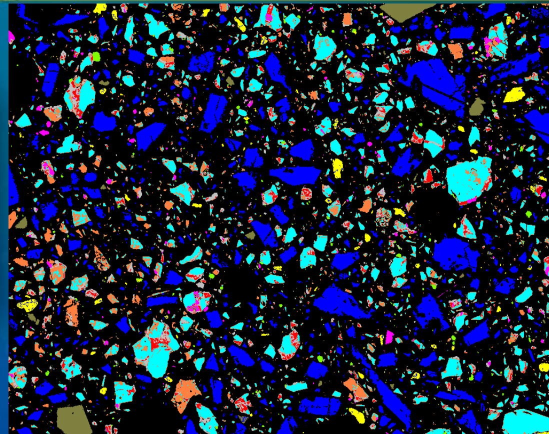
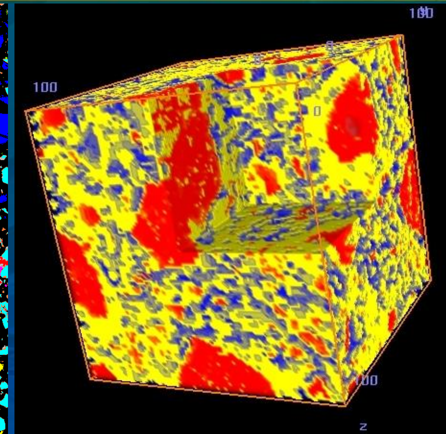
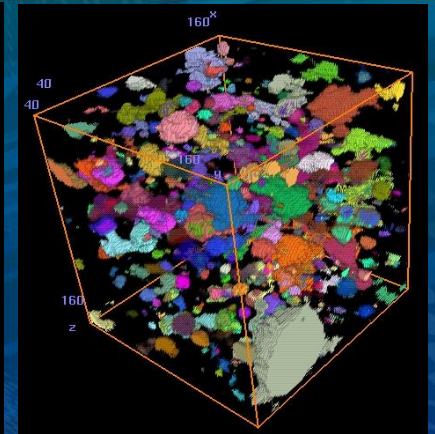






30 Years of Imaging and Modeling Building Materials at NBS/NIST: From PIXAR to MICROCHAR

Dale P. Bentz (dale.bentz@nist.gov)

Imaging of Construction Materials and Geomaterials

July 7, 2016

NIST
National Institute of
Standards and Technology
U.S. Department of Commerce

Back in the day, the day being 1985 (year of Back to the Future I)

NBS had built a brand new state-of-the-art imaging system for building materials

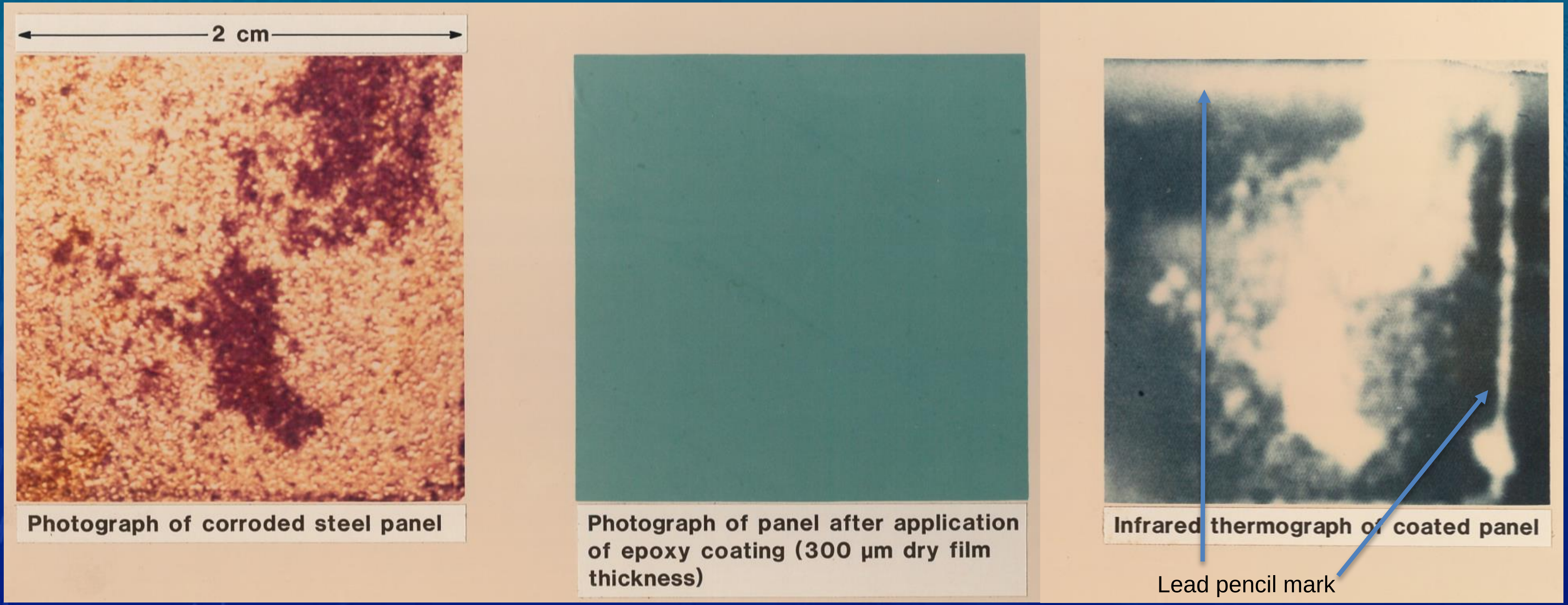
Hardware – Intel 8086 with four IP-512 Imaging Technology imaging boards

home built joystick, color selector, video and **infrared** (2 to 5 µm) cameras

Software – Fortran and assembly language with scripting (batch) capabilities

Infrared Thermography - 1

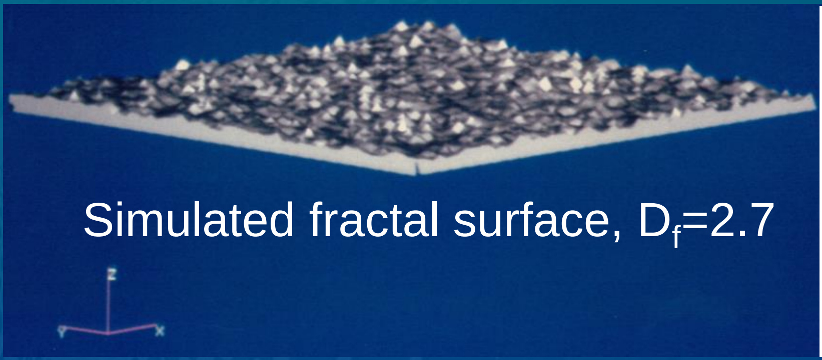
Detection of corrosion under opaque (organic) coatings

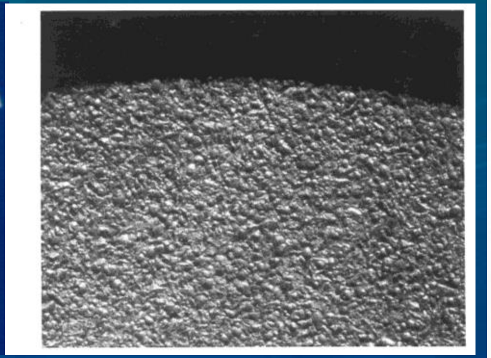


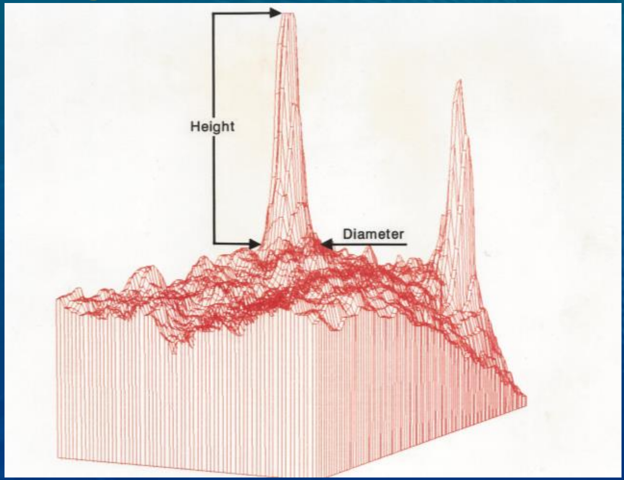
Emissivity of iron oxides (rust) is higher than that of base metal, so that when heated above ambient temperature, corrosion appears bright in infrared image even when viewed under a visually opaque coating of 300 µm or more in thickness

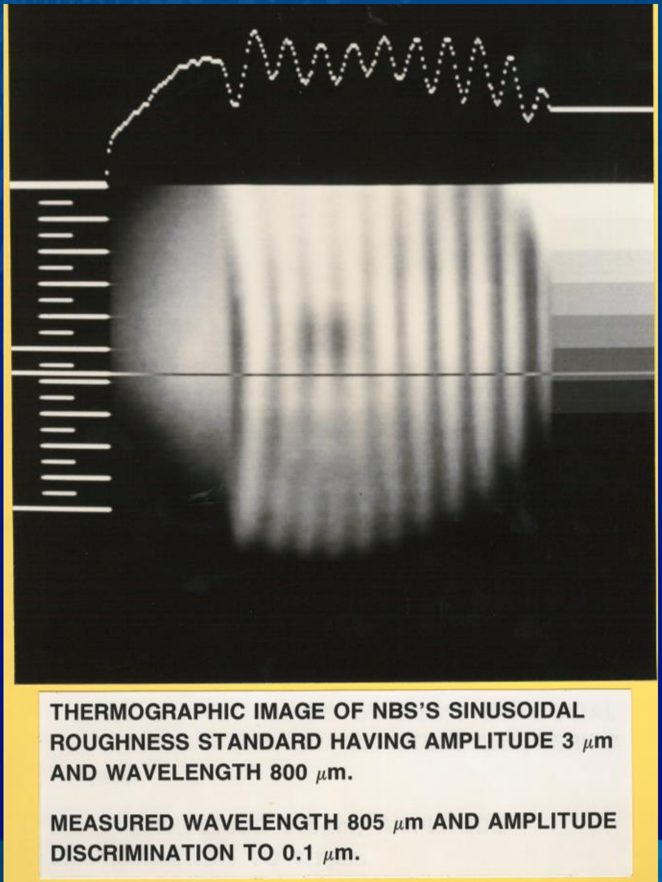
Still in use for field evaluation of steel in bridges and other infrastructure in 2016!

Infrared Thermography - 2





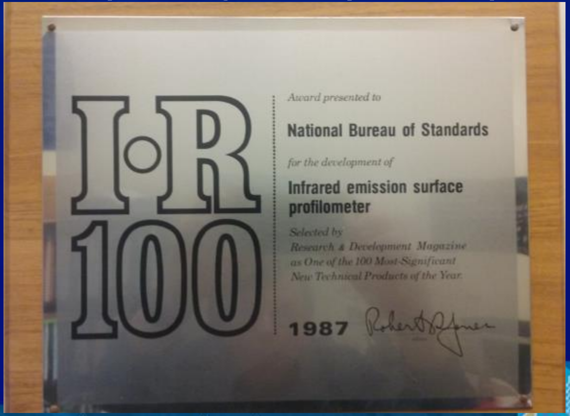




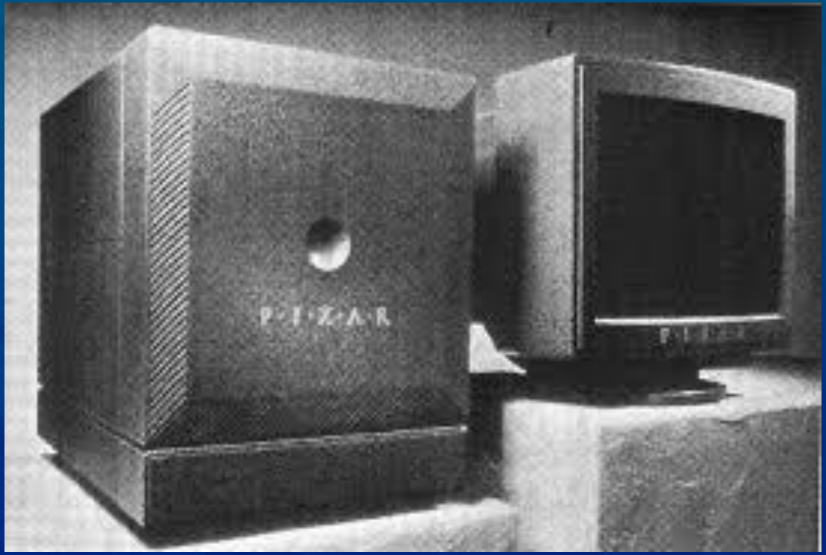
Fractal-based Description of the Roughness of Blasted Steel Panels

Characterization of Cylindrical Holes in Metallic Substrates via Their Infrared Emission Patterns (theory of Sparrow)

