Indentation, plastic shrinkage cracking, sulfate attacks, freeze-thaw behavior: Various topics on which I interacted with Gilles

M. Vandamme

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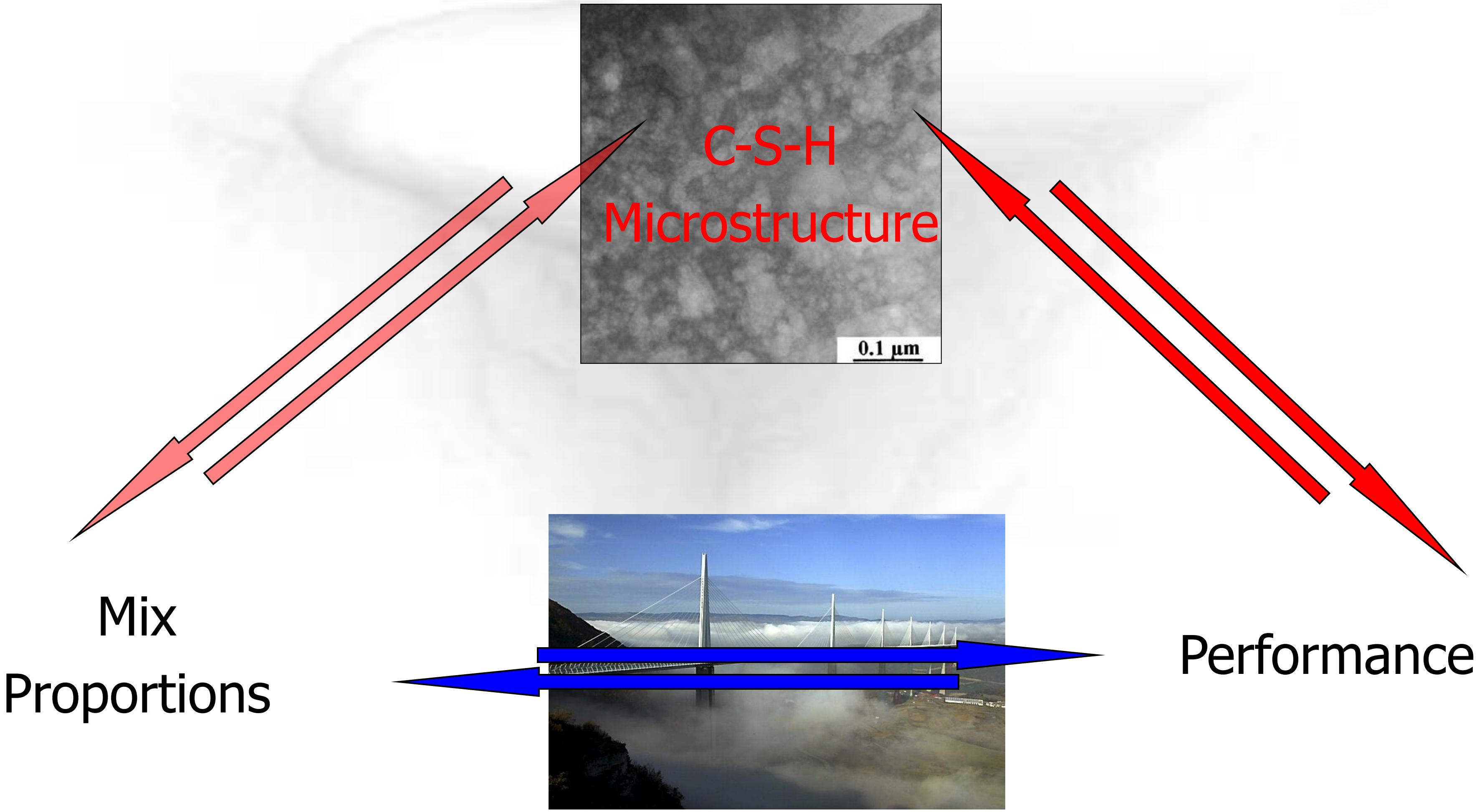
Outline

- <u>C-S-H at submicrometer scale</u>
- Plastic shrinkage cracking
- Sulfate attacks
- Resistance to freeze-thaw cycling

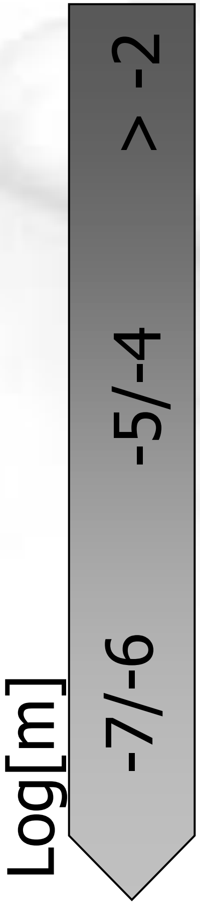
with: F.-J. Ulm (MIT), J. Chen (LCR), L. Sorelli (LCR), & Gilles

Approach

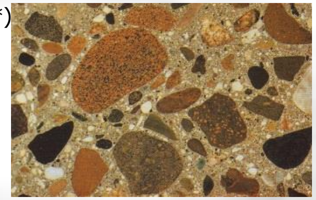
[Gatty et al., J. Mat. Sc., 2001]



Concrete multiscale structure



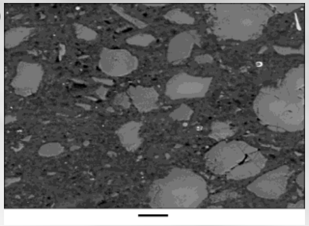
Level III
Concrete

(*)


1 cm

Cement paste +
aggregates

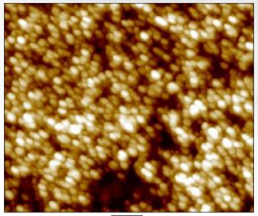
Level II
Cement Paste

(**)


10 mm

C-S-H matrix
+ other hydration products
(CH,…)
+ unhydrated clinker

Level I
C-S-H Matrix

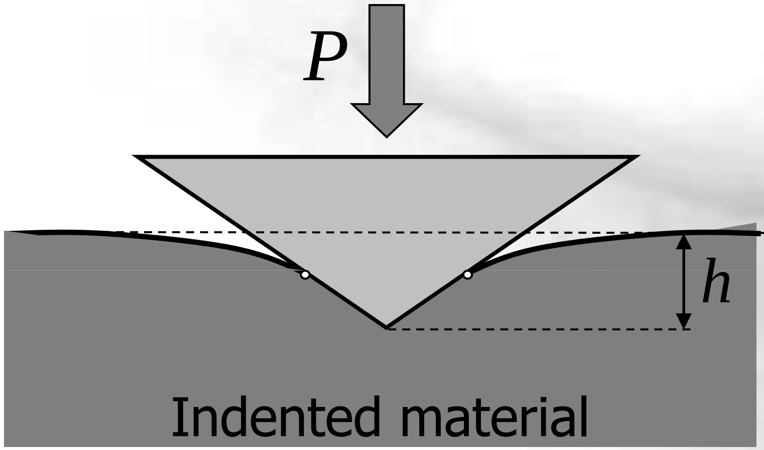
(***)


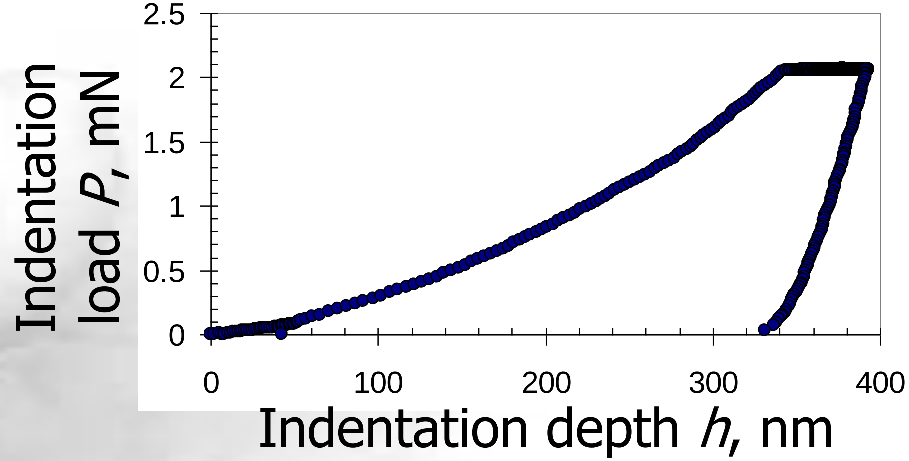
100 nm

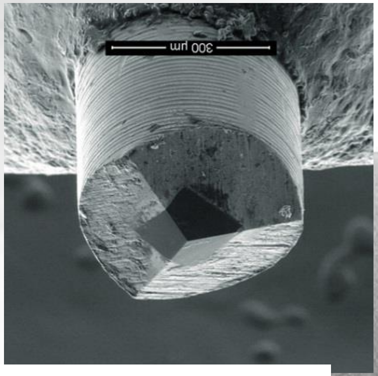
C-S-H solid +
pores

(*) Kosmatka et al., *Design and control of concrete mixtures,* 2002.
(**) Miller et al., *CCC*, 2008.
(***) Nonat A., *CCR*, 2004.

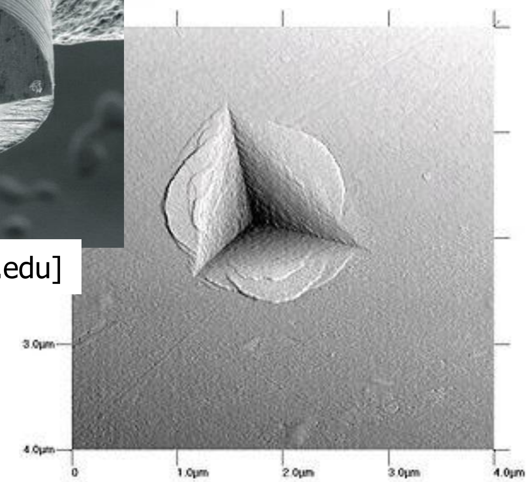
Indentation testing







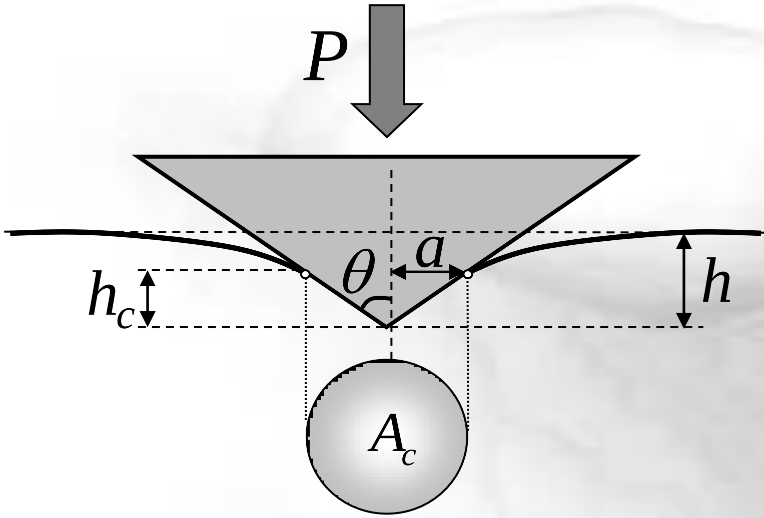
[http://www.unl.edu]

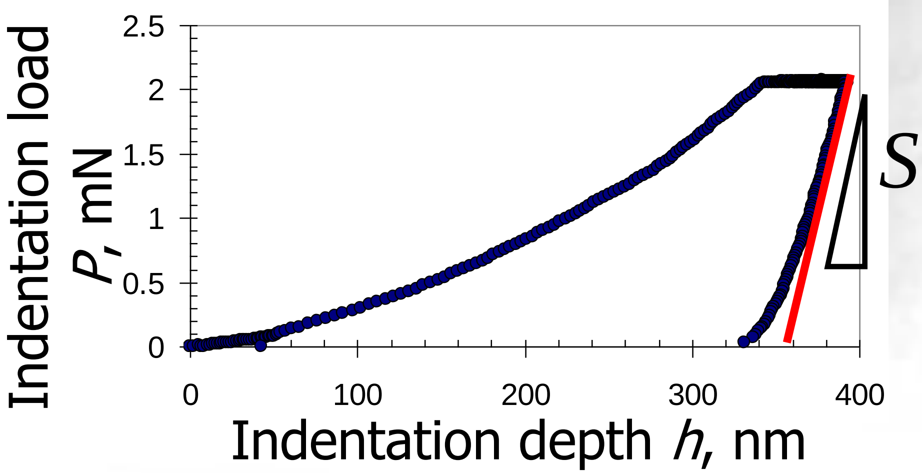


[www.absoluteastronomy.com]

