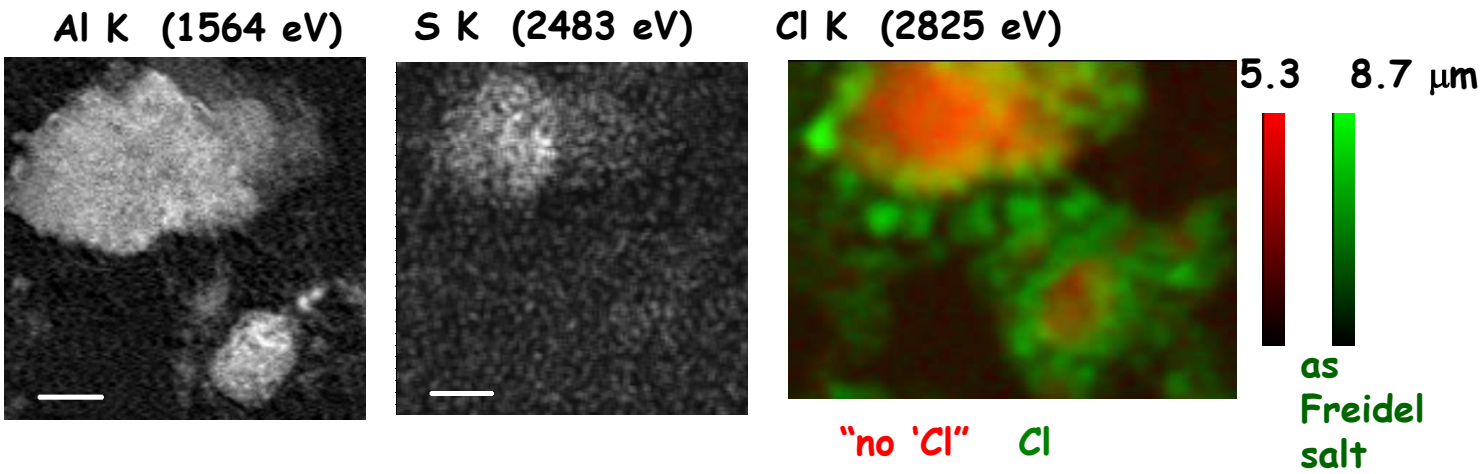
Fractal dimension



New Microscope Design



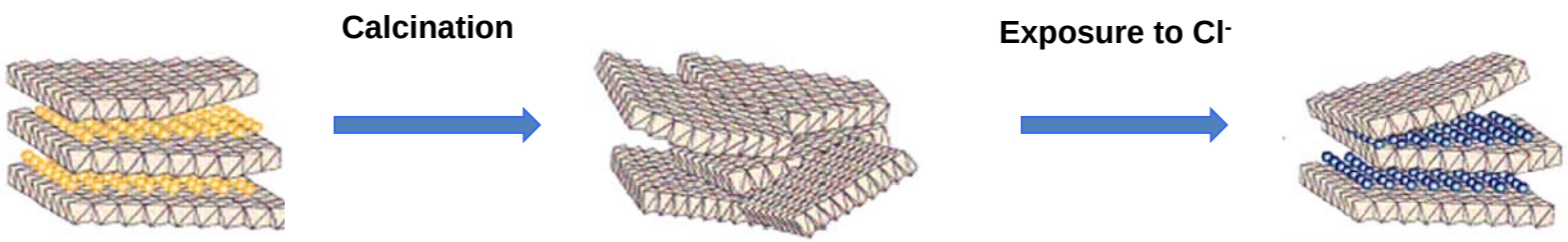
Developed by Tolek Tyliszczak , David Kilcoyne and Tony Warwick.



Monosulfoaluminate immersed in 5 M NaCL washed, deposited on Si3N$ window

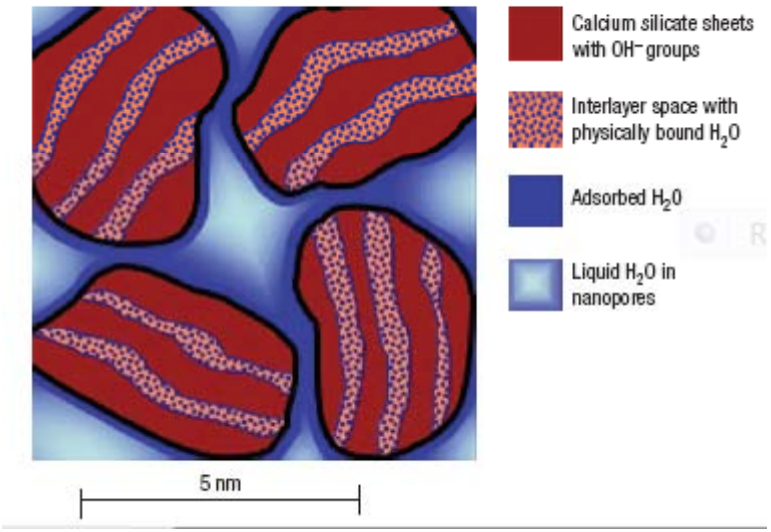
Work with Seyoon Yoon, Rosie Chae, Adam Hitchcock and David Kilcoyne