Holes/valleys as cavity emitters
$2 \le D_f \le 3$



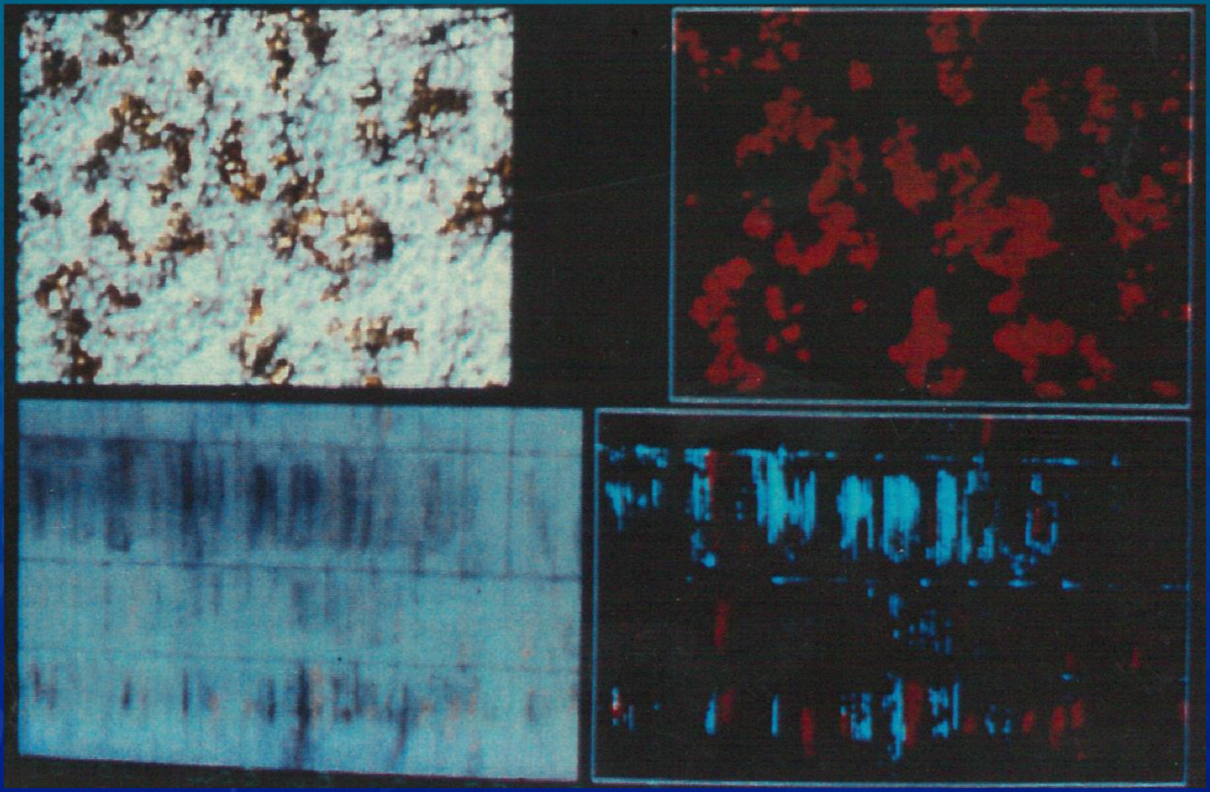
Before *Finding Dory*, before *Brave*, before *Up*, before *Cars*, before *Toy Story*, there was a **PIXAR Imaging Computer**



Released in 1986 by the Graphics Group (computer division of Lucasfilm -> PIXAR)
Fewer than 300 units sold; NBS bought one of these
Without the PIXAR, the NBS/NIST cement hydration modeling program(s) would likely not have been possible

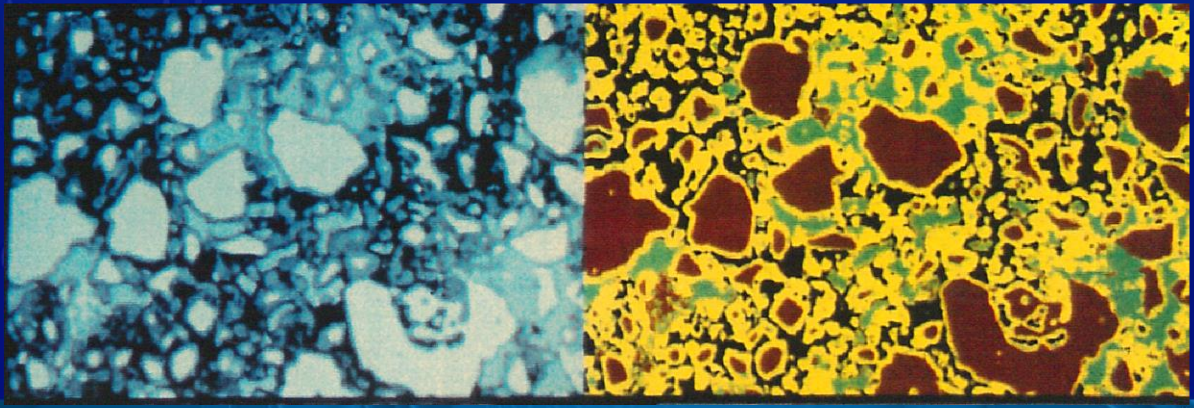
PIXAR Imaging Applications at NBS

Originally connected to a color video camera and used to analyze defects in building materials and structures



Rust on a steel panel - threshold selected based on red color signal

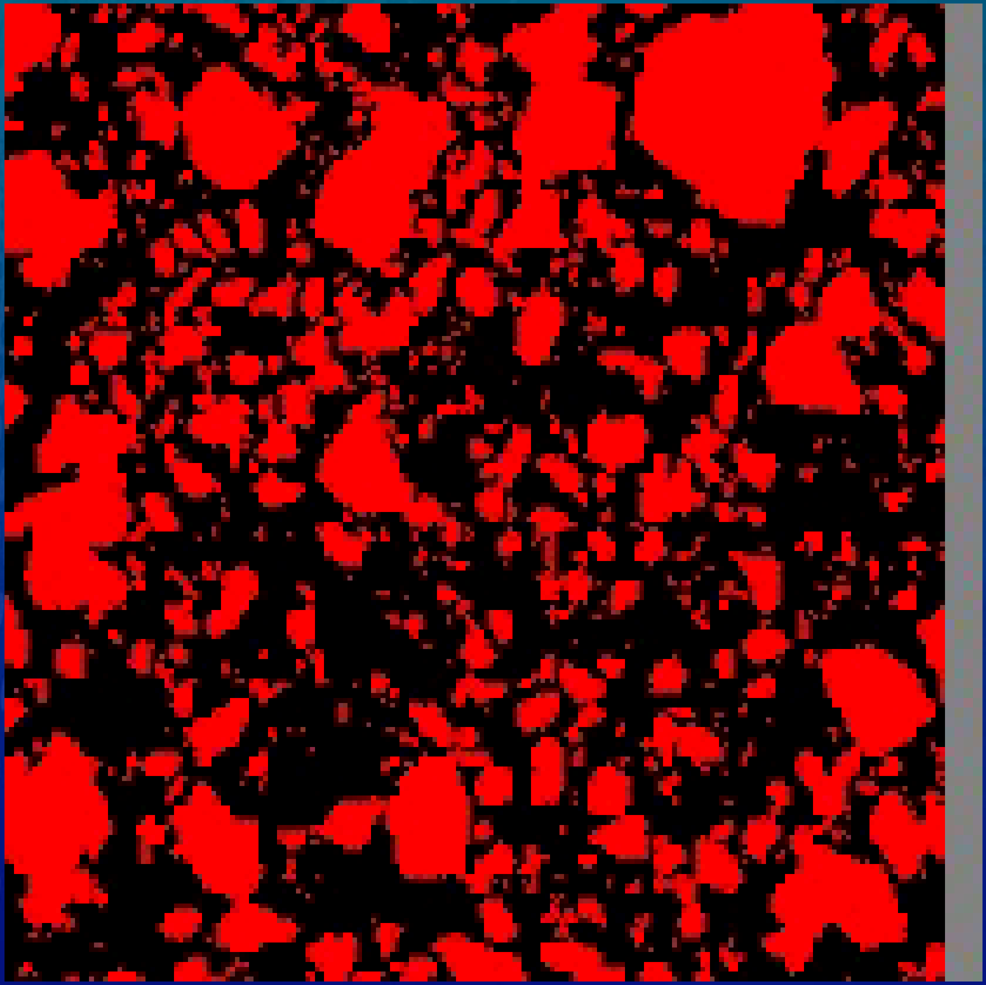
Debonding and corrosion on a coated steel building- thresholds selected based on RGB color signals



But, we were also processing SEM images of cement microstructure and that led to….

SEM image of hydrated C_3S paste courtesy of Paul Stutzman

C_3S Hydration Model



2-D C_3S model with real shape particles running on NBS' PIXAR imaging computer

Before THAMES…..

Before CEMHYD3D …….
versions 3, 2, and 1 (VCCTL)

There was……
HYDRA3D and HYDRA2D

One phase: tricalcium silicate (C_3S)

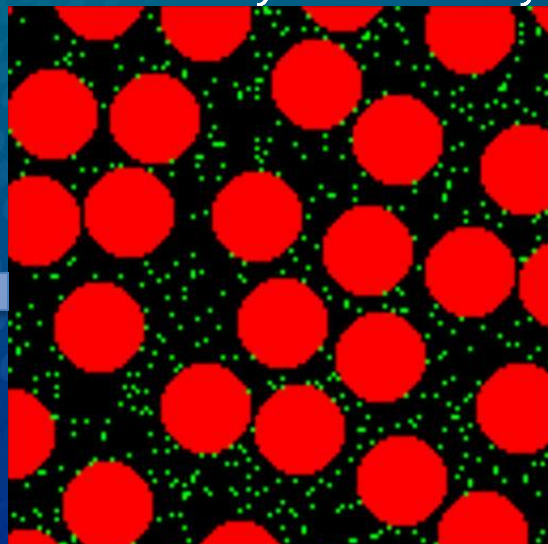
One reaction:

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

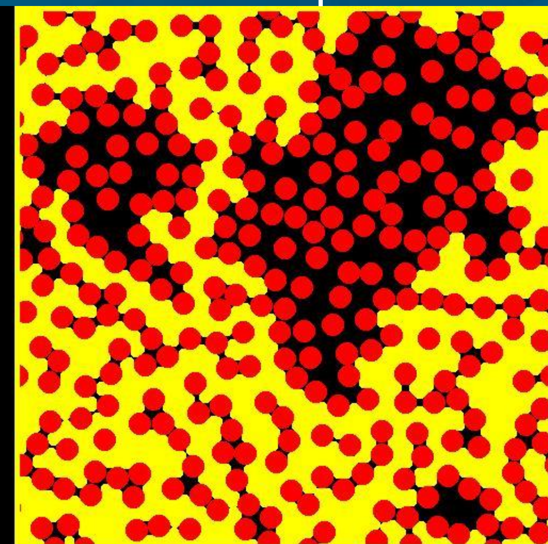
Cement chemistry notation:
C=CaO, S=SiO_2, H=H_2O

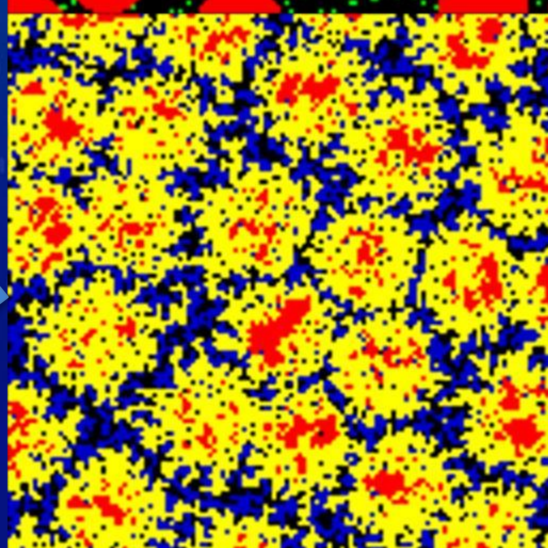
Oh what fun it was to have a PIXAR - 1

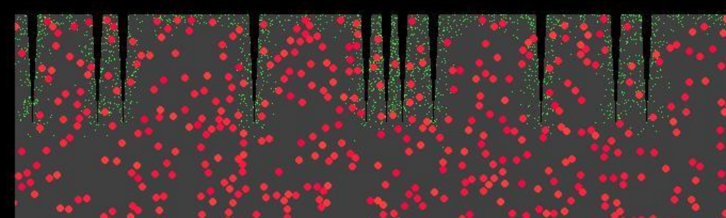
Blind ant/random walkers for diffusivity/conductivity



Two-dimensional mercury intrusion/desorption simulation







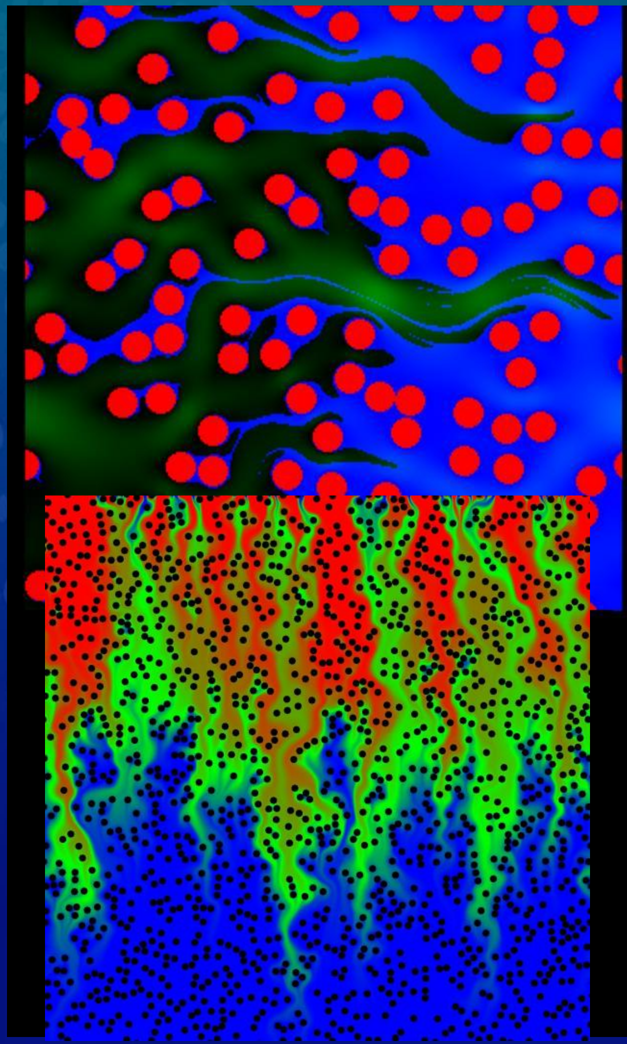
Simulation of diffusion in pigmented coatings

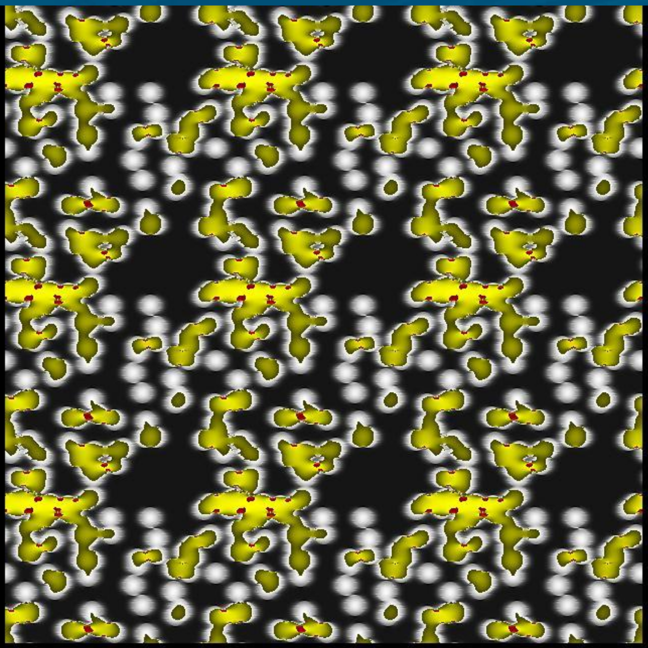
C_3S hydration model

Oh what fun it was to have a PIXAR - 2

Simulation of dispersion during flow (Martys)