Nanoindentation:
Typical Depth ~ 100 nm
Typical Load ~ 1 mN

Indentation testing



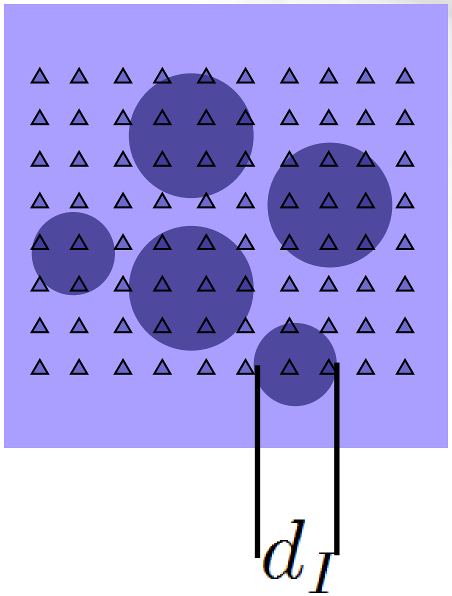


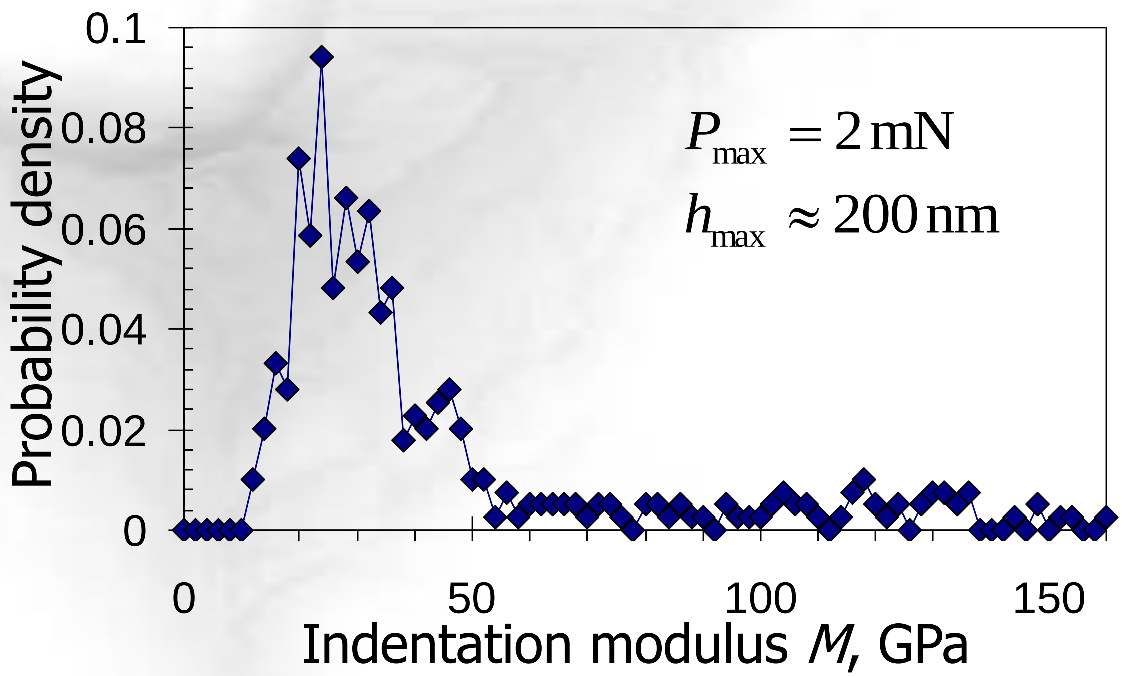
Indentation modulus

$$M = \frac{\sqrt{\pi}}{2}\frac{S}{\sqrt{A_c}} = \frac{E}{1-\nu^2}$$

Indentation of heterogeneous material

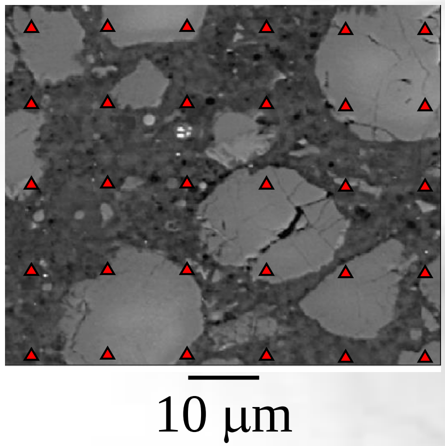
$h \ll d_I$

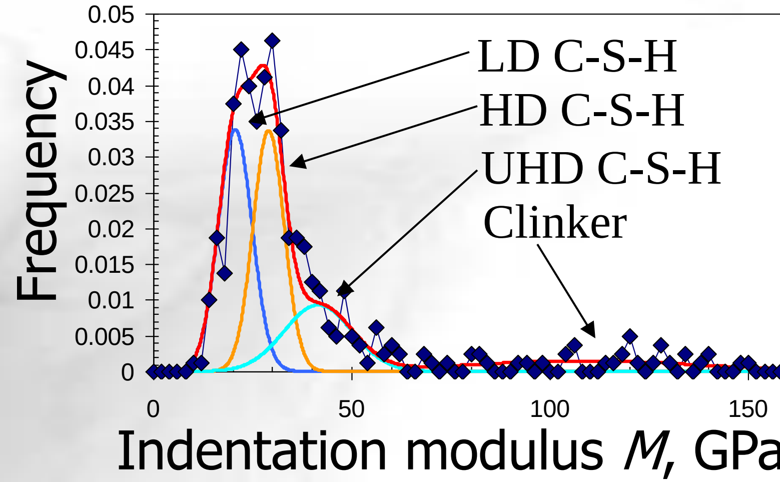


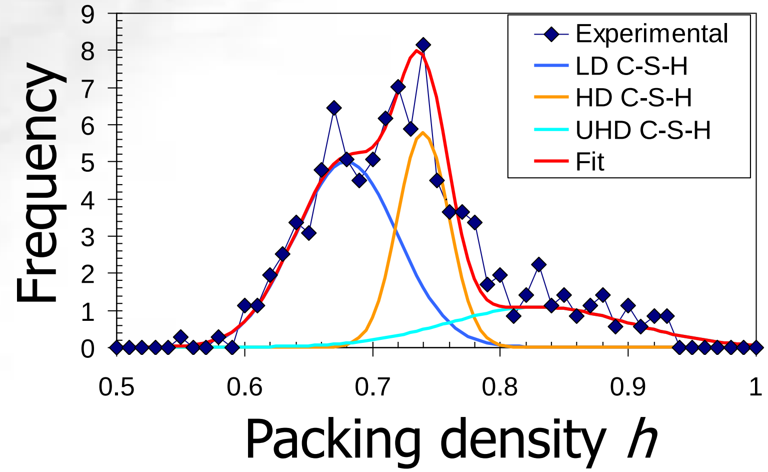


Heterogeneous response

Deconvolution process







- We identified 4 phases:
  - Clinker
  - 3 C-S-H phases:
    - Low-Density (LD)
    - High-Density (HD)
    - Ultra-High-Density (UHD)

My only paper with Gilles

journal

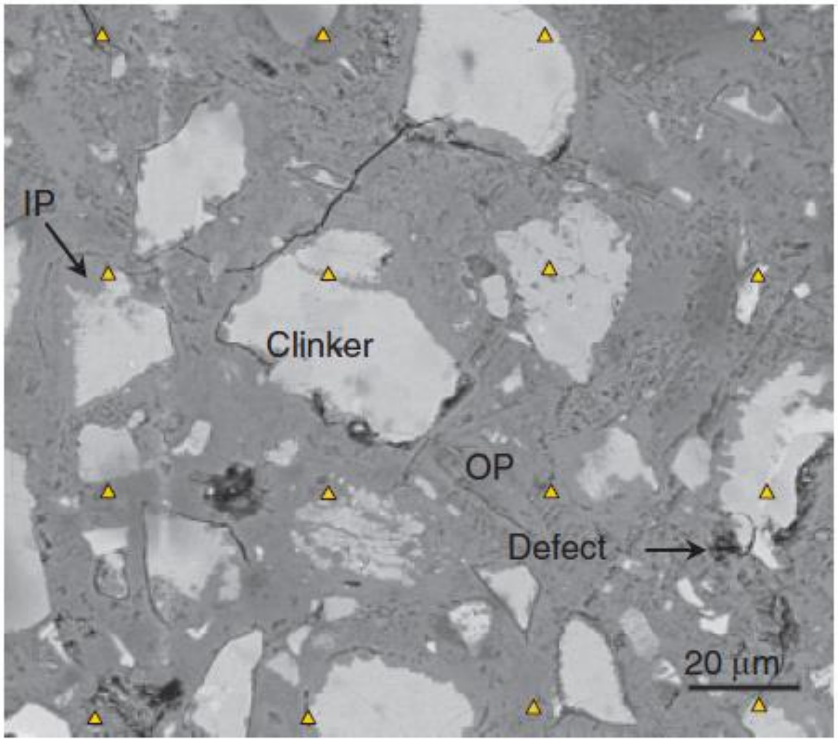
J. Am. Ceram. Soc., **93** [5] 1484–1493 (2010)
DOI: 10.1111/j.1551-2916.2009.03599.x

A Coupled Nanoindentation/SEM-EDS Study on Low Water/Cement Ratio Portland Cement Paste: Evidence for C–S–H/$Ca(OH)_2$ Nanocomposites

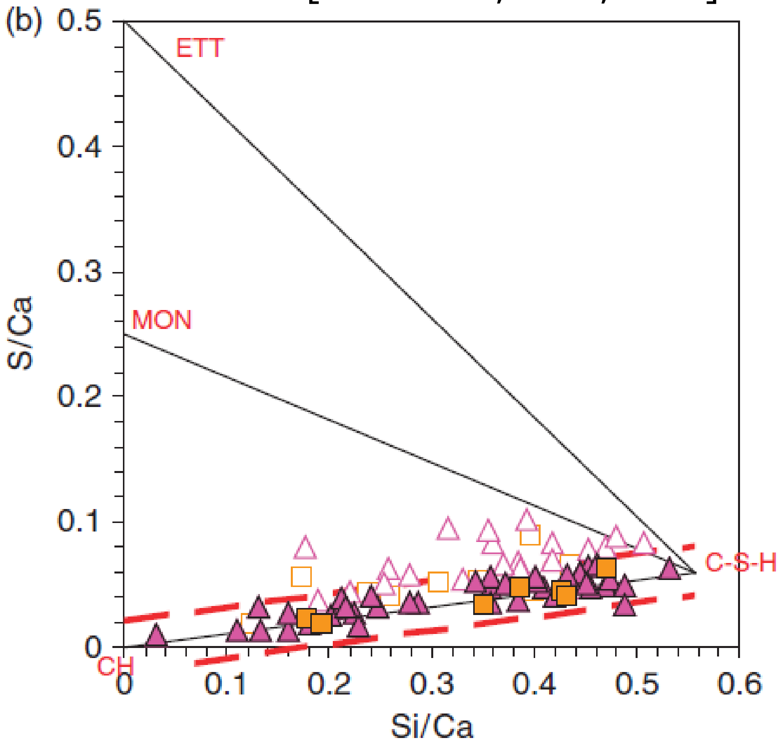
Jeffrey J. Chen,[†,‡] Luca Sorelli,[‡,¶] Matthieu Vandamme,[§,||] Franz-Josef Ulm,[§] and Gilles Chanvillard[‡]

[‡]Lafarge Centre de Recherche, 38291 Saint Quentin Fallavier, France
[§]MIT, Department of Civil and Environmental Engineering, Cambridge, Massachusetts 02139