Cl^- Scavenging Mechanism

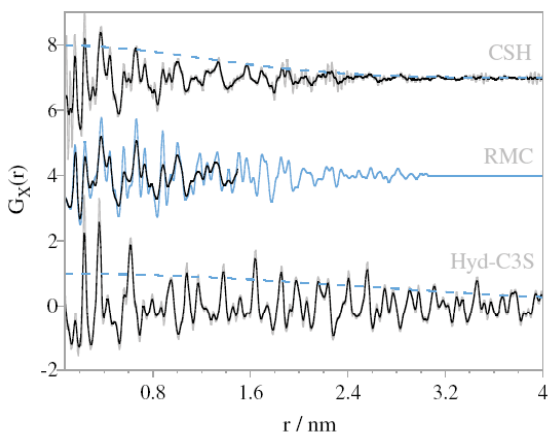


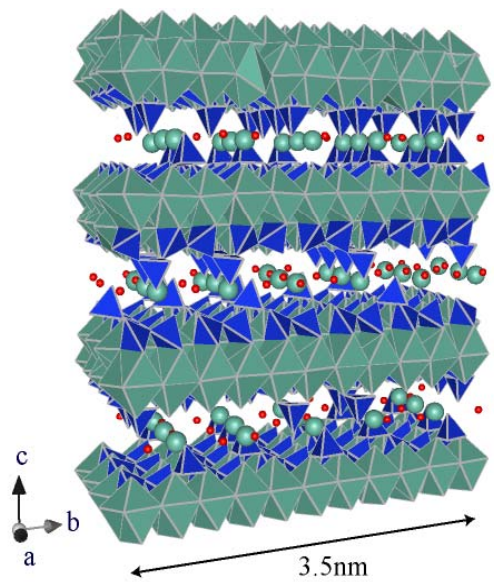
Layered Double Hydroxide, LDH **Layered Complex Oxide, LCO** **Reconstructed LDH**