Visualization of stresses from finite element model (Garboczi)

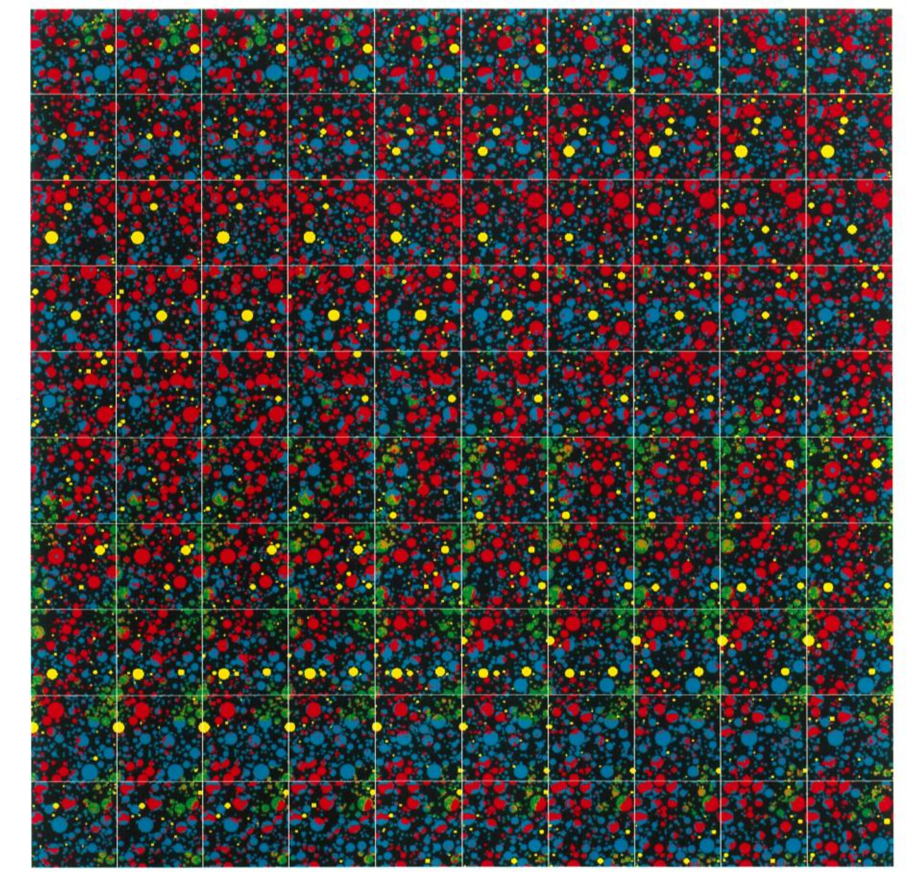




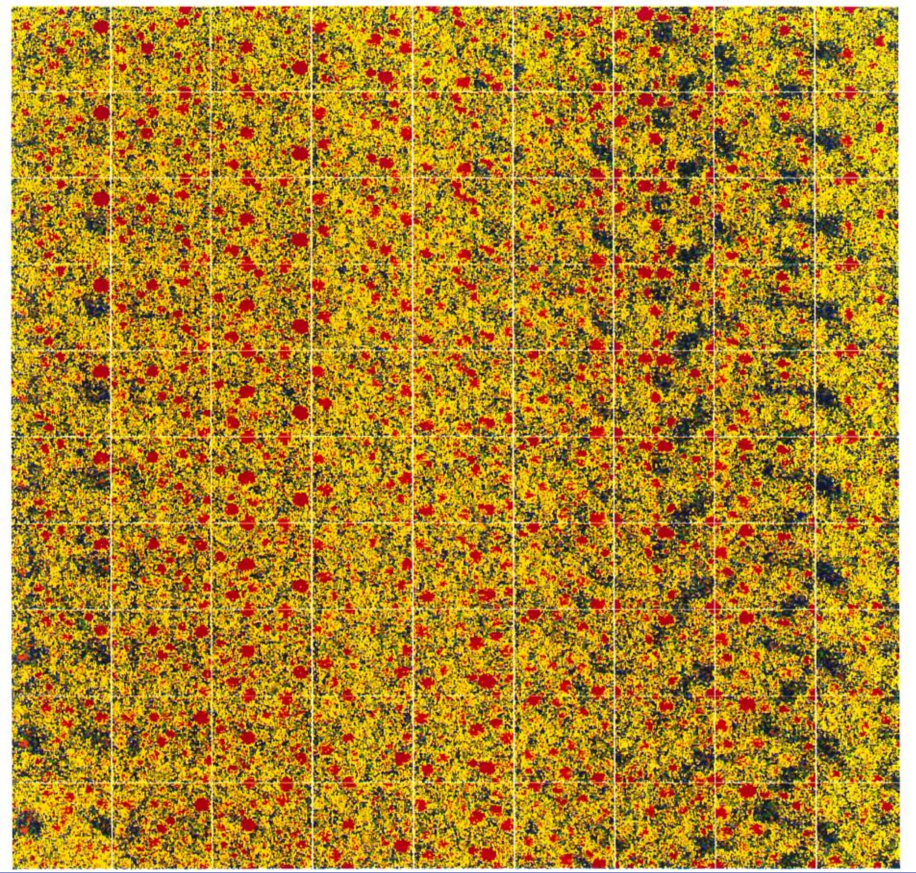
Two-dimensional simulation of sintering (Pimienta)

And we were by no means limited to 2-D!

Mapping a 3-D 100x100x100 microstructure into a 2-D 1000x1000 image



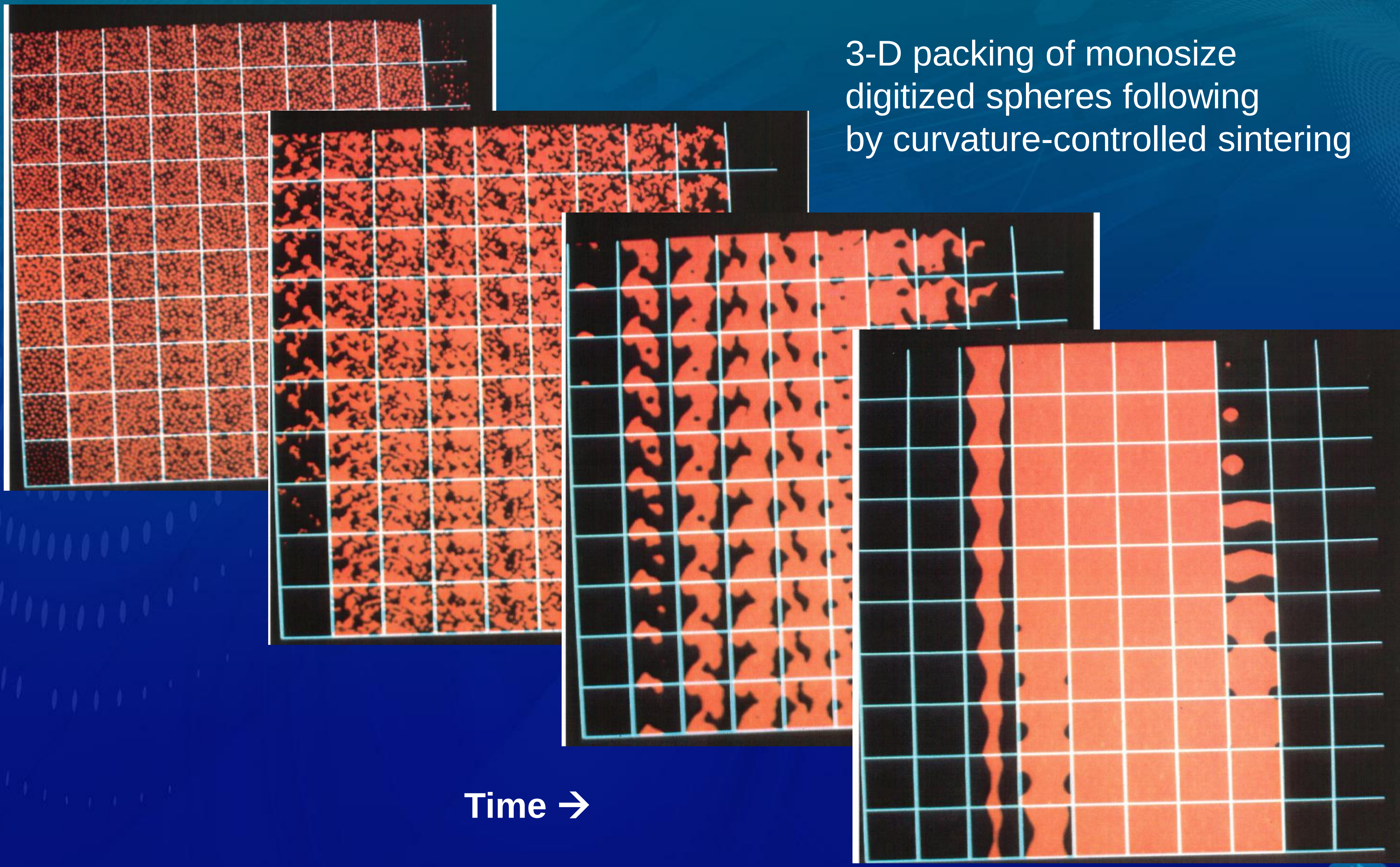
100x100x100 multi-phase cement particle microstructure



Hydrated microstructure after 50 cycles
Porosity=16 %, Deg. of hyd.=0.67

3-D Sintering Model

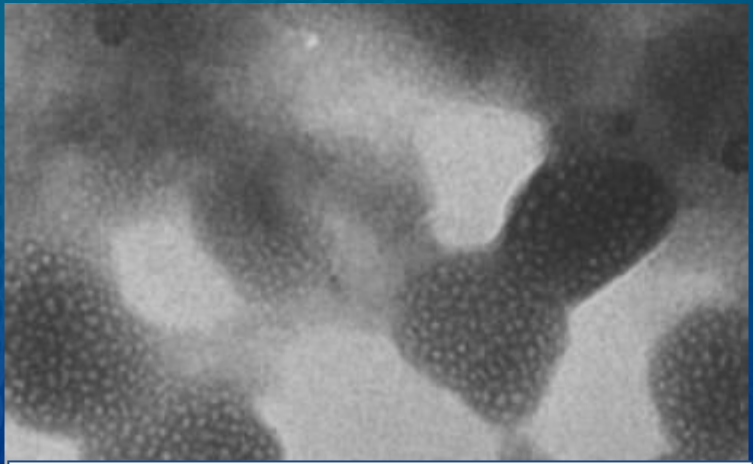
3-D packing of monosize
digitized spheres following
by curvature-controlled sintering



Time →

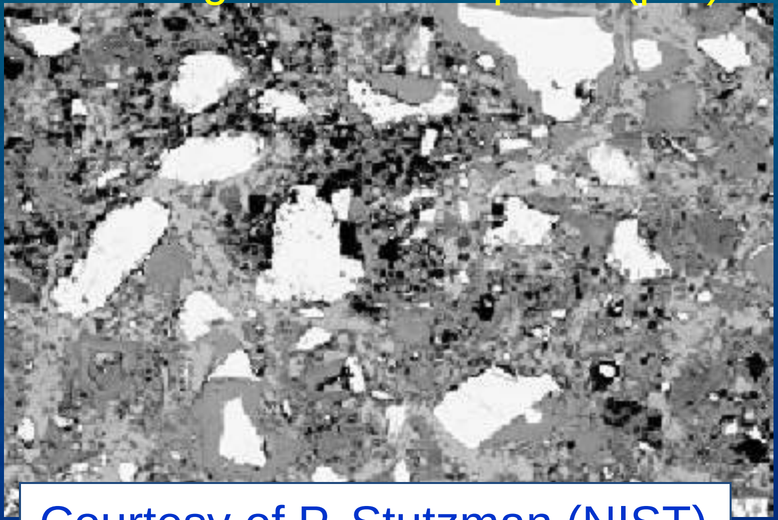
Multi-Scale Concrete Microstructure

TEM image of C-S-H (nm)

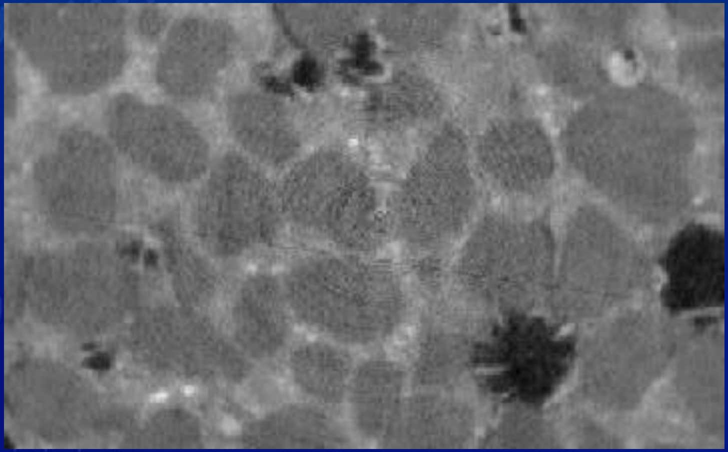


Courtesy of R. Maggion (France)

SEM image of cement paste (µm)