What “Ultra-High-Density” C-S-H is



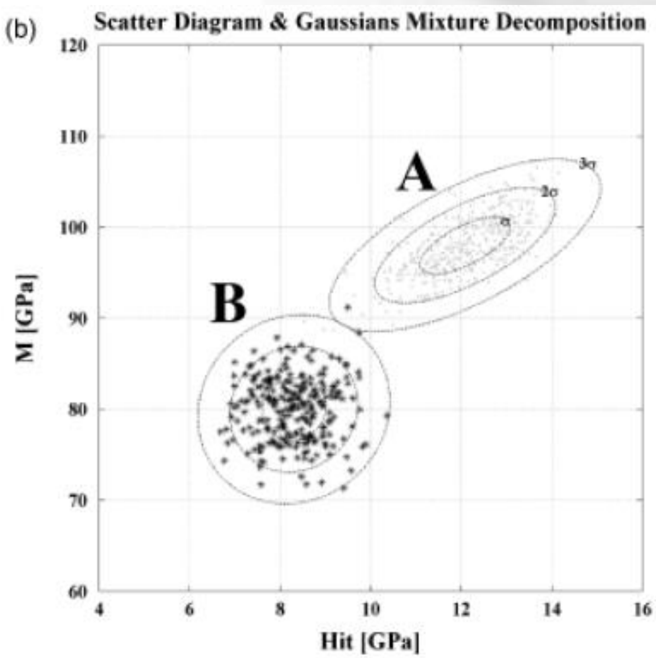
[Chen et al., *JACS*, 2010]

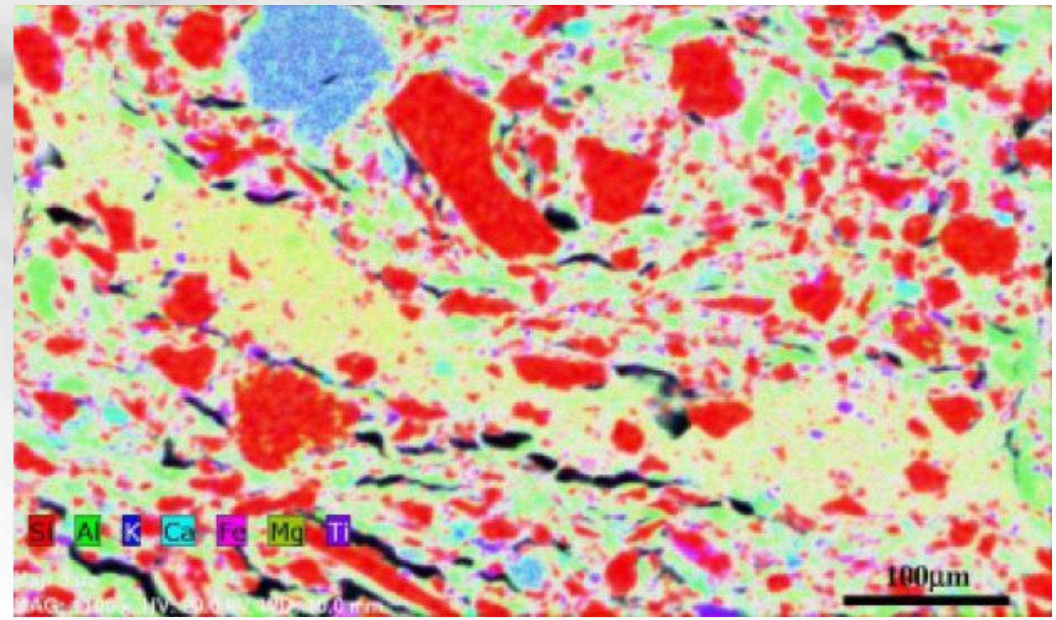


Ultra-High Density C-S-H was in fact:
High-Density C-S-H + portlandite (CH)

Multi-technique characterization

- Coupling indentation with chemical analysis





[Krakowiak et al., *JACS*, 2011]

Outline

- C-S-H at submicrometer scale
- Plastic shrinkage cracking
- Sulfate attacks
- Resistance to freeze-thaw cycling

with: W. Wang (Navier),
F. Bégaud (LCR), A. Delaplace (LCR), & Gilles

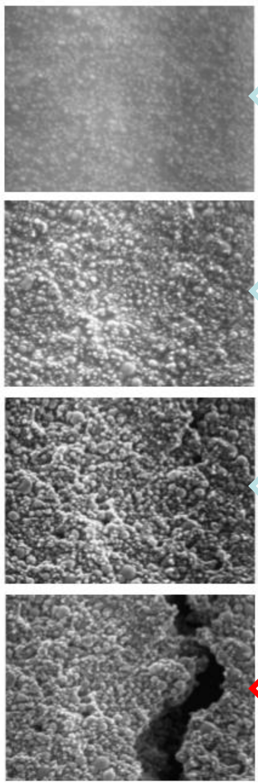
Plastic shrinkage cracking

[Slowik et al., *CCC*, 2009]

[http://www.oldearth.org]

[http://designshack.net]

Dyring-induced cracking



[Slowik, Schmidt, & Fritzsch, *CCC*, 2008]

Covered with water

Menisci form

Shrinkage

Cracking

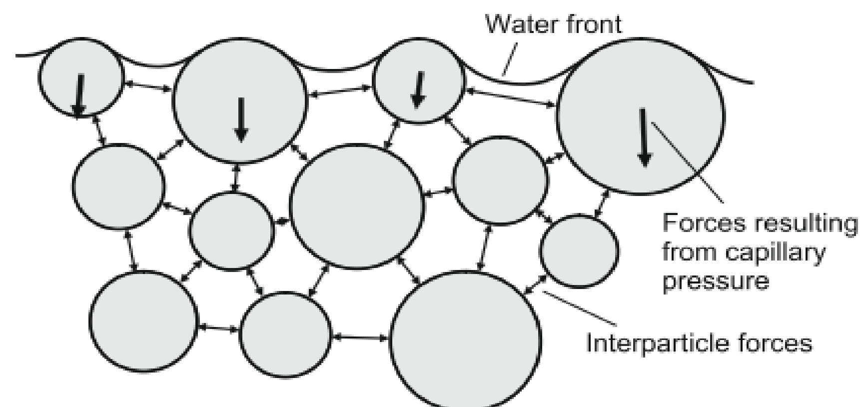
Evaporation

Capillary pressure increases

Radius of menisci

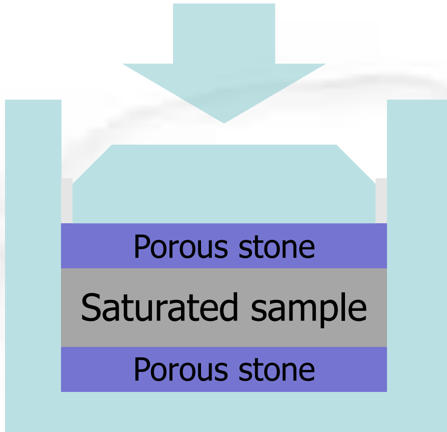
$$p_{cap} \propto 1/r$$

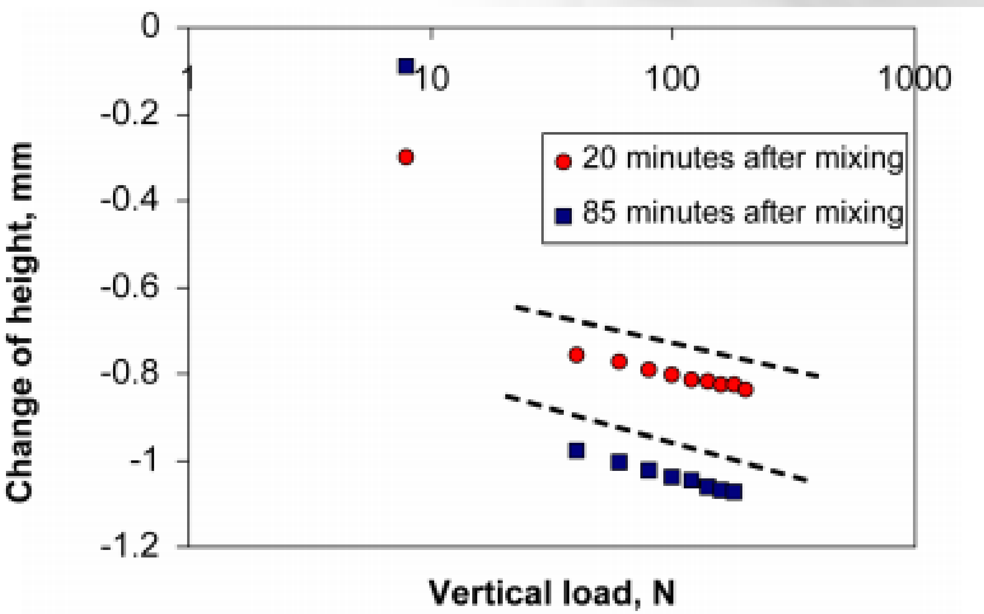
Capillary pressure = pressure of air - pressure of liquid

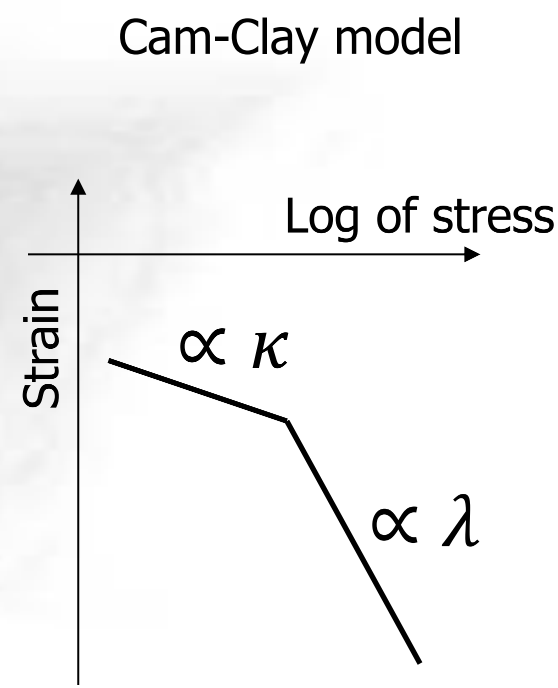


[Slowik et al., *CCC*, 2009]