Need for improved resolution: The importance of COSMIC



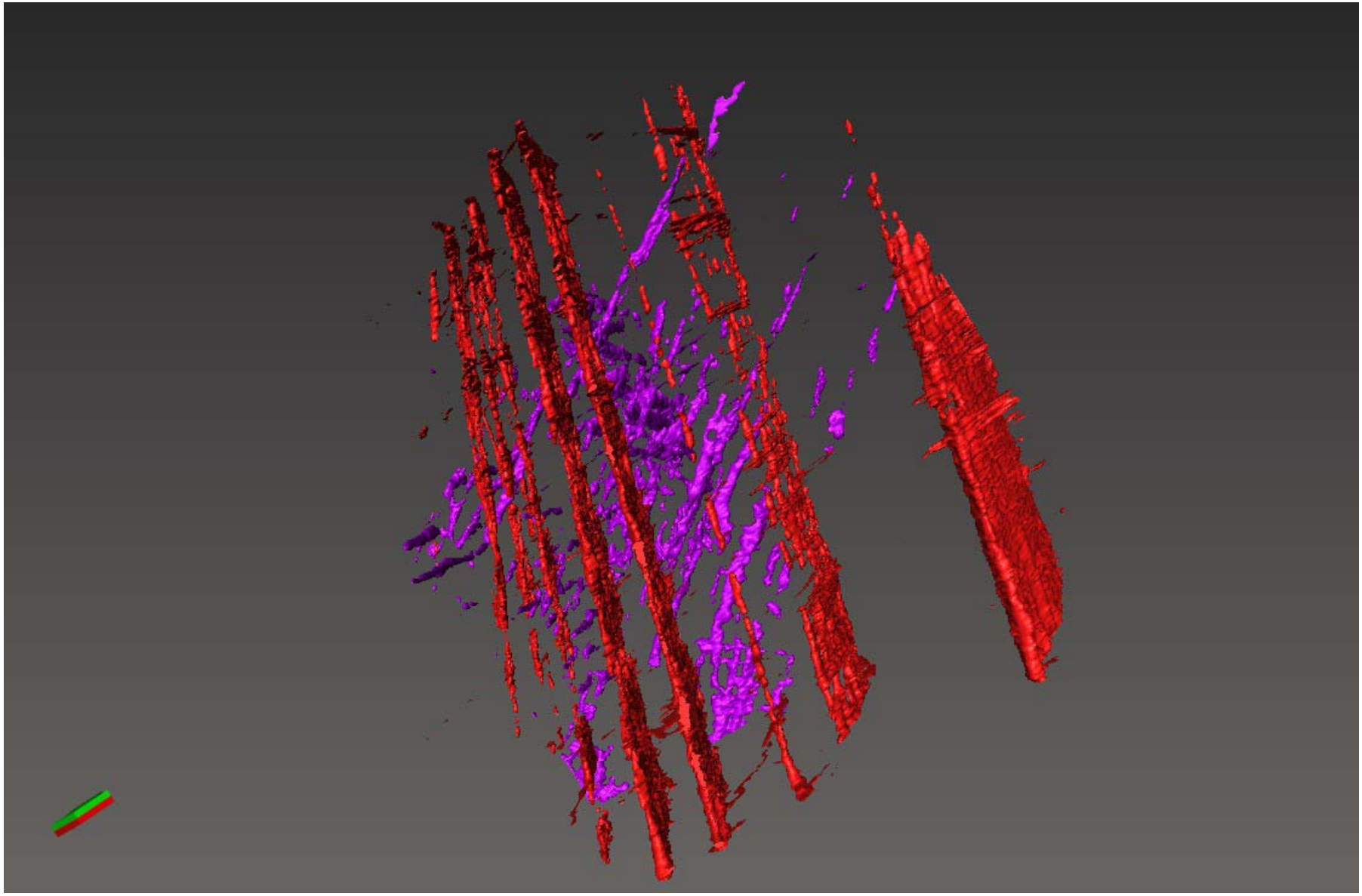
Allen et al.
Nature Materials, 2007



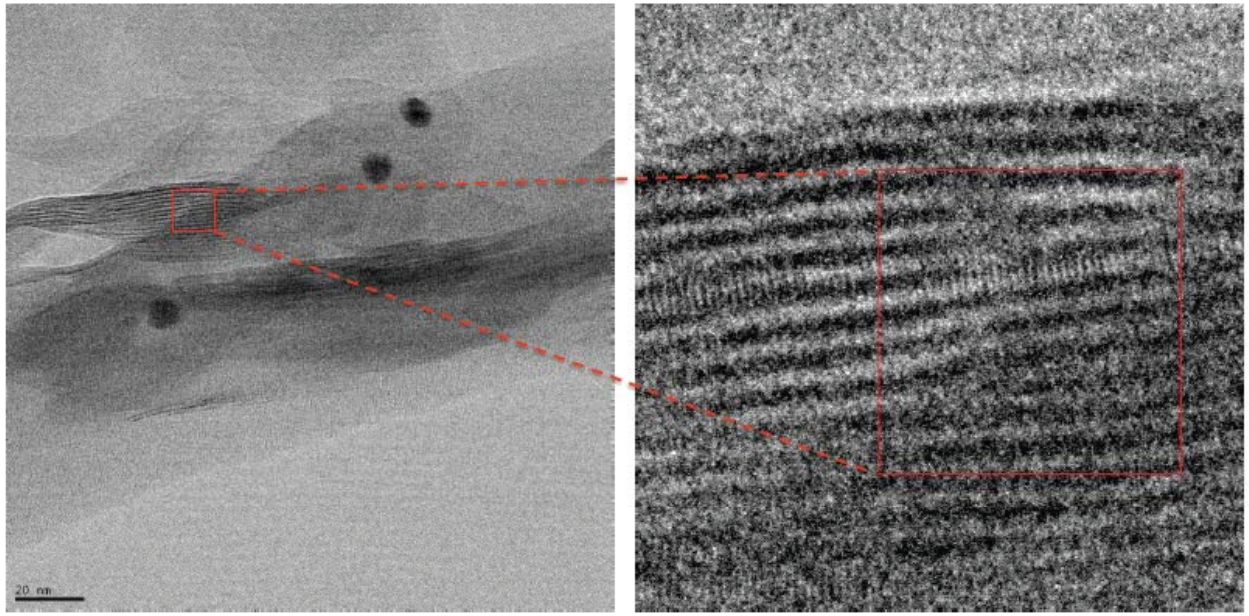


Skinner et al.
PRL, 2010.

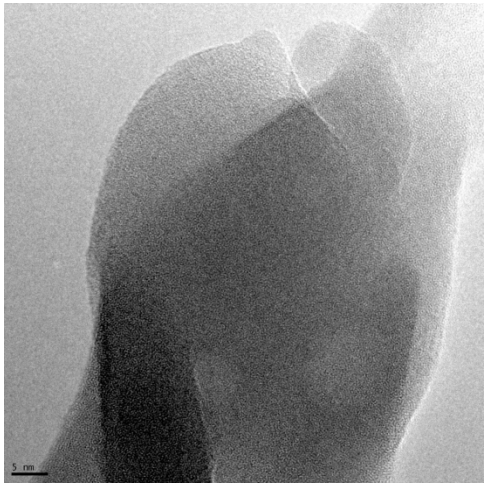
Why not TEM tomography?



Potential damage



cryo - HRTEM



Summary

Exciting developments in x-ray microscopy will allow to image highly complex chemical reactions. This can lead the way to development of sustainable construction materials.

Funding

KAUST

National Science Foundation

Recovery Act

DOE