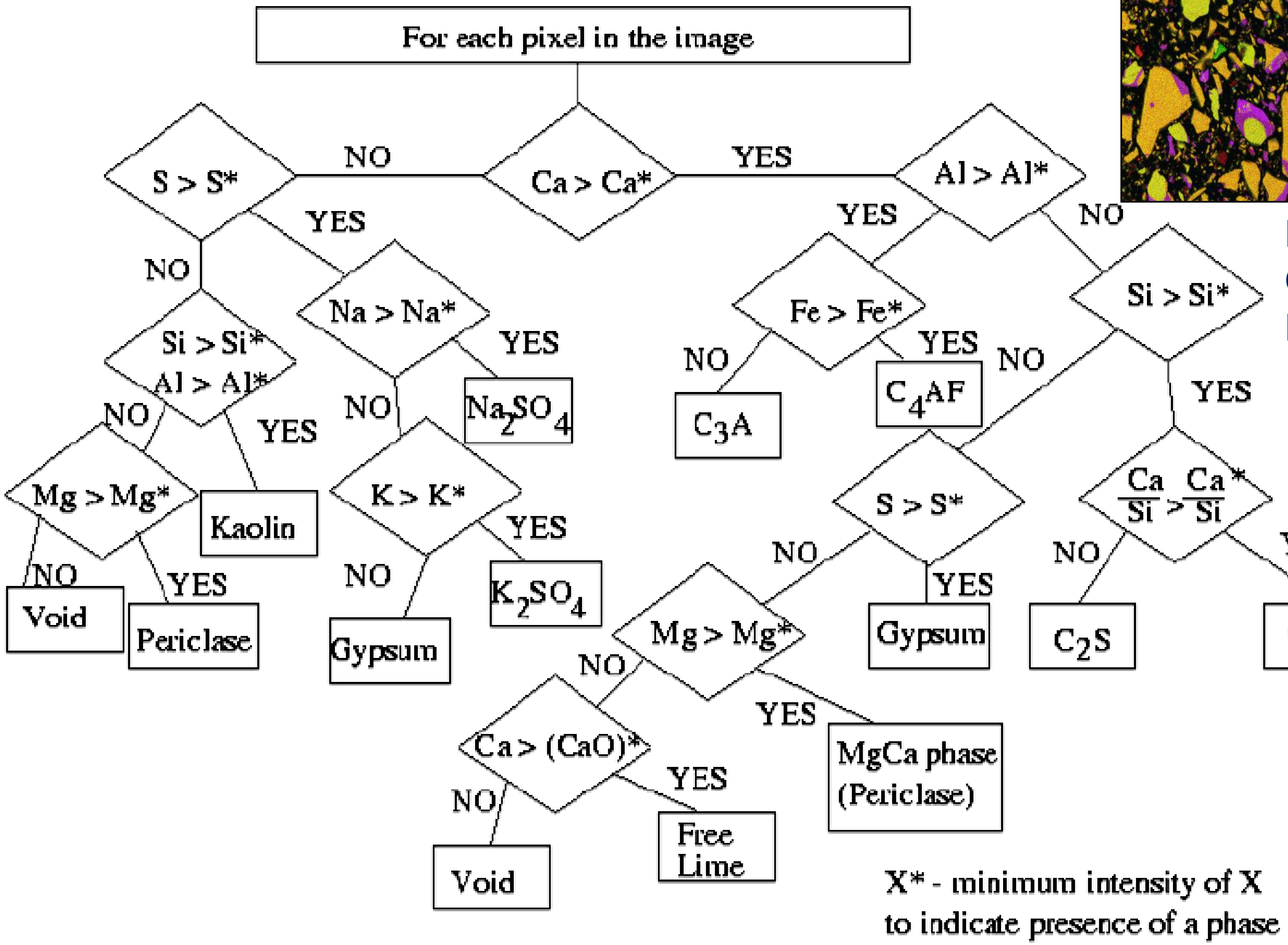
Courtesy of P. Stutzman (NIST)

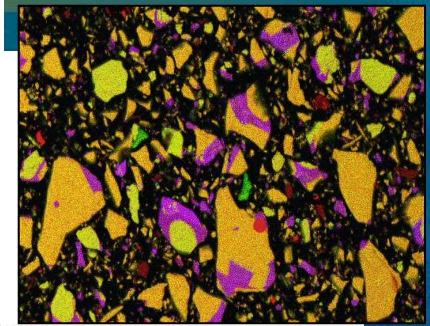


Microtomography of mortar (mm)

Optical microscopy of concrete (cm)

Imaging of Cement Particle Microstructure

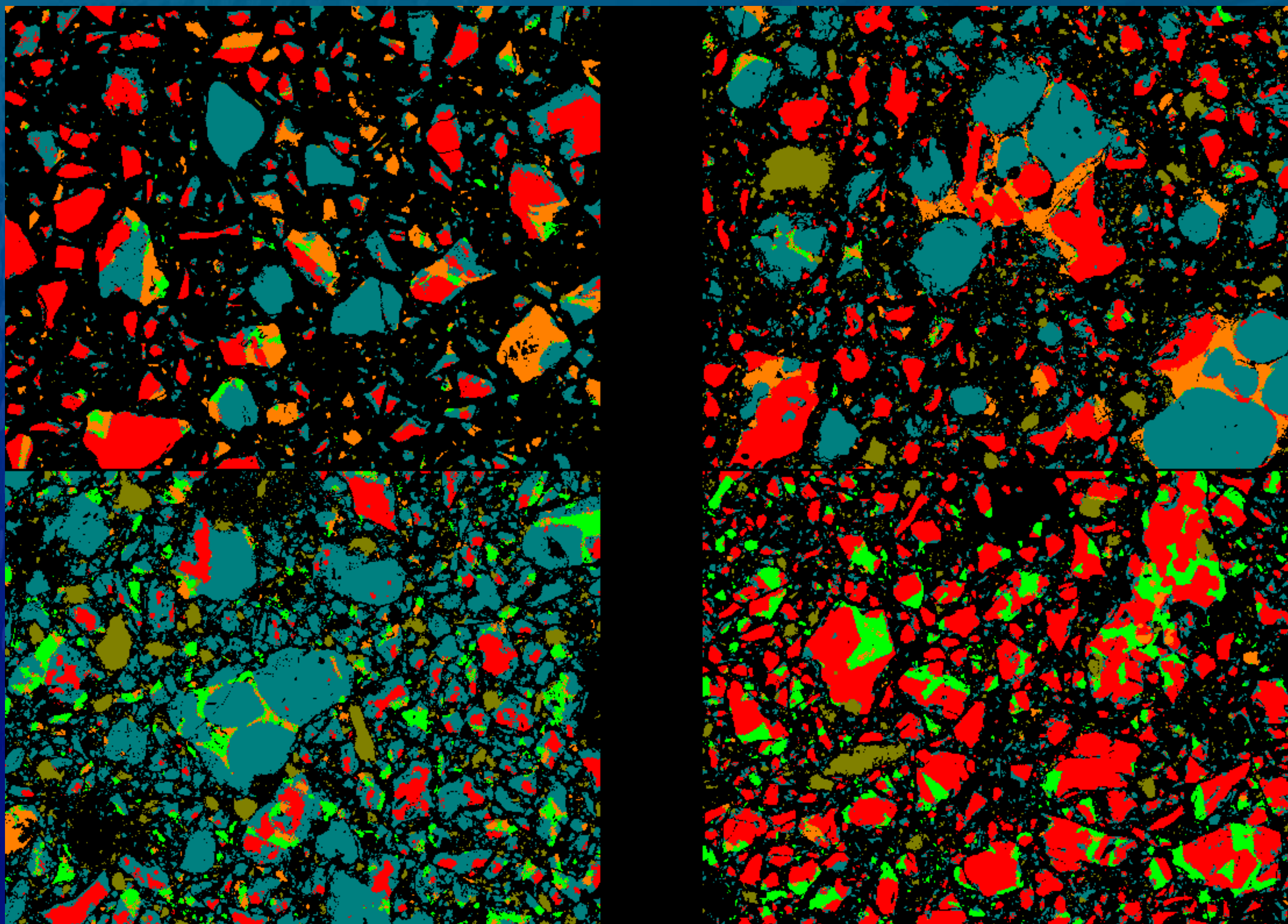




R=Ca
G=Si
B=Al

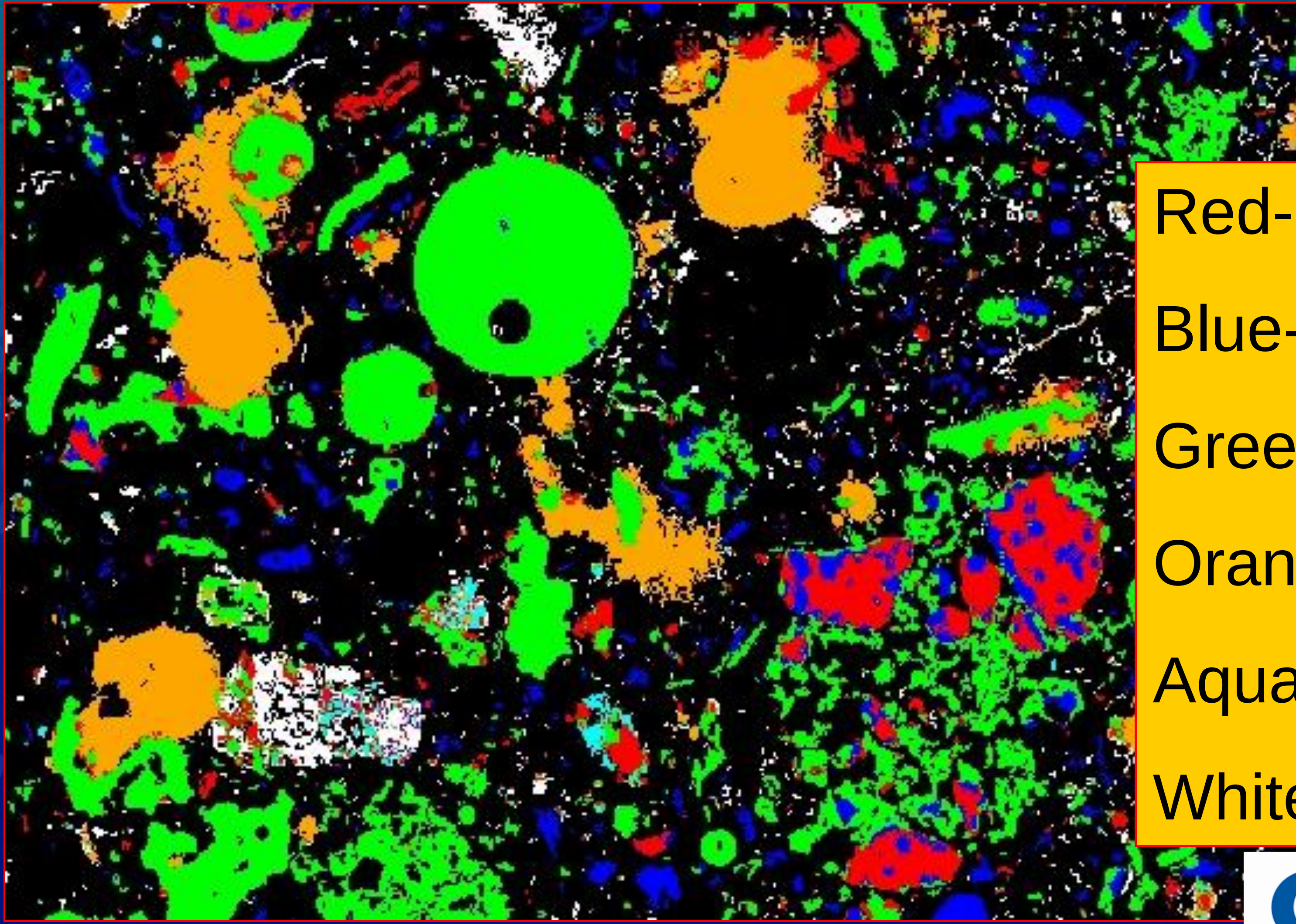
Decision tree for the segmentation of the image set (9) into its component phases
2015 release of MicroChar code by Jeff Bullard, NIST Technical Note 1876

Characterization of Cement Powder via SEM/X-ray Imaging



Red- C_3S
Blue- C_2S
Green – C_3A
Orange – C_4AF
Olive - Gypsum

SEM/X-ray **Characterization** of Fly Ash (Municipal Waste Fly Ash from France)



Red- S

Blue- AS

Green- CAS_2

Orange- $CaCl_2$

Aqua- $CaSO_4$

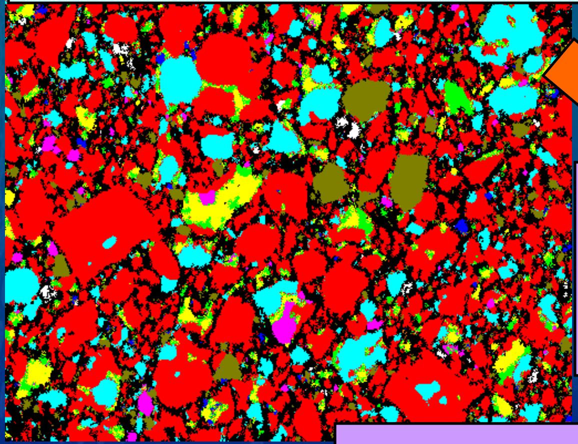
White- inert

Collaborators: Sébastien Rémond and Pierre Pimienta

CSTB le futur en construction

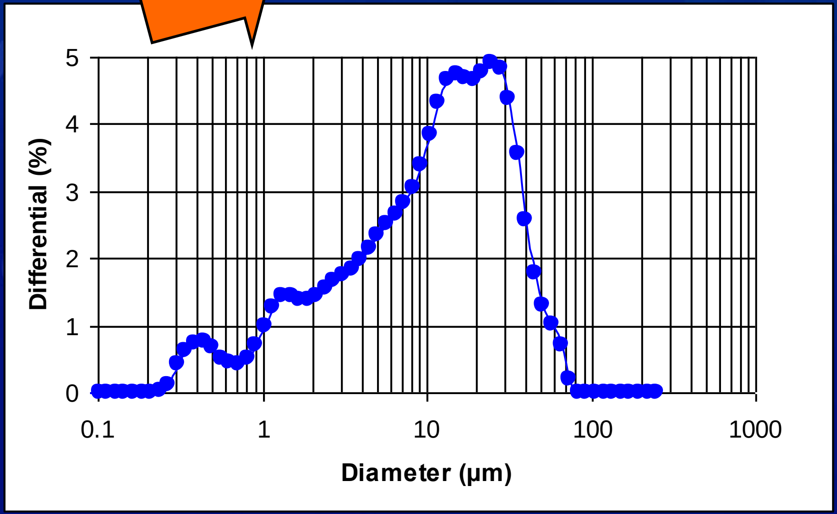
3-D Starting Microstructures for Hydration Models