Oedometer testing of early-age mortar

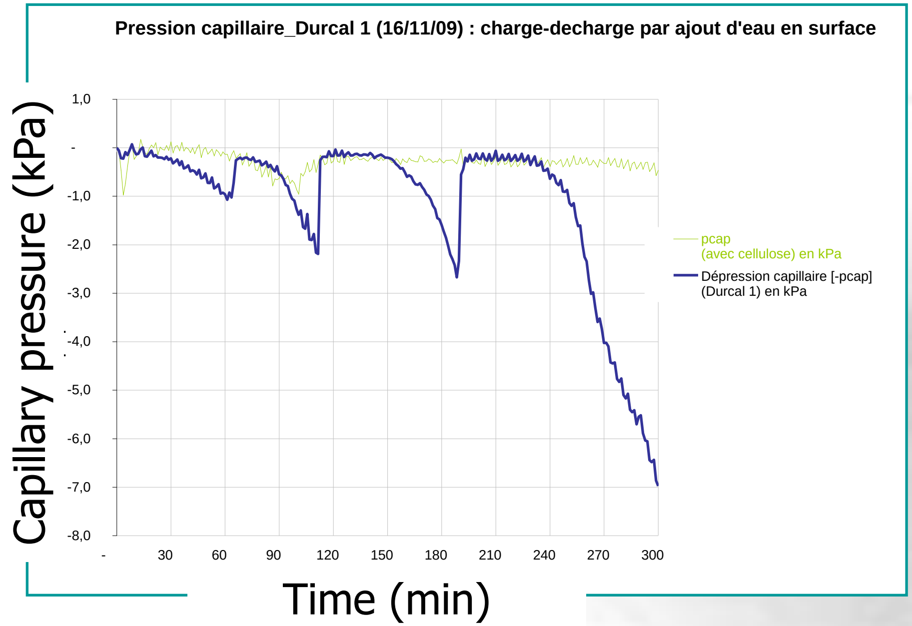


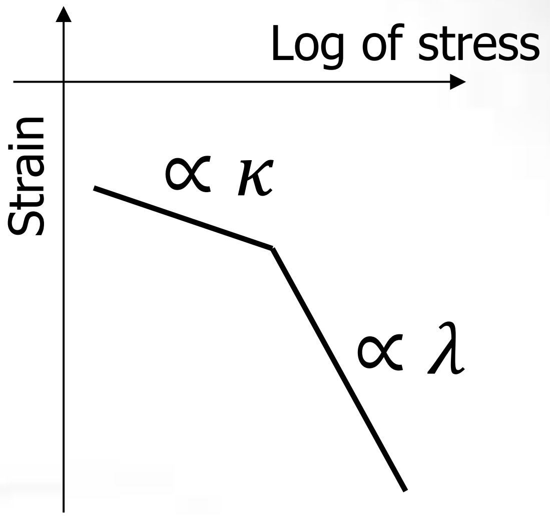


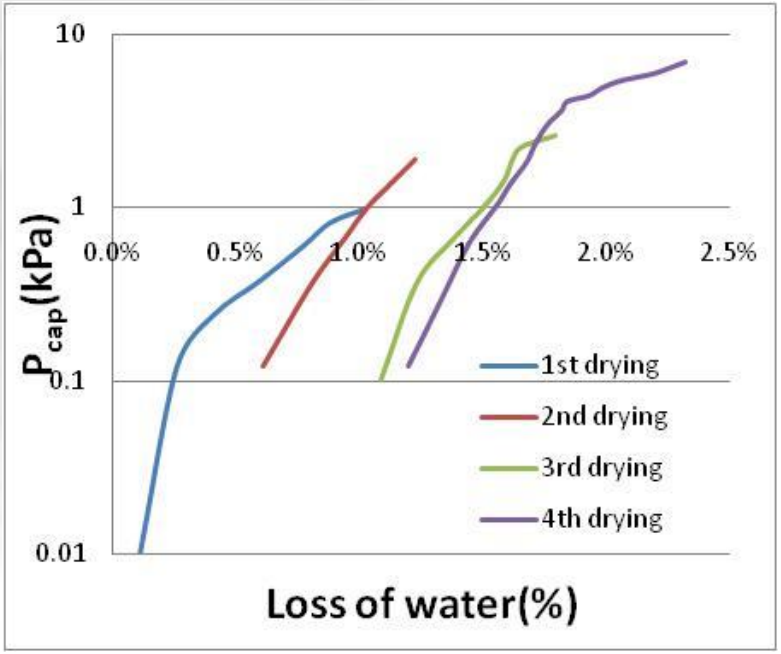


- → Bring concepts from soil mechanics

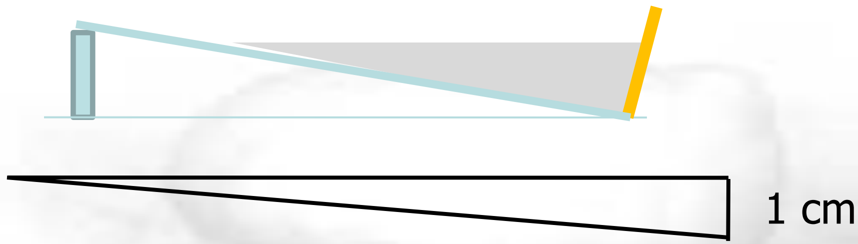
Drying & humidification of mortar slab

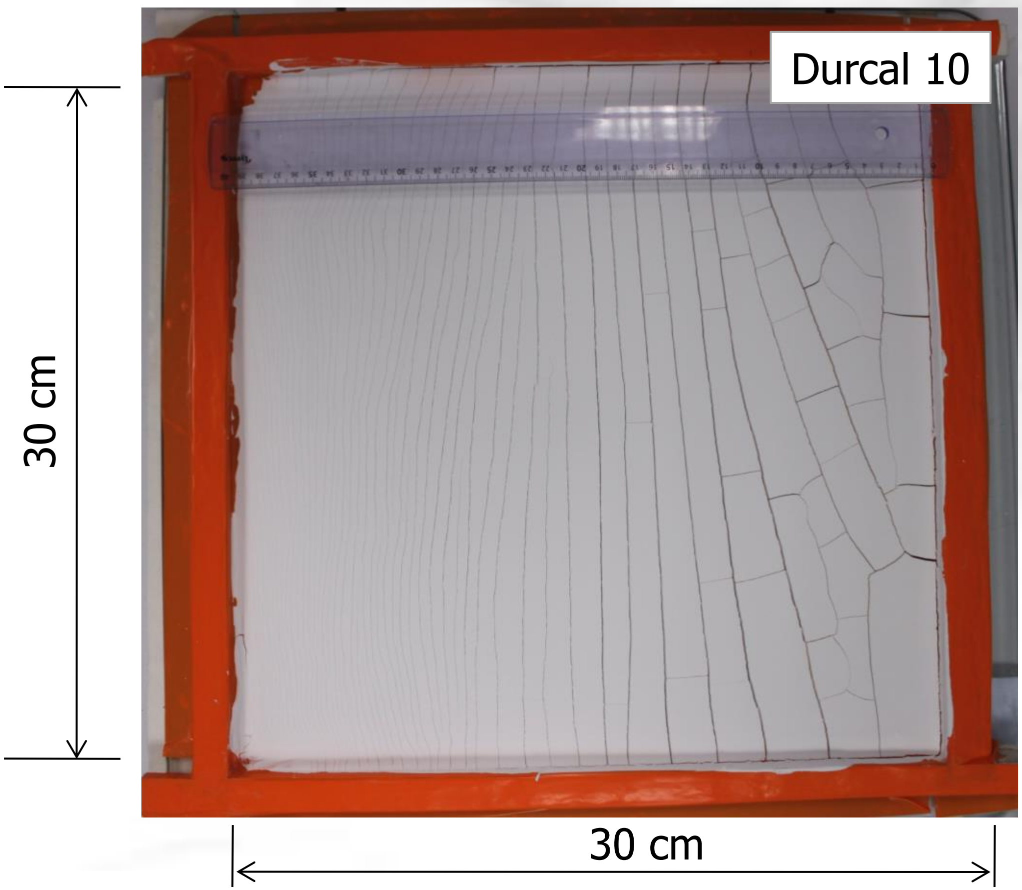


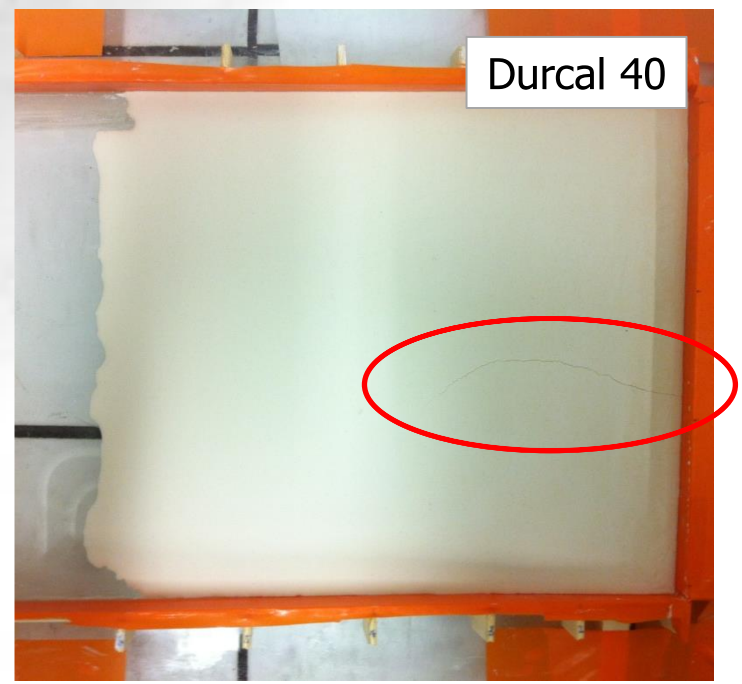




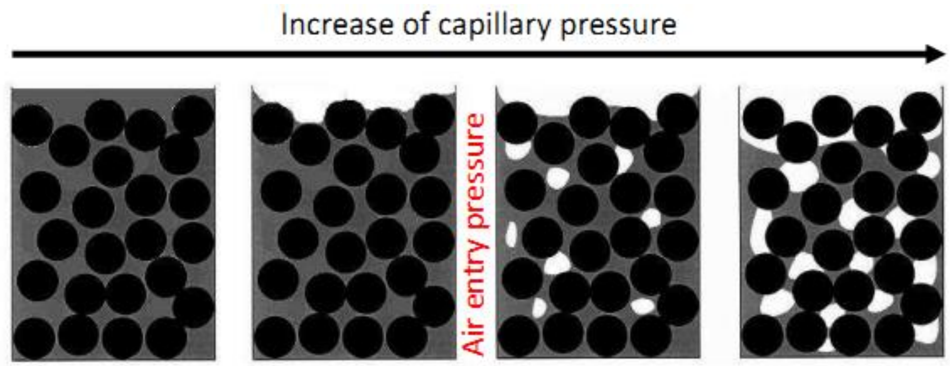
Inclined board test with filler



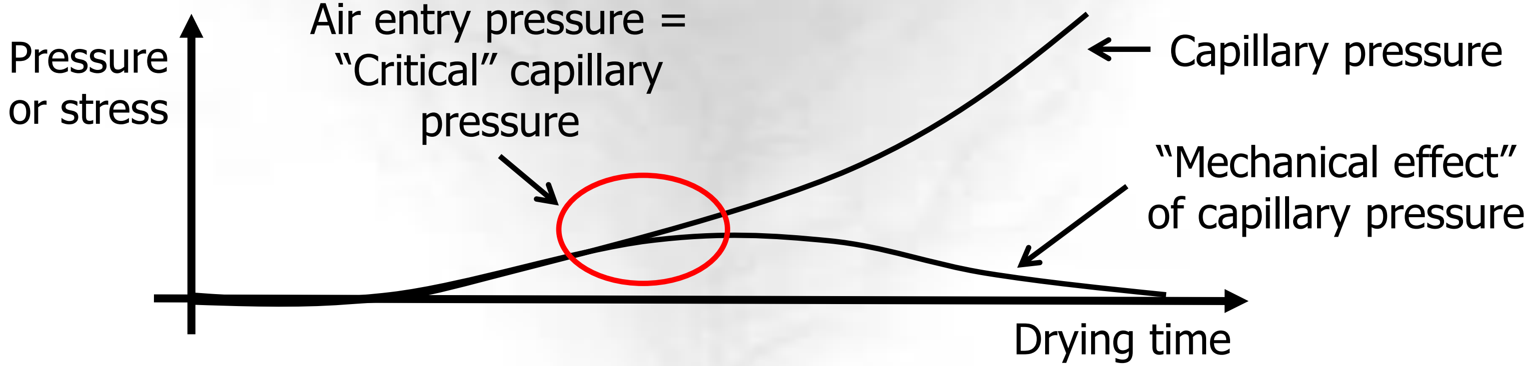




Role of air entry pressure



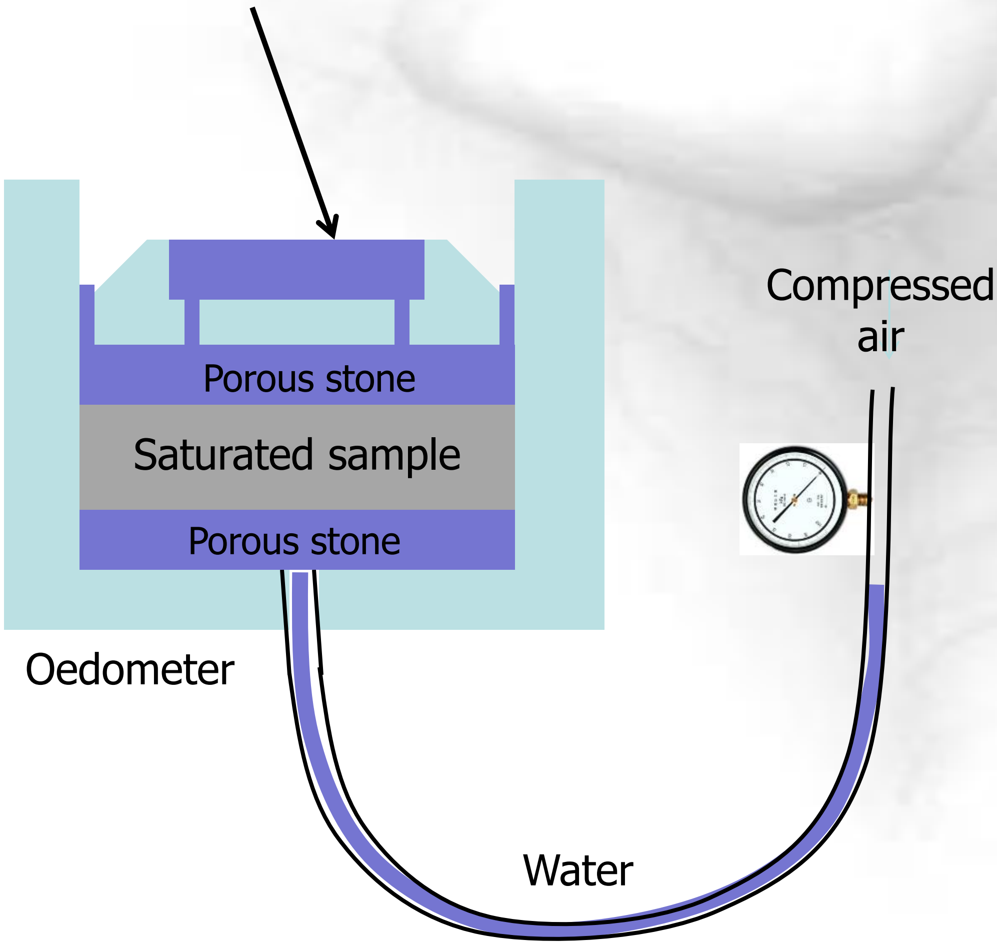
Adapted from [Coussot, *Eur. Phys. J. B*, 2000]