Segmented SEM/X-ray Image

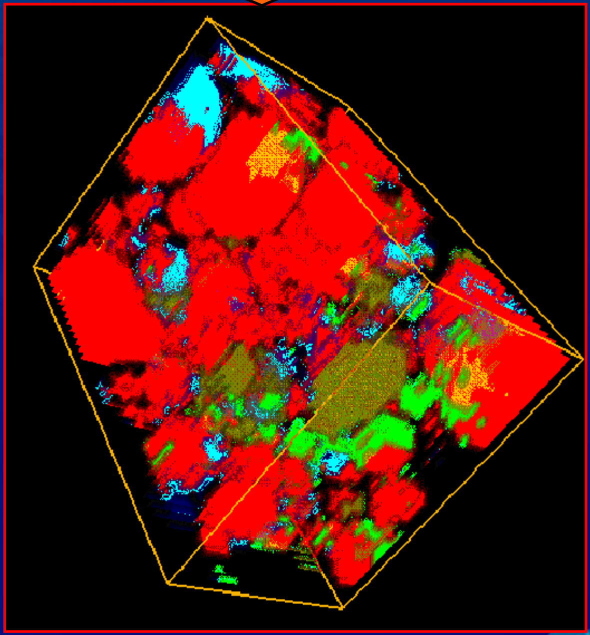


Phase volume fraction
Surface area fractions
Correlation functions

Particle Size Distribution



3-D reconstruction with flocculation, random placement, or dispersion



ftp://ftp.nist.gov/pub/bfrl/bentz/CEMHYD3D/

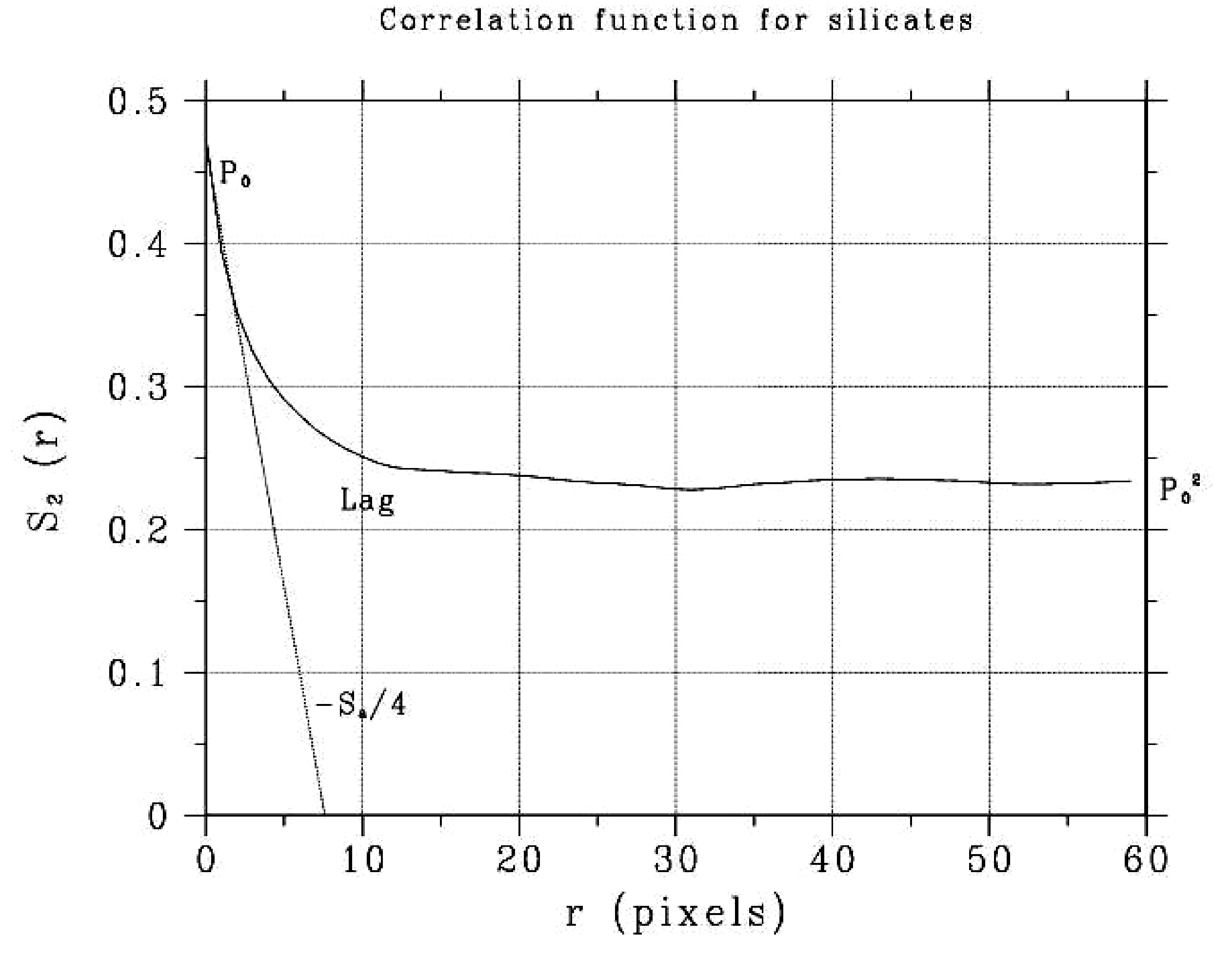
Autocorrelation function

- Measures degree of correlation within a phase (or phases) within a microstructure

$$S_r(\tau) = \int_{-\infty}^{\infty} f(t)*f(t+\tau)dt$$

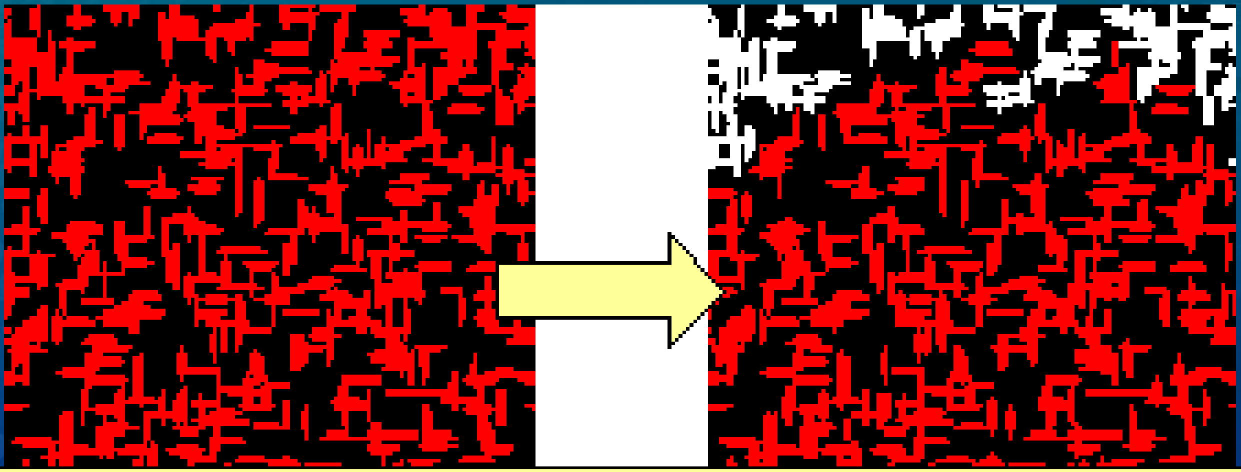
- Every function (image, microstructure) has a unique autocorrelation function, but the converse is not true.
- Equivalent to overlaying a displaced image on an original and observing the common porosity (or phase(s) of interest) for various displacements

- Can also be used to compare real and model microstructures (C&CR, 2006)
- Merci á Daniel Quenard (CSTB) for introducing NBS to correlation functions (circa 1987)

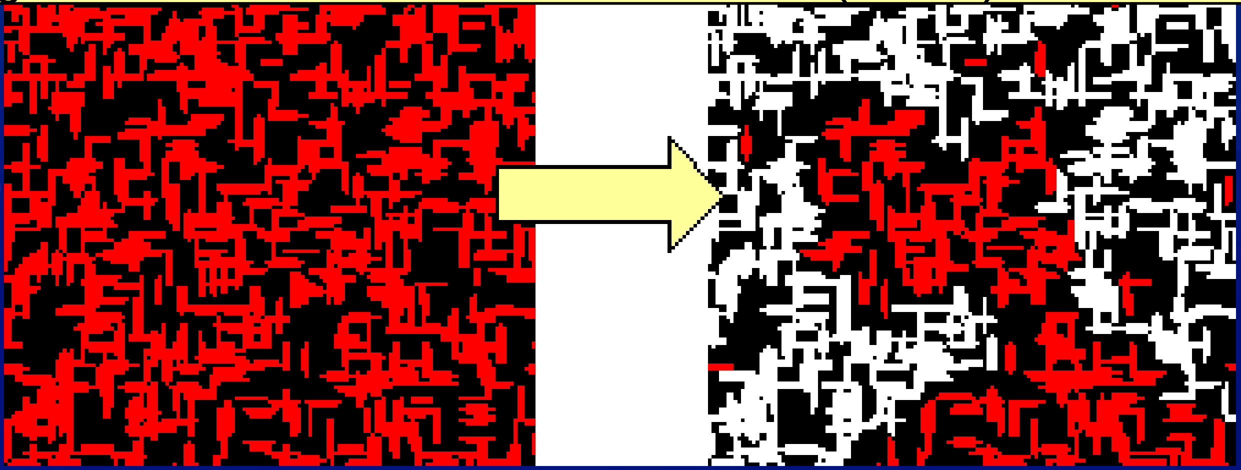


P_0 = phase(s) fraction, r = displacement, S_a = surface area

Percolation



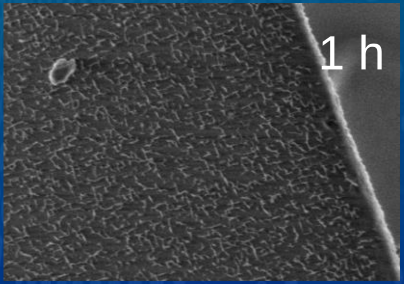
- Connectivity of a phase(s) within a microstructure
- Capillary pores (transport), solids (setting), etc.
- 2-D and 3-D burning algorithms for digital images (NIST Visible Cement Data Set)
- Overlap algorithms in the 3-D hard core/soft shell (HCSS) model



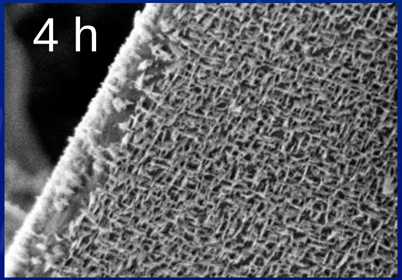
Fine limestone and setting (percolation)

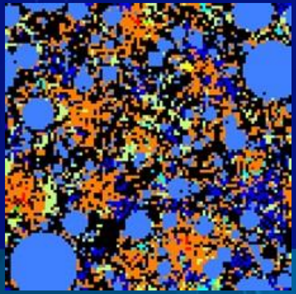
Fine limestone particles are incorporated into the network of percolated particles responsible for setting, due to precipitation/ growth of cement hydration products on their surfaces (thus, higher surface area is more effective)

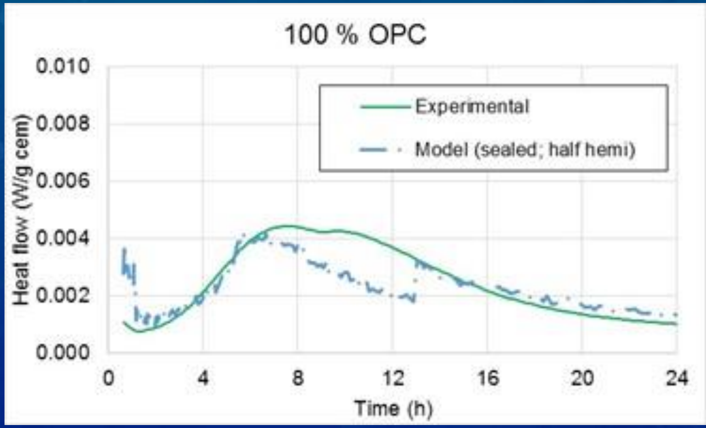
CEMHYD3D v3.0 hydration/setting simulations support this hypothesis

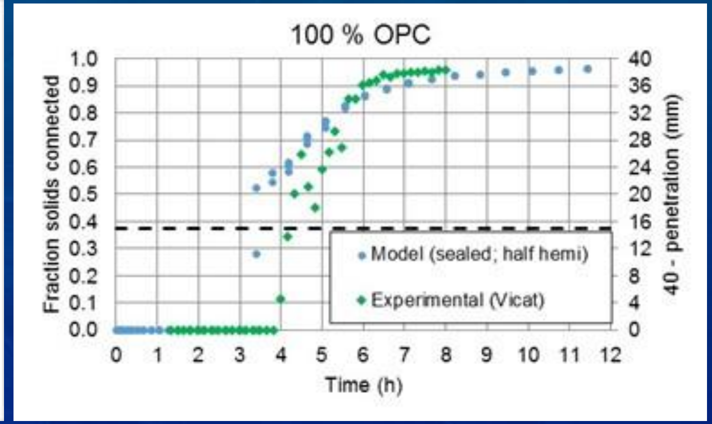


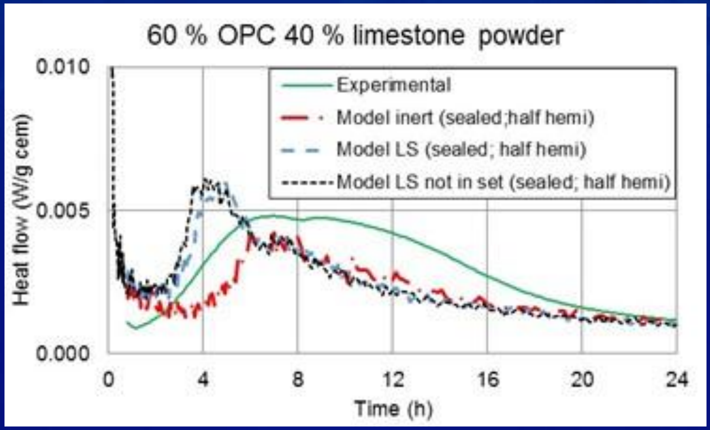
Images courtesy of Dr. Tai Sato (NRC- Canada)

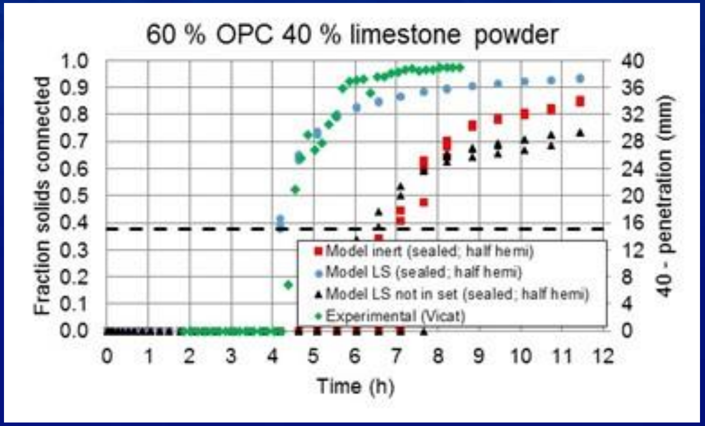










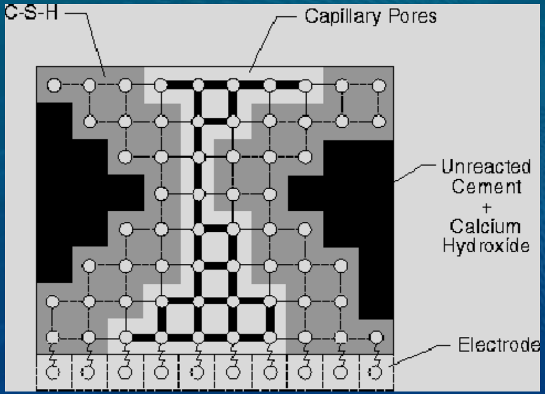


Multi-Scale Modeling of Conductivity and Diffusivity

Cement paste level

Resistance analogy for CEMHYD3D or other microstructures

Diffusivity as a function of *w/cm*, hydration, additives

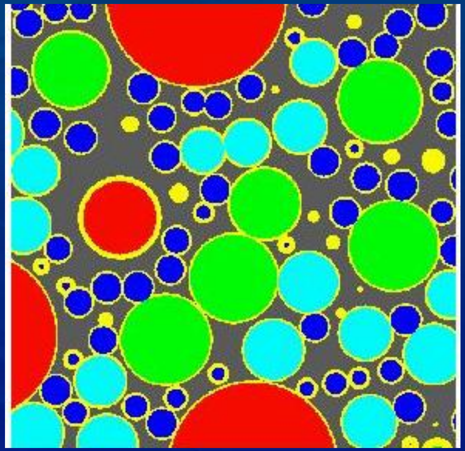


Concrete Level

Random walkers with clocks in the HCSS model

$D = \langle r^2/t \rangle * $ (cement paste fraction)

Aggregate volume fraction and ITZ properties are important at this scale



Virtual Test Methods

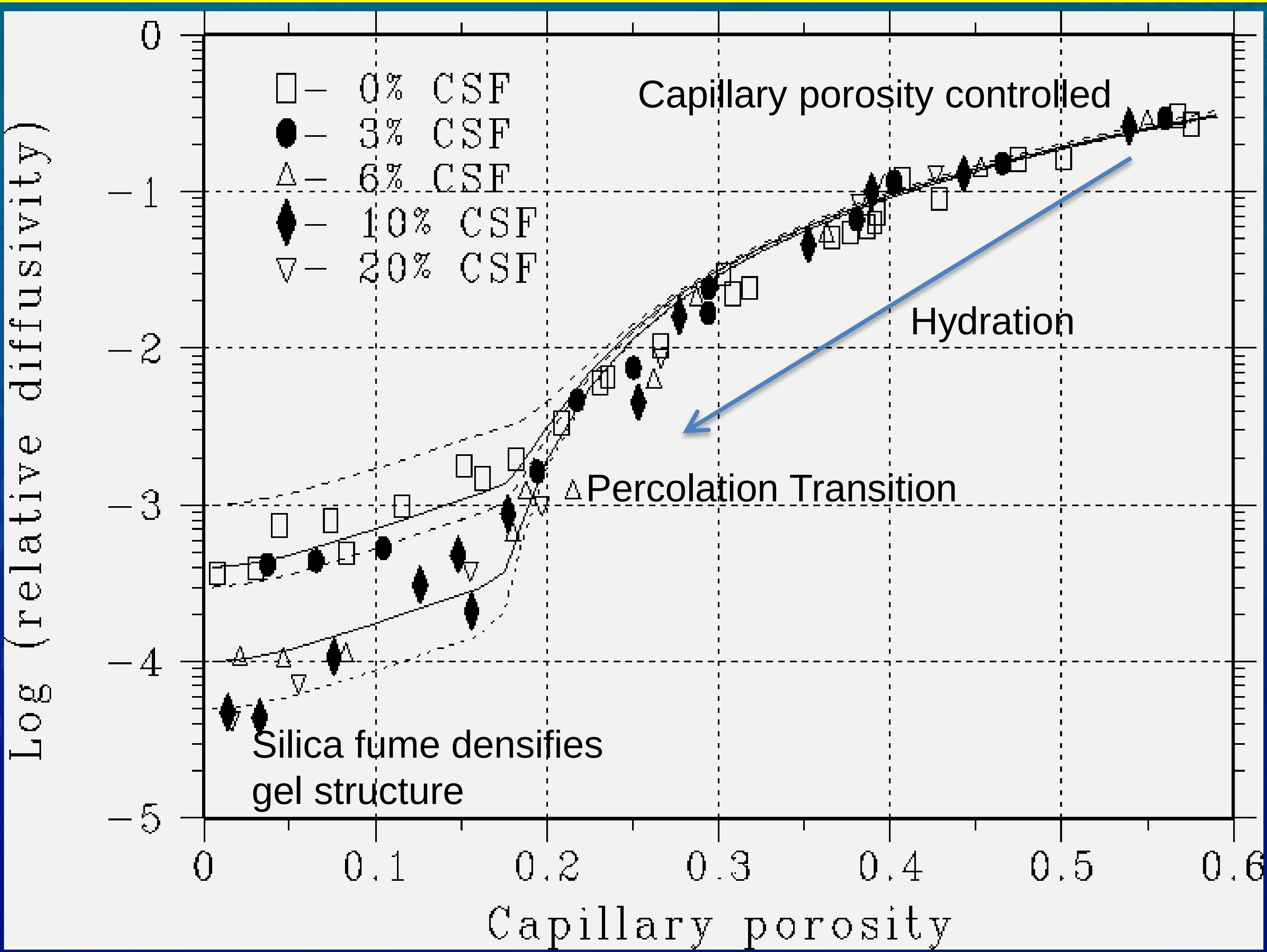
Rapid Chloride Permeability Virtual Test

http://concrete.nist.gov/VirtualRCPT.html

Concrete Electrical Conductivity Test

http://concrete.nist.gov/Concreteconductivity.html

Model Diffusivity vs. Capillary Porosity with the Addition of Silica Fume



Bentz, D.P., Jensen, O.M., Coats, A.M. and Glasser, F.P., "Influence of Silica Fume on Diffusivity in Cement-Based Materials. I. Experimental and Computer Modeling Studies on Cement Pastes," *Cement and Concrete Research*, **30** 953-962, 2000.

X-ray Absorption Studies of Water Movement

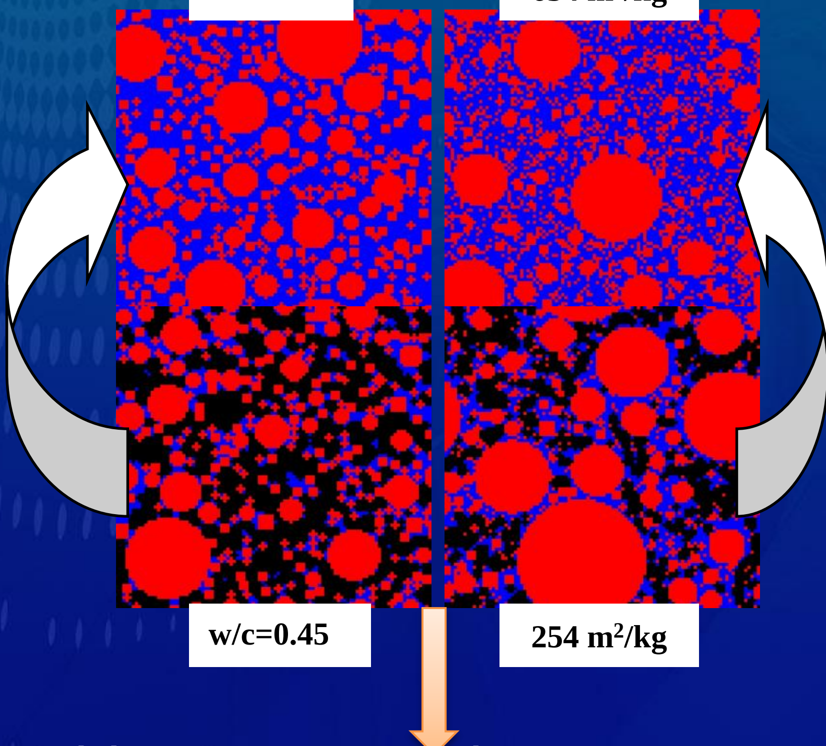
Beer's Law
$I = I_0 \exp(-\gamma t)$

Easy to distinguish air from water (e.g., in pores)

Thin (up to 10 mm) pastes dry out uniformly through their thickness

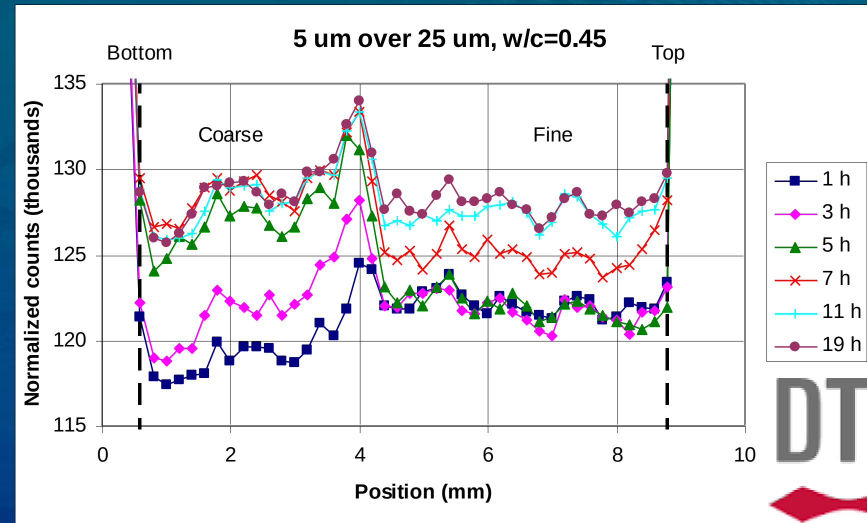
w/c=0.3

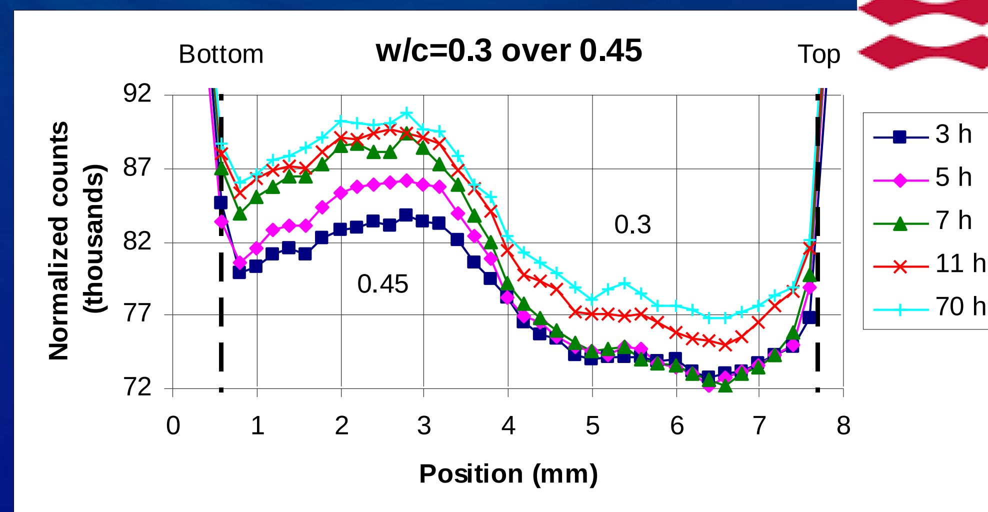
654 m²/kg



w/c=0.45

254 m²/kg

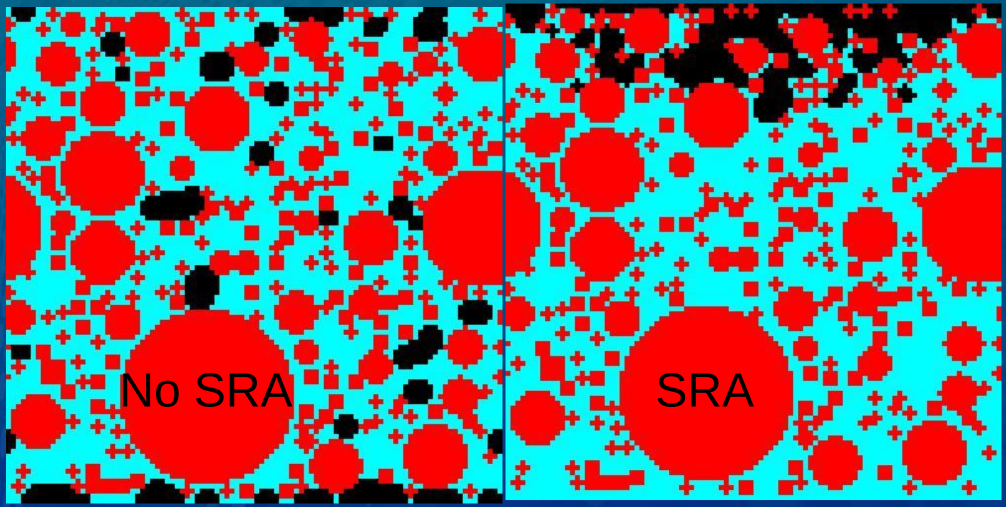


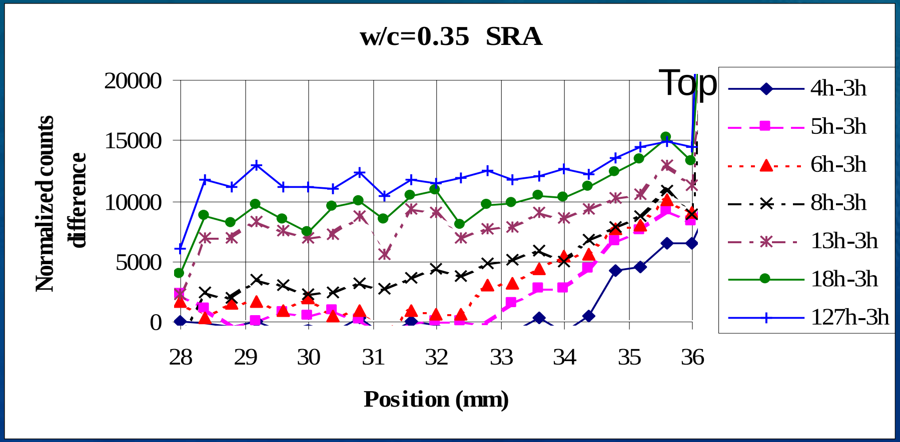


In bilayer composites, water preferentially moves from a coarser pore structure to a finer one, regardless of which one is on top