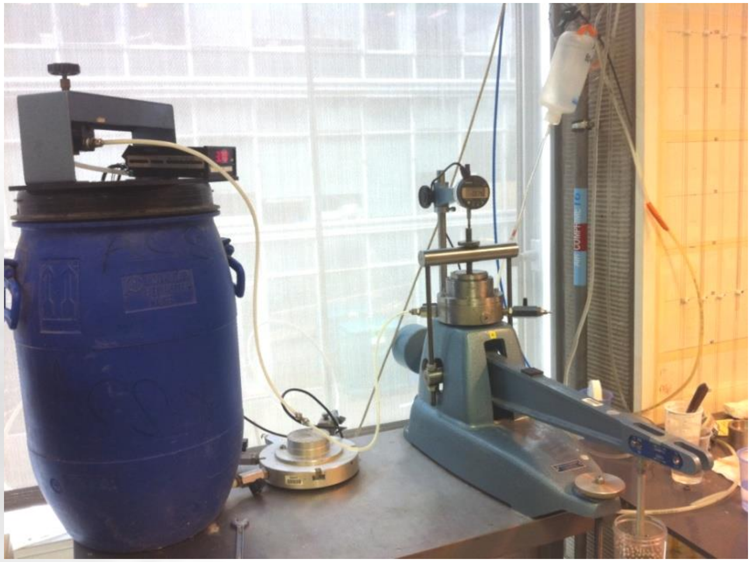


- Air entry pressure ~ most critical capillary pressure from mechanical point of view
- Air entry pressure proportional to $1/R$, with R radius of particles

Air-entry pressure measurement

Observation of first air bubble through material → Estimated air-entry pressure





Critical thickness results

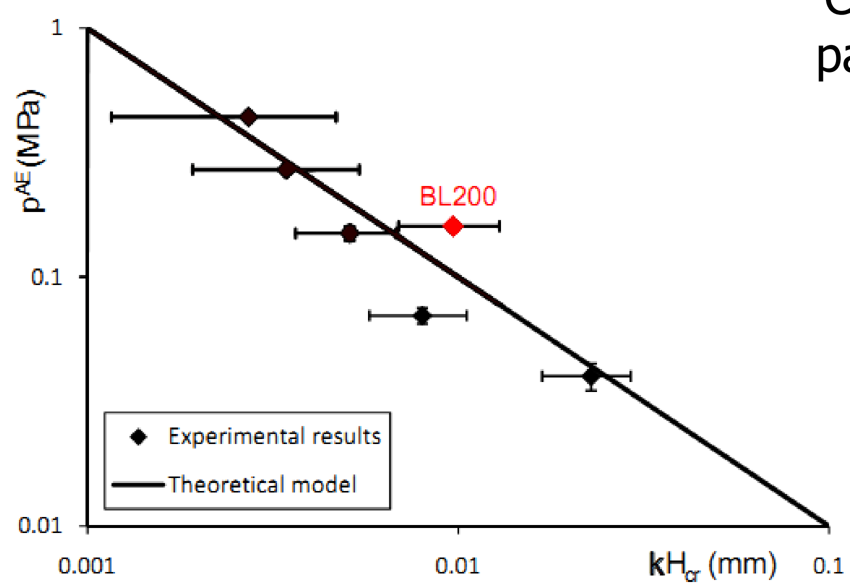
- Model for predicting risk of cracking:
  - Cam-Clay model
  - Air-entry pressure as “the” most critical capillary pressure
  - Cracking criterion based on fracture mechanics

$$p_{AE} \propto 1/\kappa H_{cr}$$

Air-entry pressure

Cam-Clay parameter

Critical height



Outline

- C-S-H at submicrometer scale
- Plastic shrinkage cracking
- Sulfate attacks
- Resistance to freeze-thaw cycling

with: N. N. Bui (Navier), J.-M. Pereira (Navier),
R. Barbarulo (LCR), & Gilles

Sulfate attacks

External or internal sulfate attacks can lead to expansion and damage of cementitious materials

[Ph.D. thesis Khelifa, *Univ. Orléans*, 2009]

Chemical reactions during sulfate attacks

$$2\ Ca^{2+} + 2\ SO_4^{2-} + 20\ H_2O + \underbrace{(CaO)_4(Al_2O_3)(SO_3)(H_2O)_{12}}_{\text{Monosulfoaluminate (AFm)}}$$

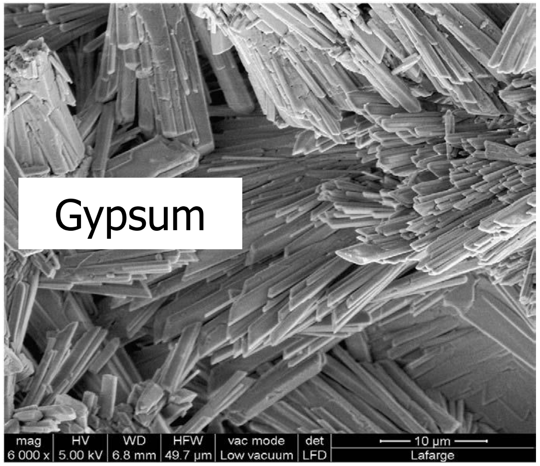
$$\Updownarrow$$

$$\underbrace{(CaO)_6(Al_2O_3)(SO_3)_3(H_2O)_{32}}_{\text{Ettringite (AFt)}}$$

$$2\ Na^{2+} + SO_4^{2-} + Ca^{2+} + 2\ (OH^-) + 2\ H_2O$$

$$\Updownarrow$$

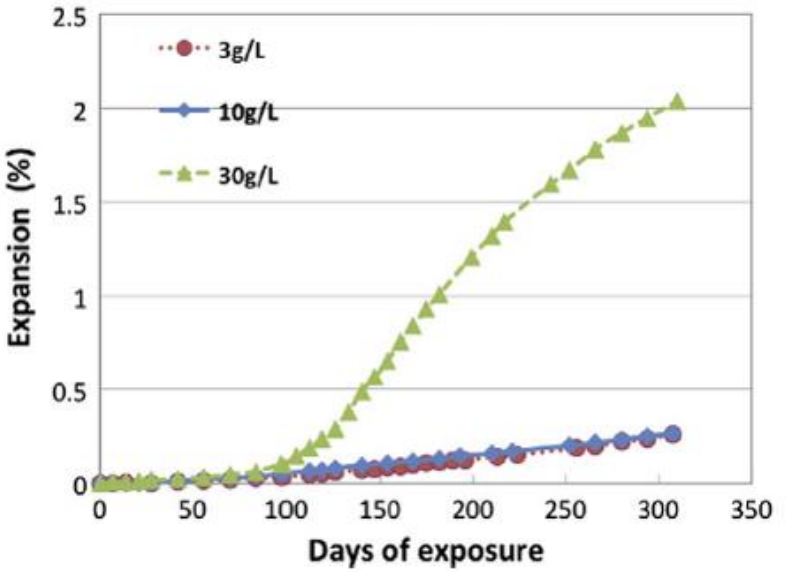
$$\underbrace{CaSO_4.2H_2O}_{\text{Gypsum } (C\$H_2)} + 2\ Na(OH)$$

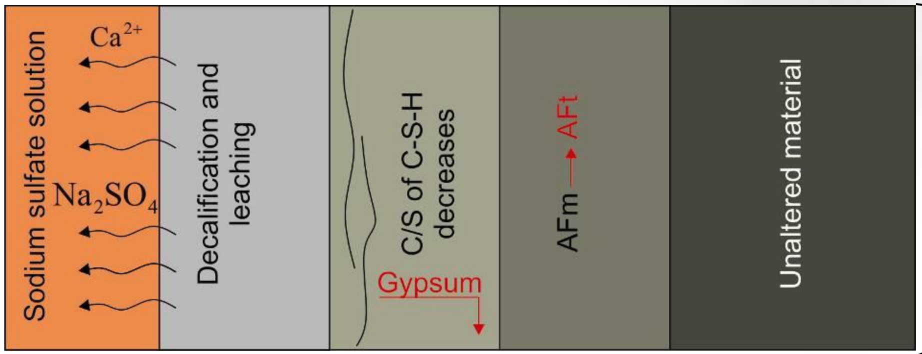


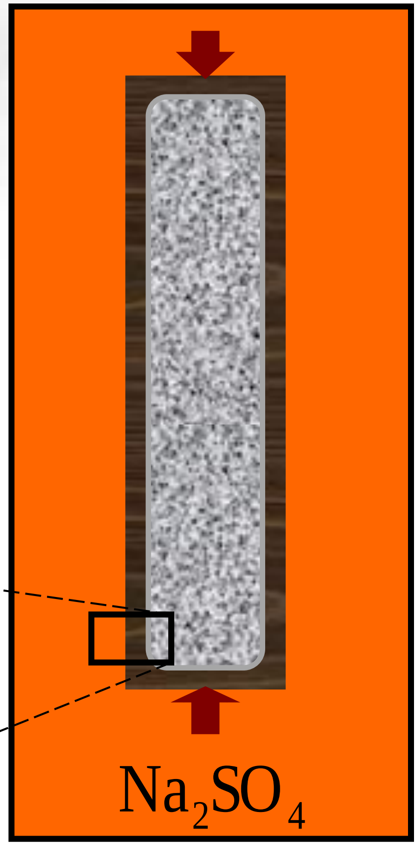
[Gartner, 2009]

Typical laboratory study

[Yu, Sun, & Scrivener, *CCR*, 2013]



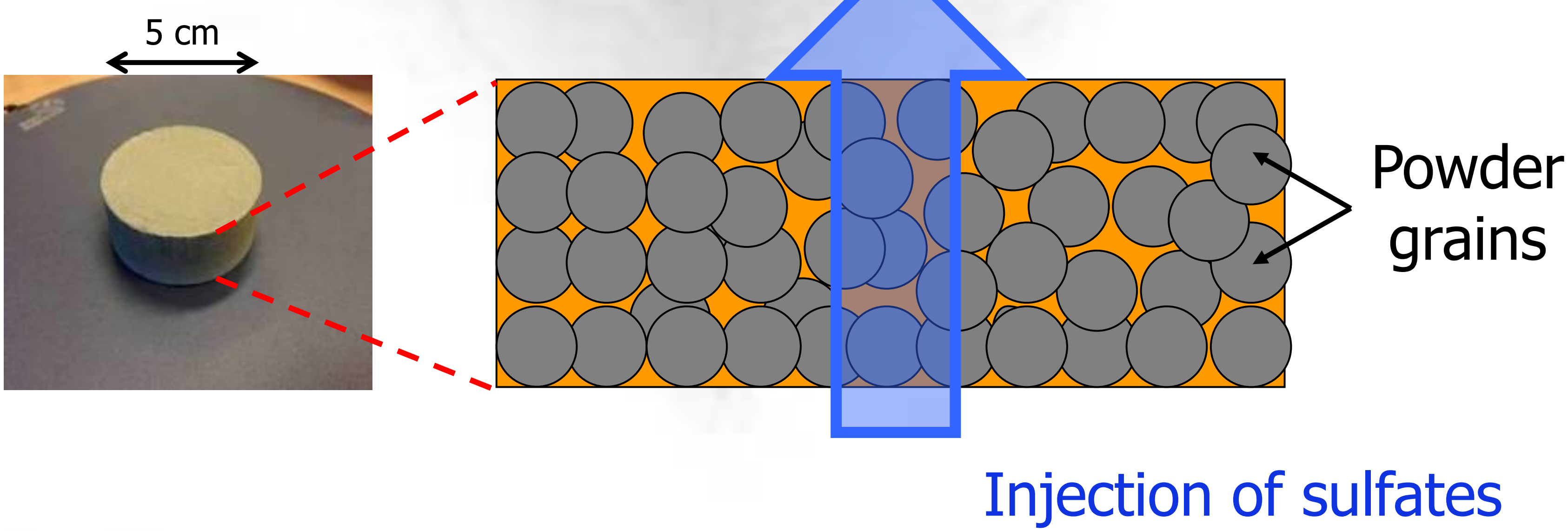




[Gollop & Taylor, *CCR*, 1992]