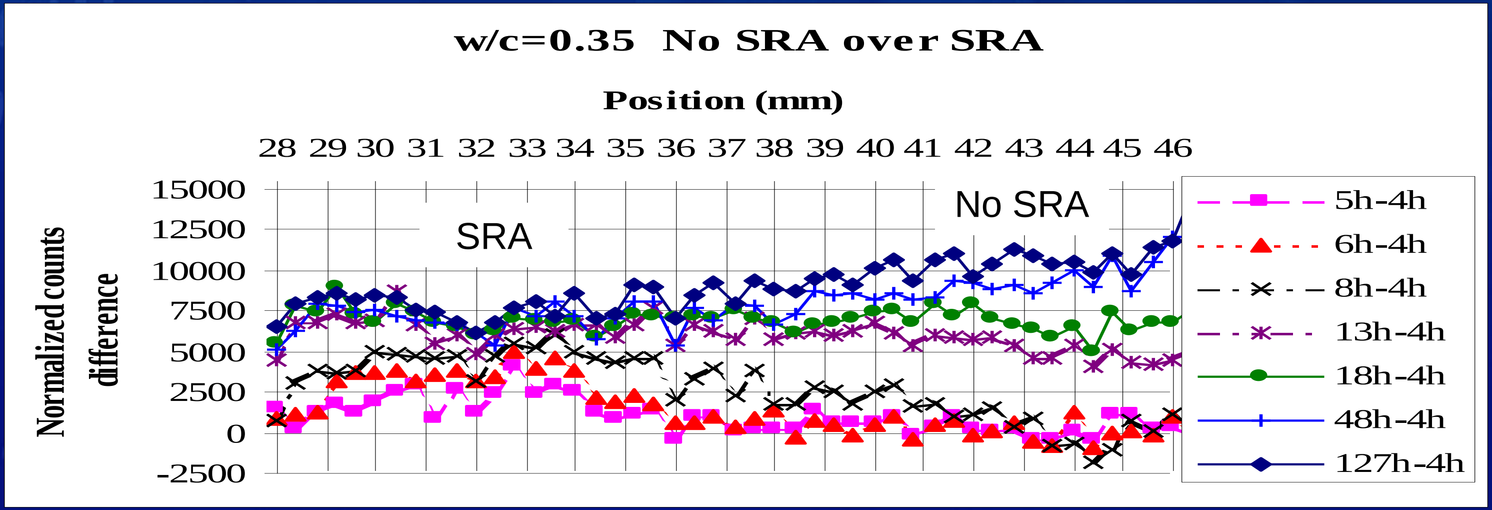
X-ray Absorption Studies of Water Movement

Shrinkage-reducing admixtures – decrease surface tension of pore solution





SRA modifies drying profile as top surface dries out first, while interior initially remains saturated

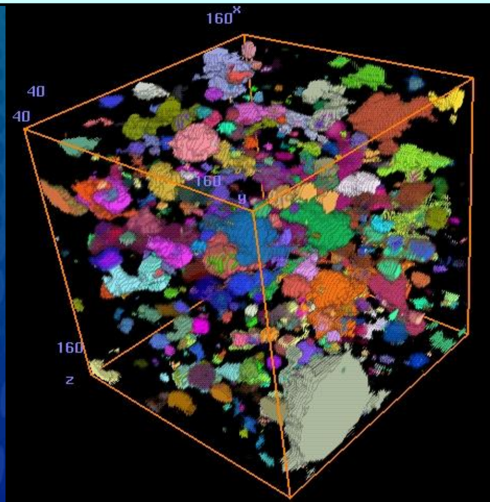


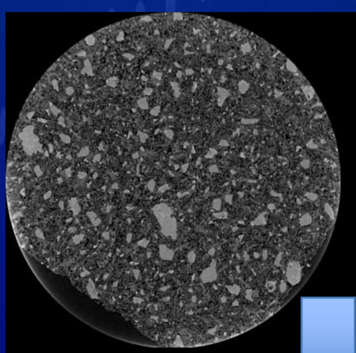
Marangoni Effect – Layer with SRA dries out first regardless of top/bottom position

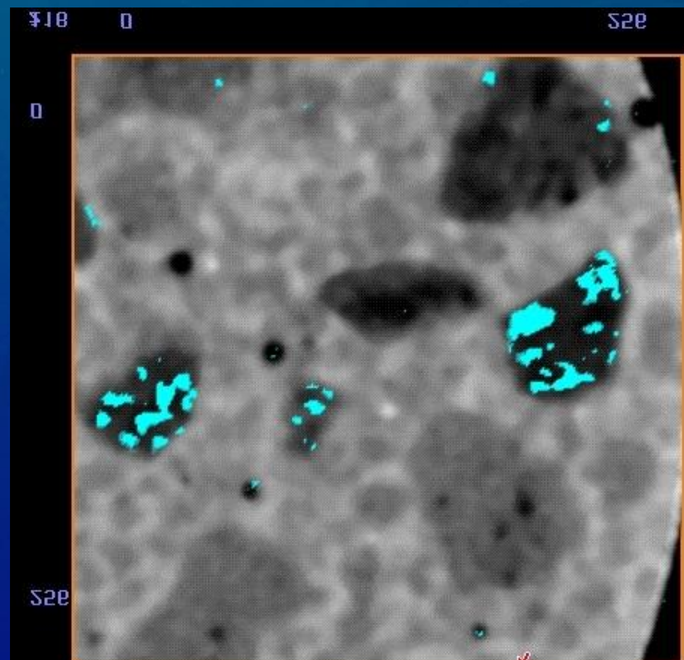
Extension to 3-D: X-ray Microtomography

- Evaluate (changes of) the 3-D microstructure of materials

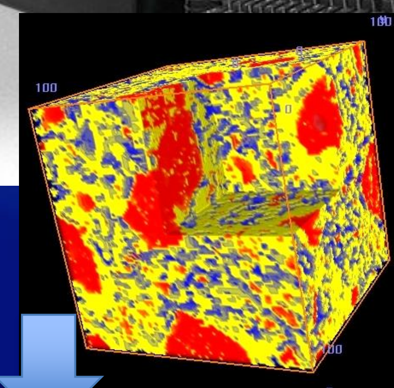
Porosity of a fire resistive material







Movement of water during internal curing



Visible Cement Dataset- http://visiblecement.nist.gov

Visible Cement Data Set

Data collected at European Synchrotron Radiation Facility in Grenoble France, in September 2000 in collaboration with CSTB (Quenard, Vallee, Sallee)

Spatial resolution of about 1 µm (state-of-the-art at that time)

Compiled into web site in 2002 at NIST
visiblecement.nist.gov

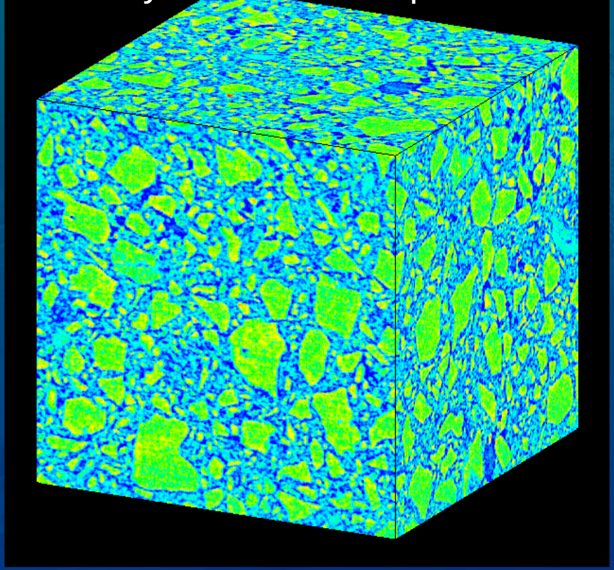
Separate data set pages for:
- hydrated cement paste (*w/c* = 0.3 to 0.45)
  - hydration times to 175 h
- hydrated Plaster of Paris (*w/s* = 1.0)
- porous bricks

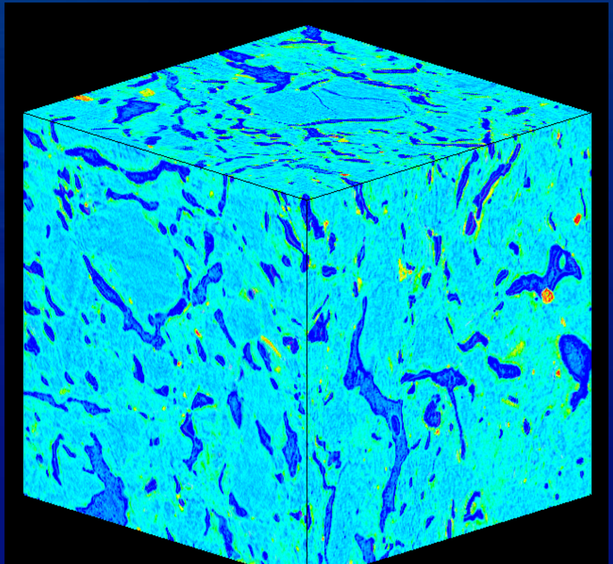
Downloadable programs for processing data sets