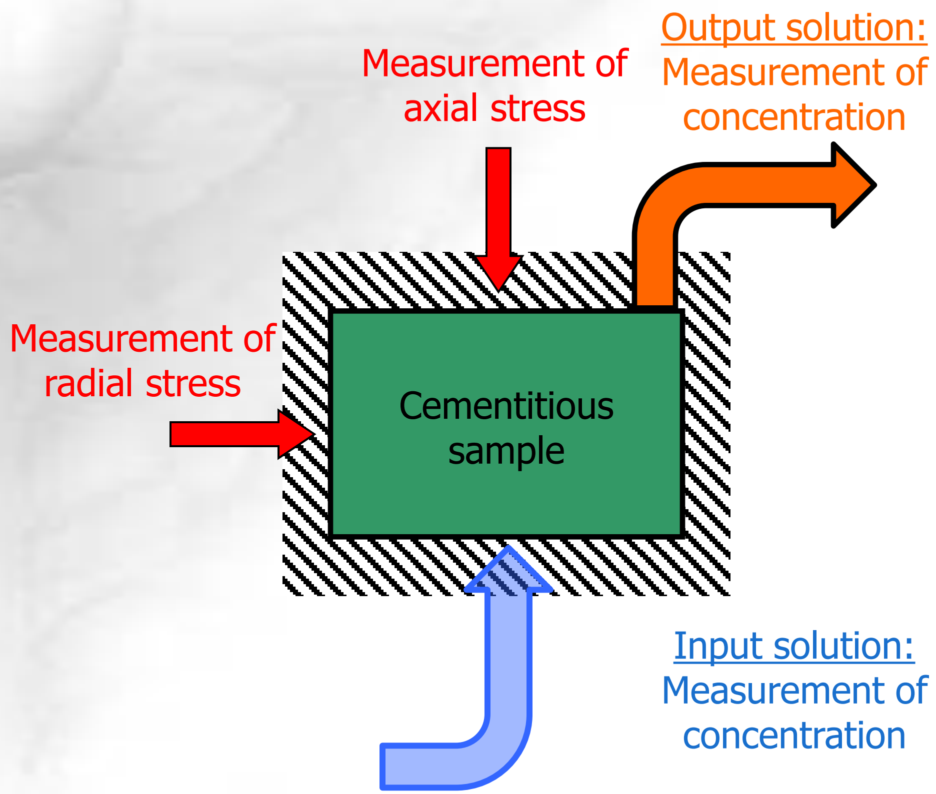
Work with compacted powders

- Objectives:
  - Faster expansion
  - More homogeneous response of sample
  - Realistic physico-chemical phenomena



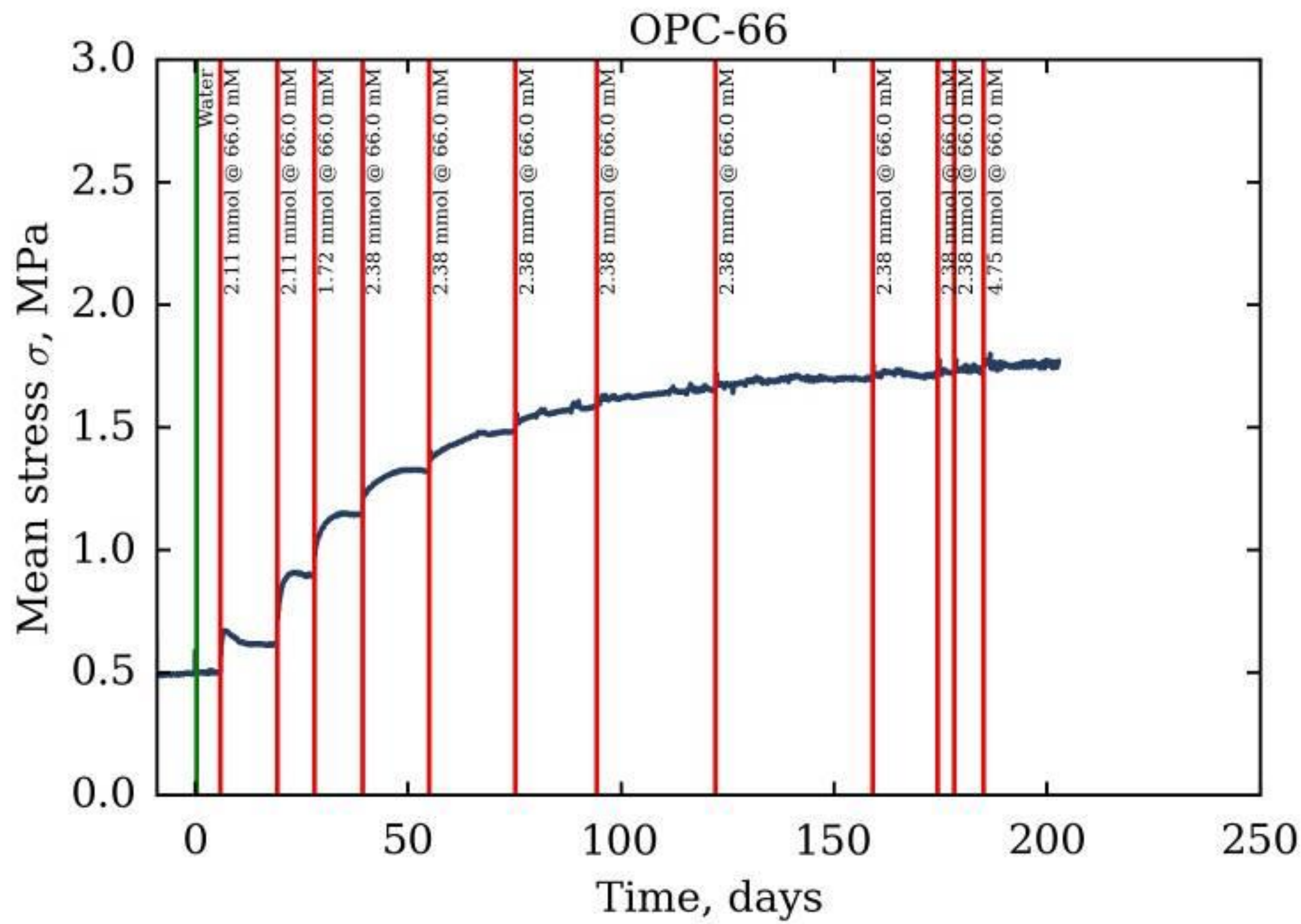
Isochoric testing

- Isochoric testing: Measurement of an expansion stress
  - No axial and radial strains
  - Measurement of axial and radial stress
  - Measurements of sulfate concentration of input and output solution



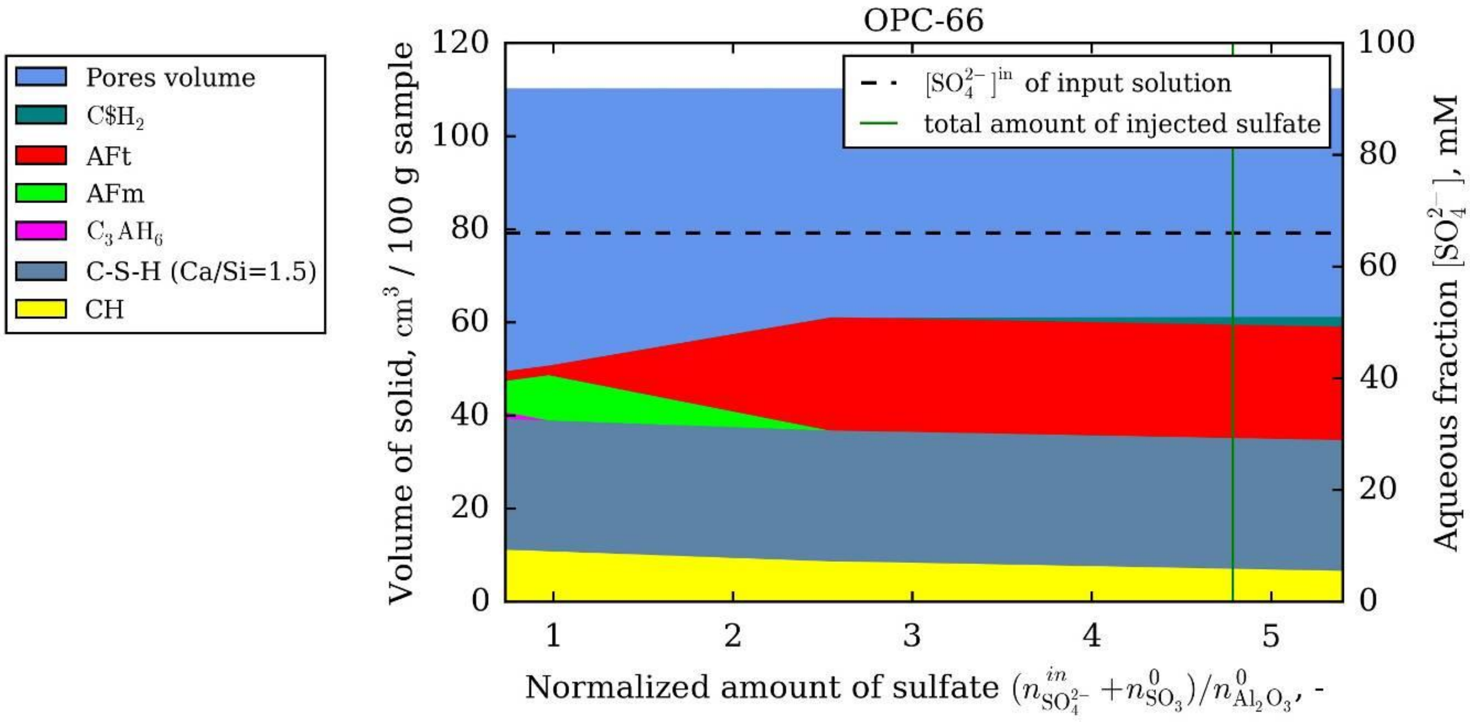
Typical isochoric experiment

Cement paste flushed with a sodium sulfate solution at 66 mM



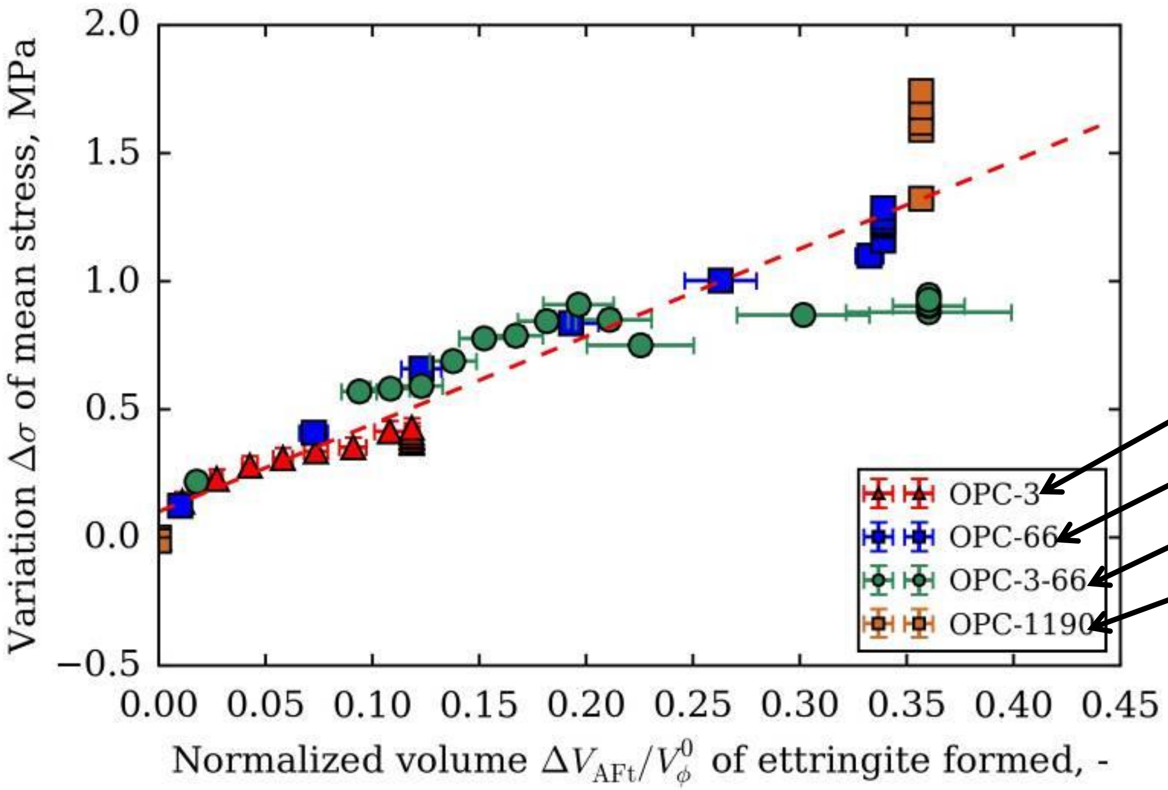
Thermodynamic modeling

- Thermodynamic models make it possibe to predict evolution of solid phase assembly over the injections



Cement paste flushed with a sodium sulfate solution at 66 mM

How crystallization induces a strain



Cement paste flushed with sodium sulfate solution at:

- 3 mM
- 66 mM
- 3 & 66 mM
- 1190 mM

- Expansion stress mostly independent of concentration, but scales linearly with amount of ettringite formed

Outline

- C-S-H at submicrometer scale
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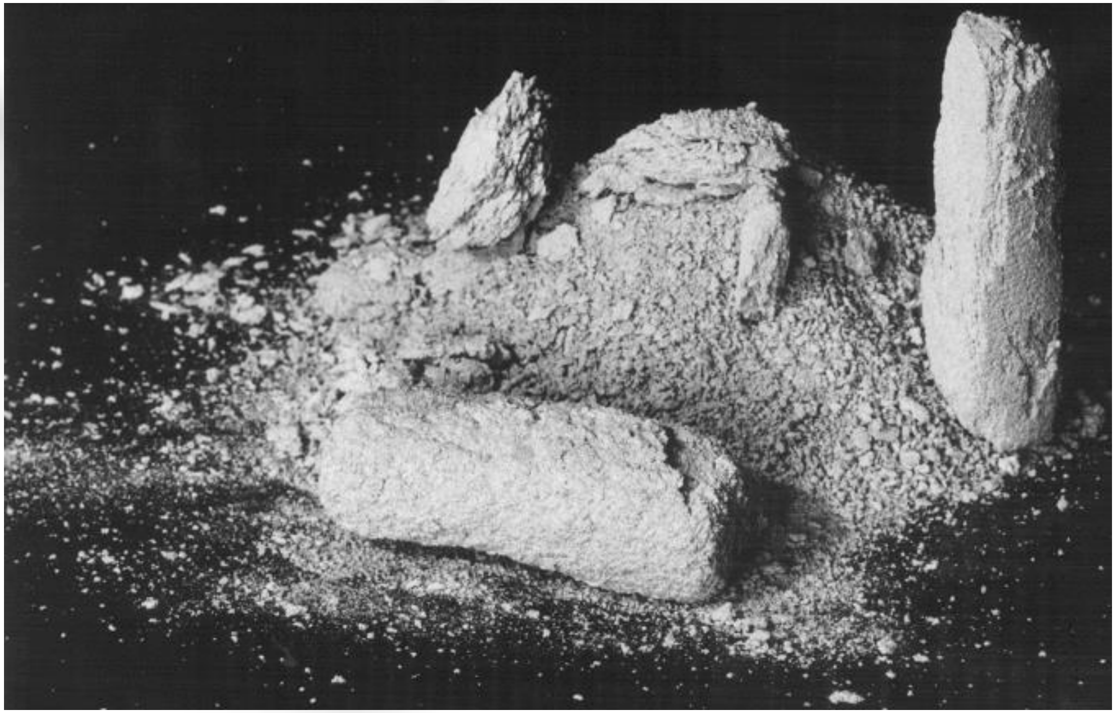
- Resistance to freeze-thaw cycling

with: N. Mayercsik (GaTech), K. Kurtis (GaTech), S. Brisard (Navier)

Damage induced by freezing

Damage caused by freezing in a stone

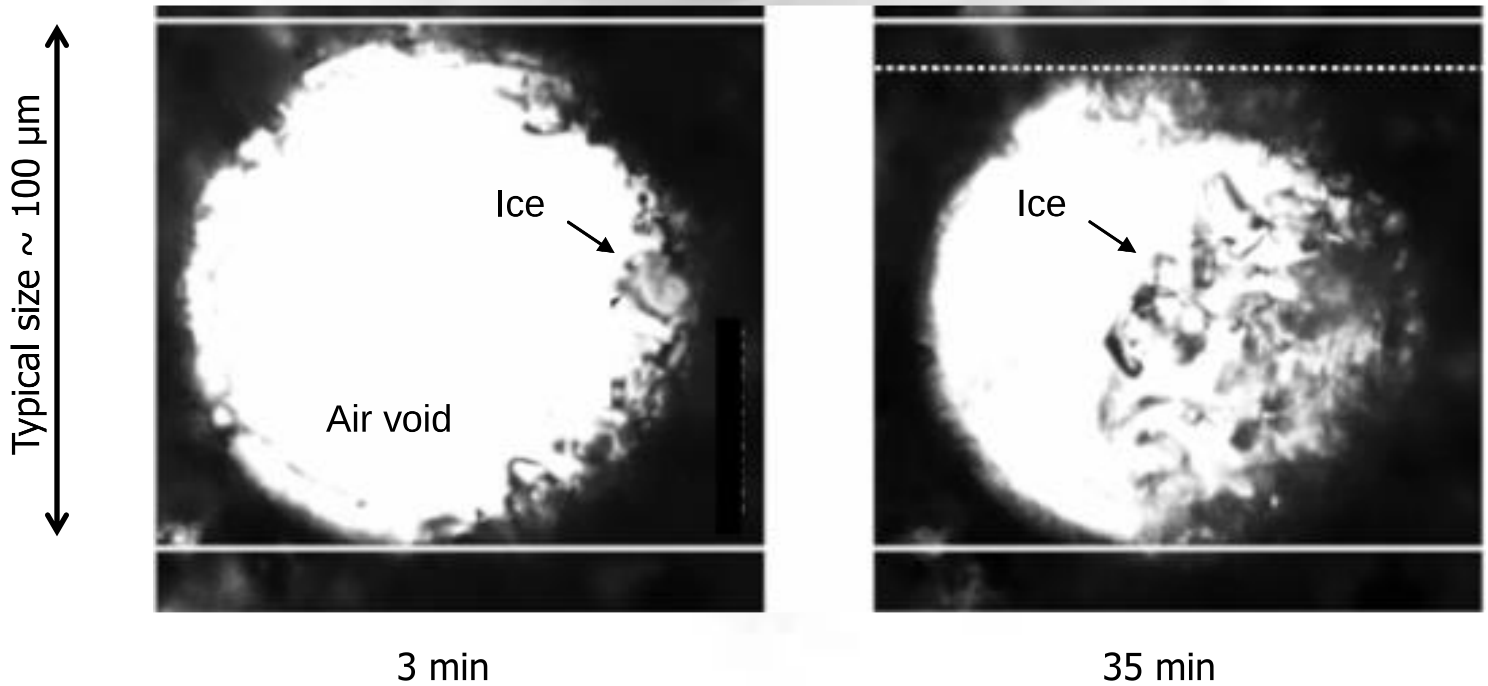


[Tourenq, *Rapp. Rech. LCPC*, 1967]

Role of air voids

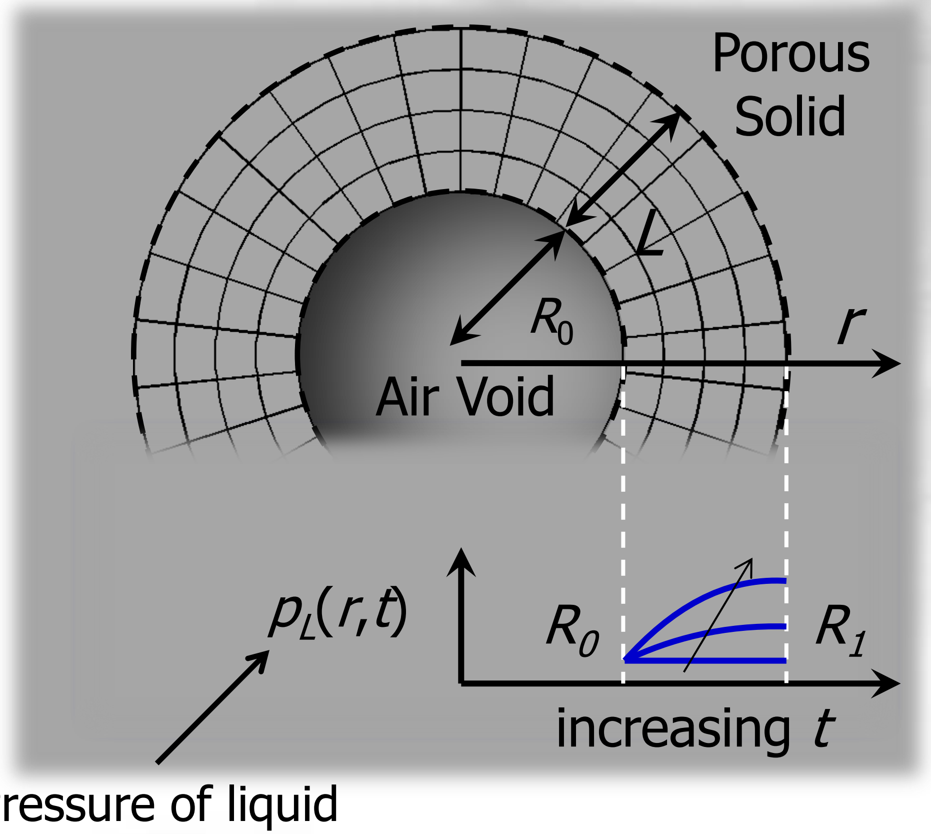
- Air voids serve as reservoirs of expansion, to avoid buildup of pore pressure during freezing

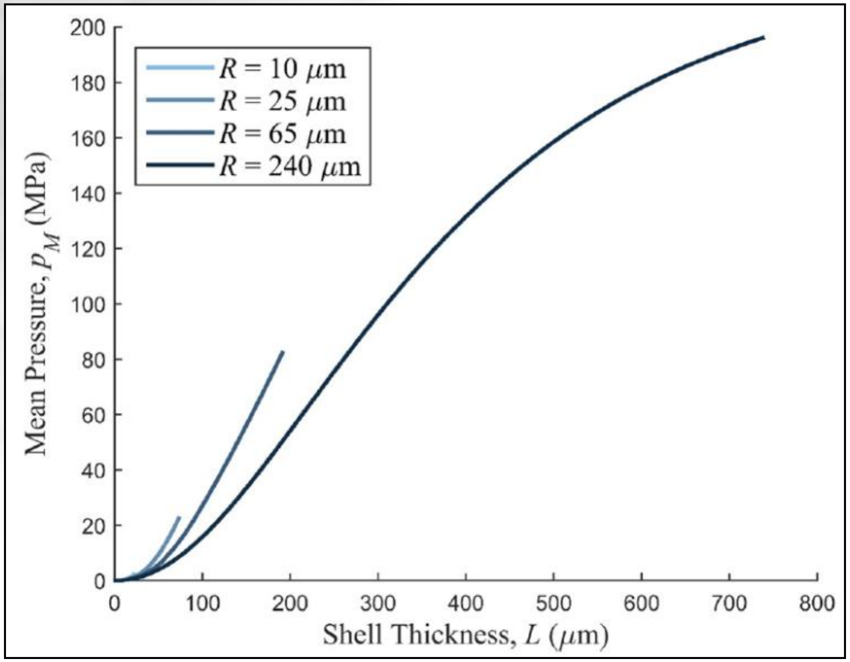
[Coussy, Mech. Phys. Por. Sol., 2010], courtesy from P. Monteiro



Poromechanical analysis of 1 air void

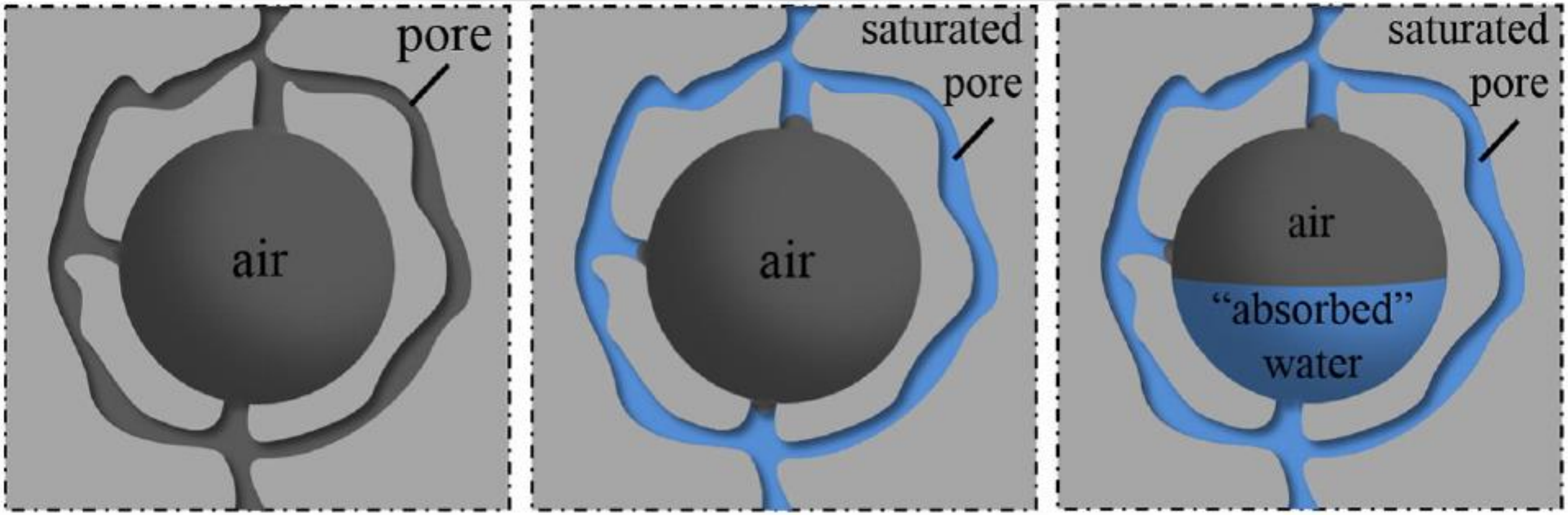
- From Coussy: $\frac{\partial}{\partial t}\left(S_C\left(\frac{1}{\overline{V}_L^0}-\frac{1}{\overline{V}_C^0}\right)\right)+\frac{\partial}{\partial t}\left[\left(\frac{S_C}{K_C}\left(\frac{\overline{V}_L^0}{\overline{V}_C^0}\right)^2+\frac{S_L}{K_L}\right)\frac{p_L}{\overline{V}_L^0}\right]=\frac{p_L}{\overline{V}_L^0}\frac{\kappa}{\phi_0\eta}\frac{1}{r^2}\frac{\partial}{\partial r}\left[k_{rL}(S_L)r^2\frac{\partial p_L}{\partial r}\right]$



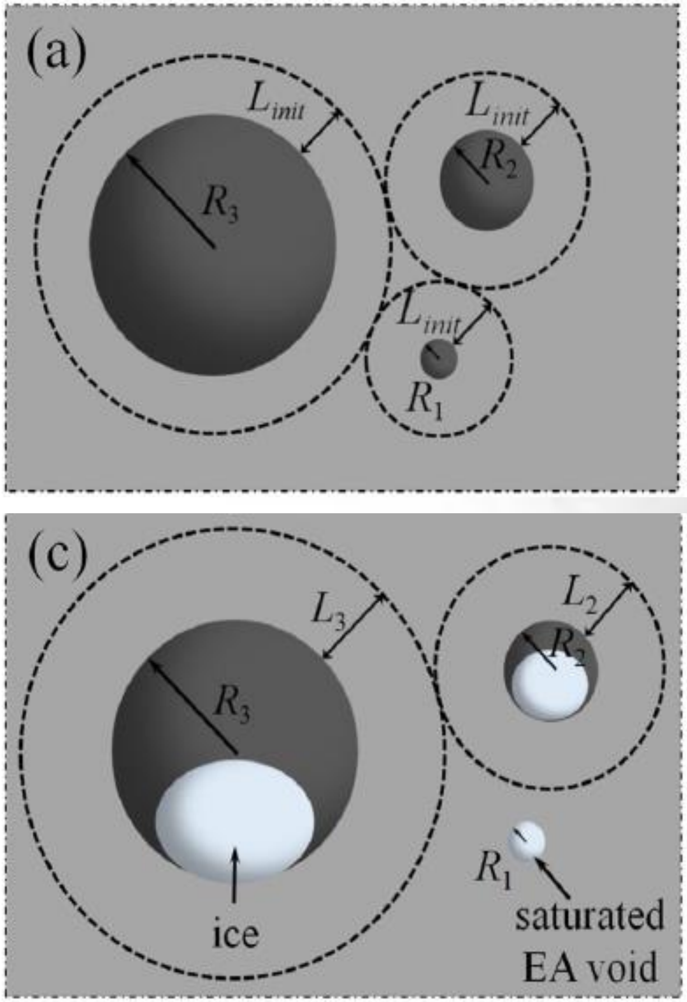


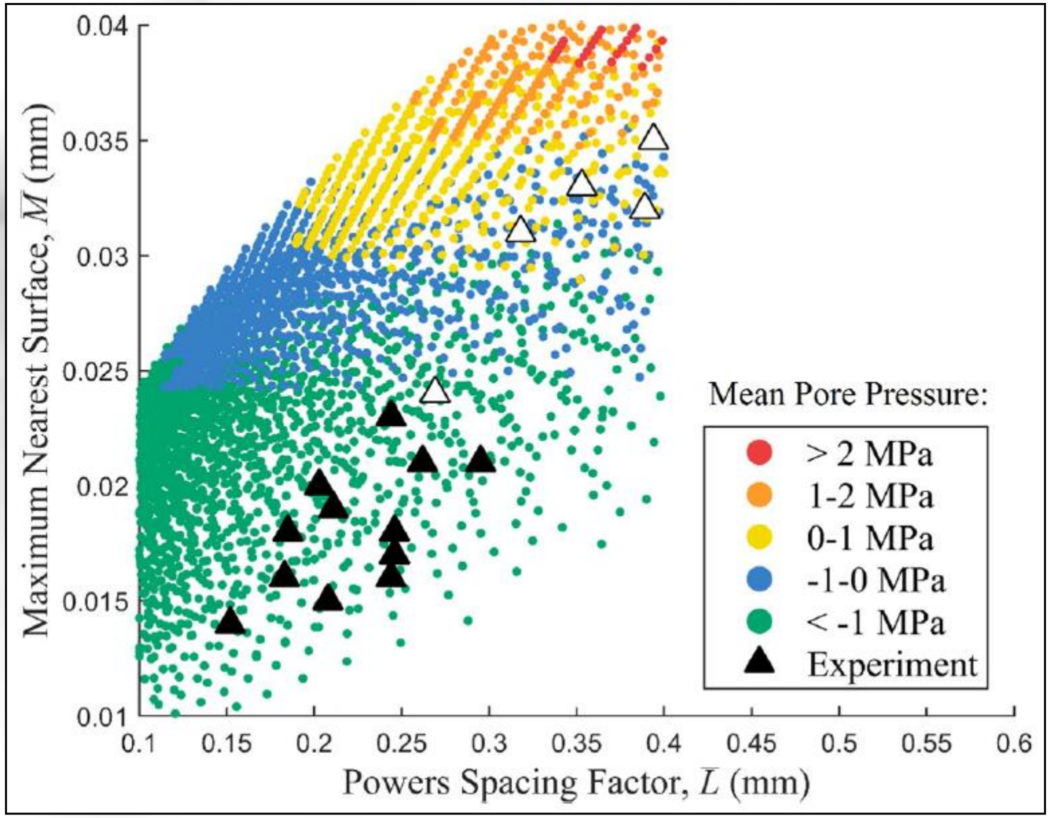
What happens upon immersion

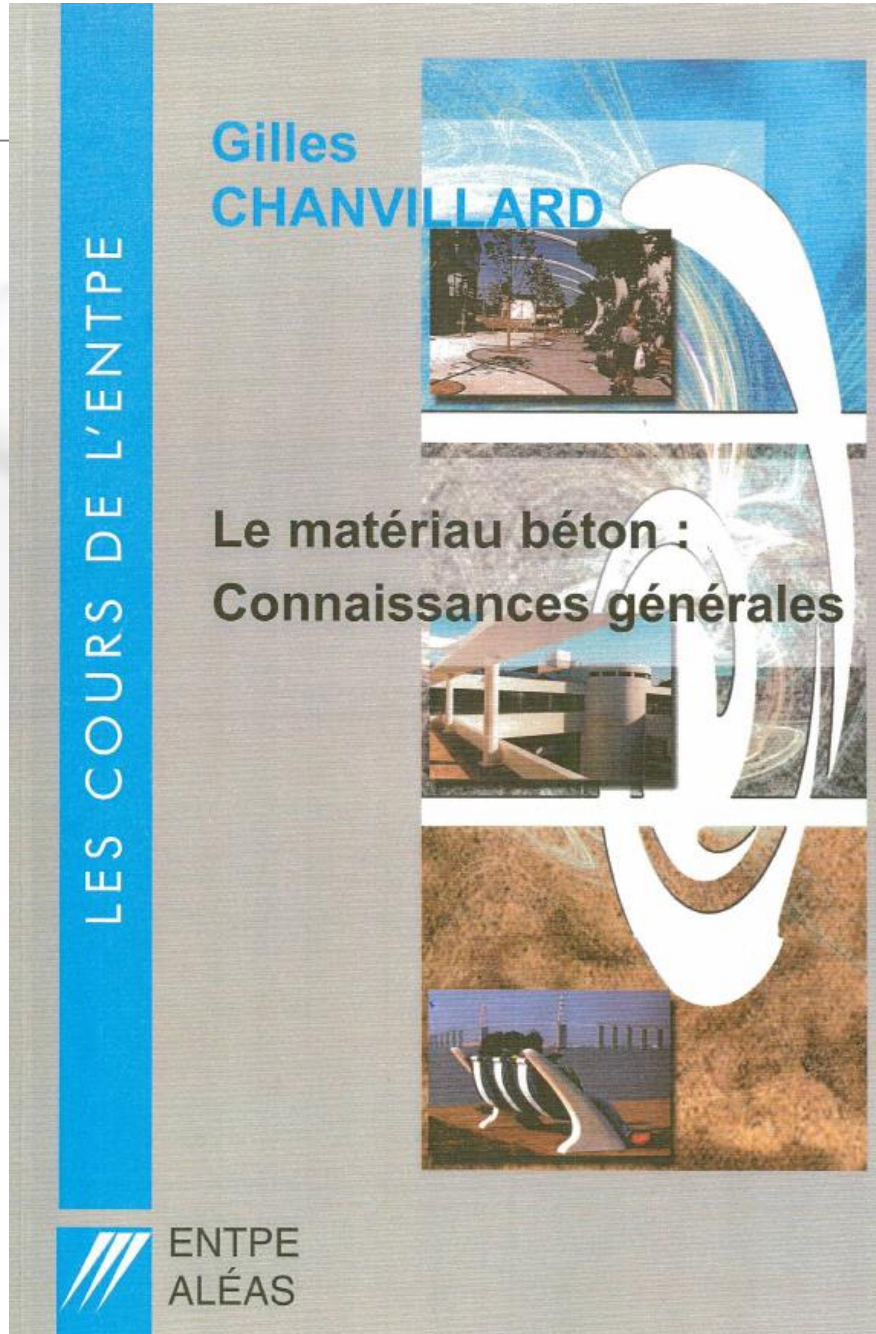
- Some air bubbles remain trapped. Trapped air needs to diffuse out.



Evolution of response upon cycling






LES COURS DE L'ENTPE
Gilles
CHANVILLARD
Le matériau béton :
Connaissances générales
ENTPE
ALÉAS