Citations and collaborator pages

CSTB
le futur en construction

Hydrated cement paste

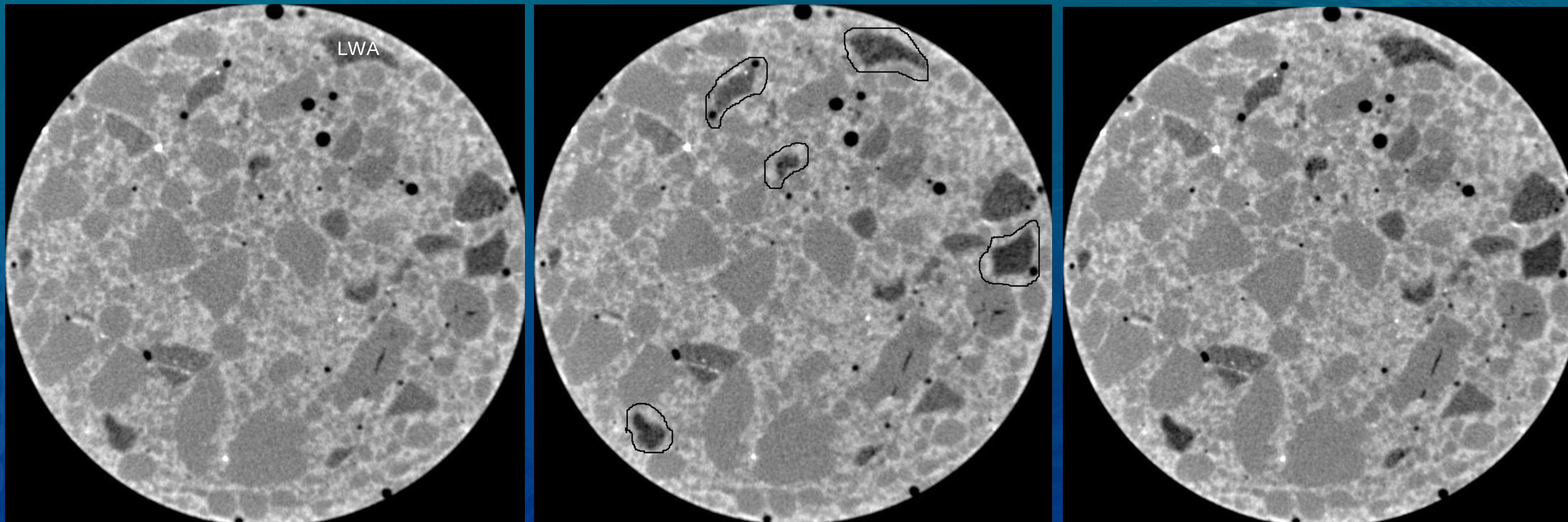




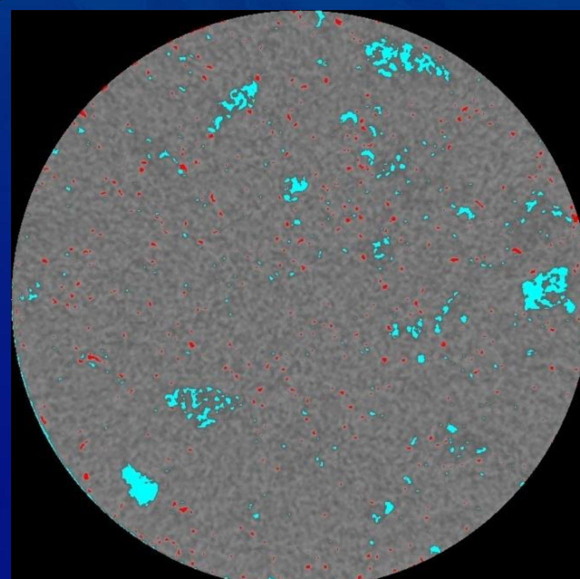
Porous Brick

3-D X-ray microtomography to monitor internal curing in situ

After mixing | 1 d hydration | 2 d hydration



All images are 13 mm by 13 mm

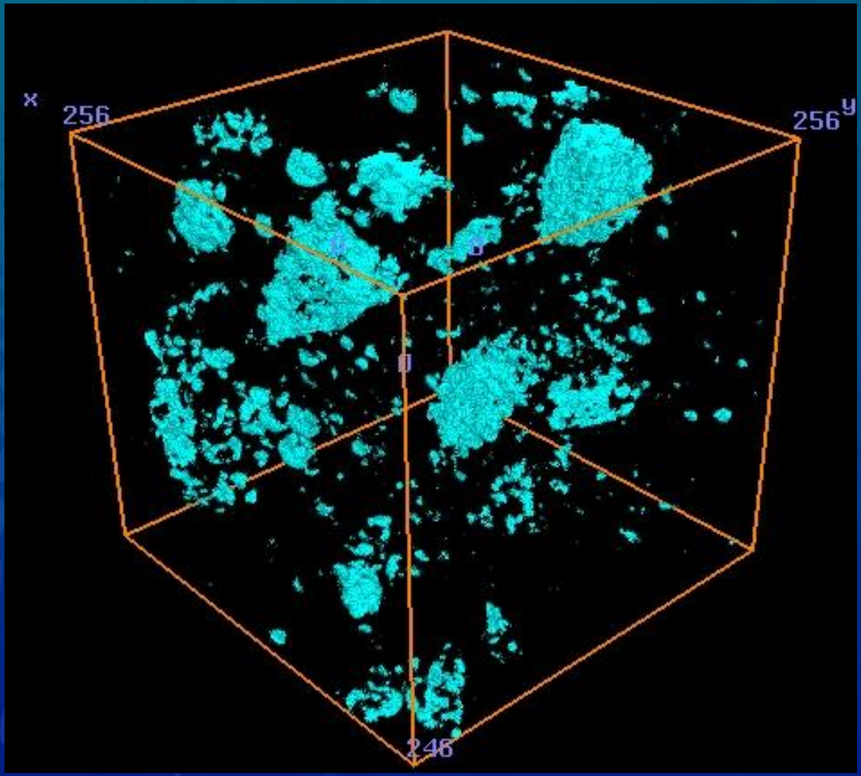


Aqua indicates drying

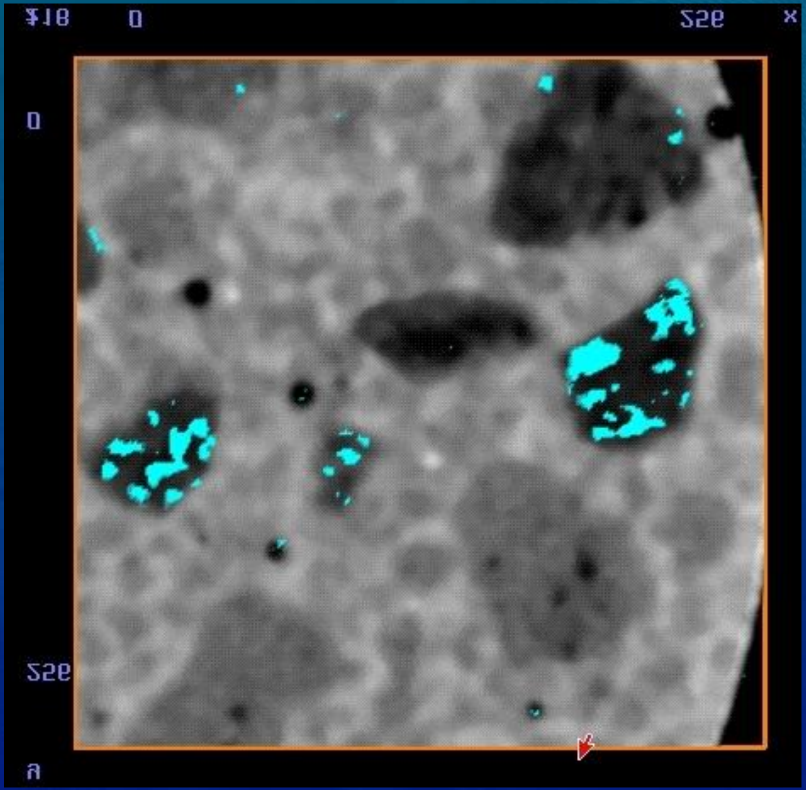
Red indicates wetting

Subtraction: 1 d – after mixing

Three-Dimensional X-ray Microtomography



Three-dimensional subtracted image of 1 d hydration – initial microstructure showing water-filled pores that have emptied during internal curing (4.6 mm on a side)

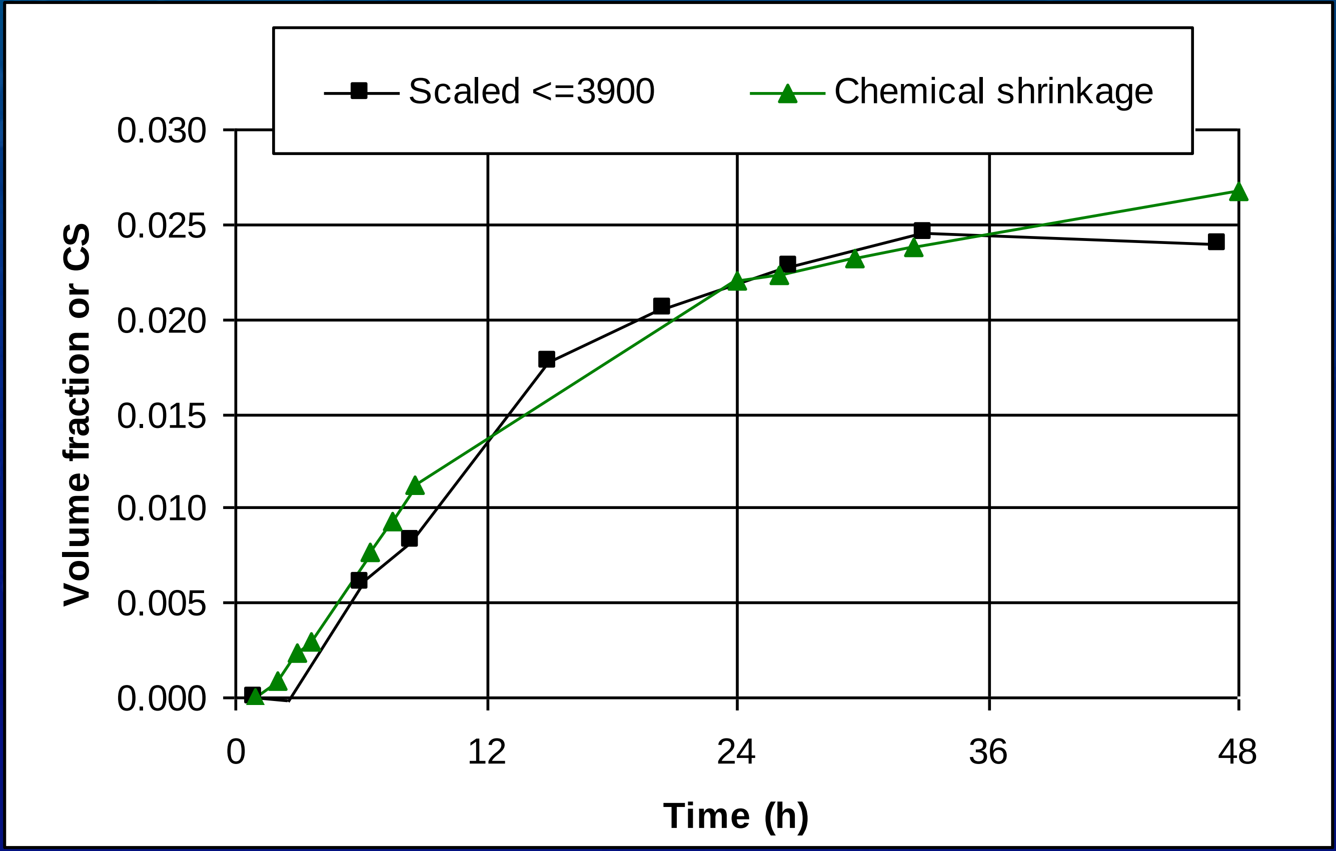


2-D image with water evacuated regions (pores) overlaid on original microstructure (4.6 mm by 4.6 mm)

Three-Dimensional X-ray Microtomography

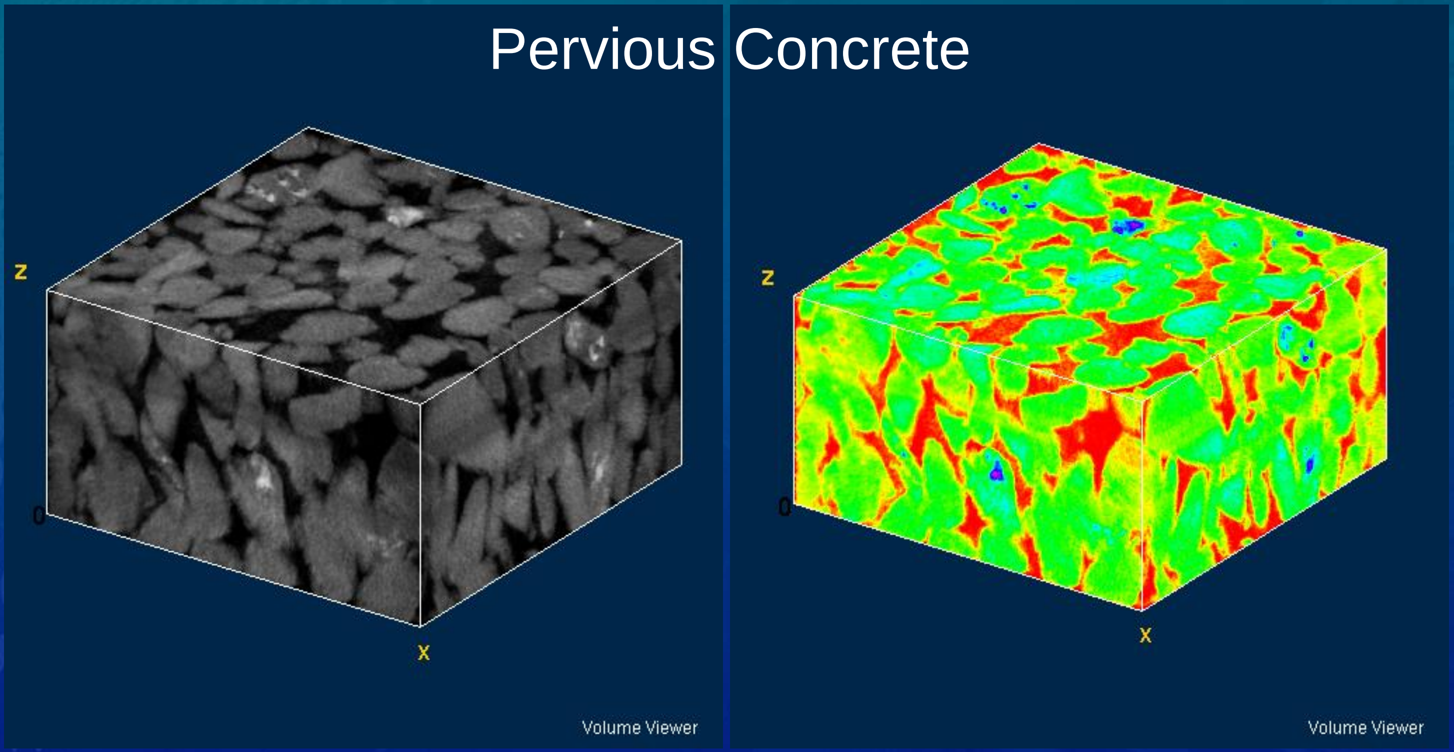
DEMAND==SUPPLY

Empty porosity within LWA from analysis of 3-D microtomography data sets scales “exactly” with measured chemical shrinkage of the cement for first 36 h of curing

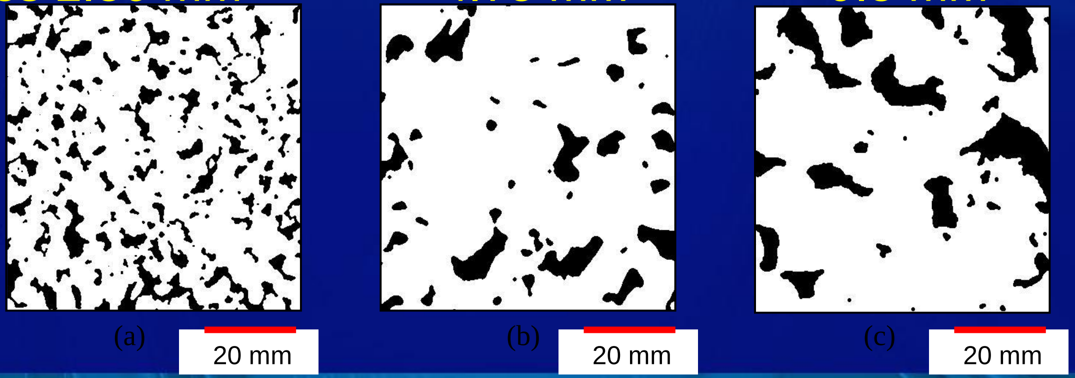


Quantitative analysis

3-D Microstructure via Tomography



Aggregates 2.36 mm   4.75 mm   9.5 mm



Modeling Permeability of Pervious Concrete

- **3-D** Stokes solver code available from NIST at:
  - ftp://ftp.nist.gov/pub/bfrl/bentz/permsolver

Original 2-D Image

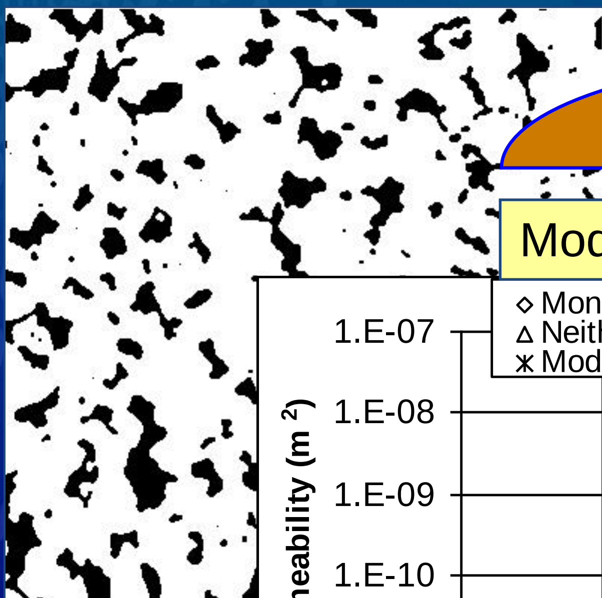
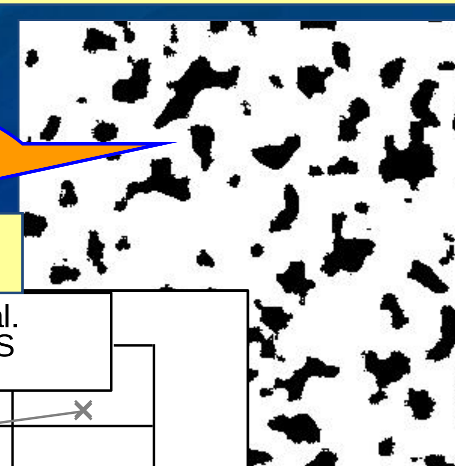
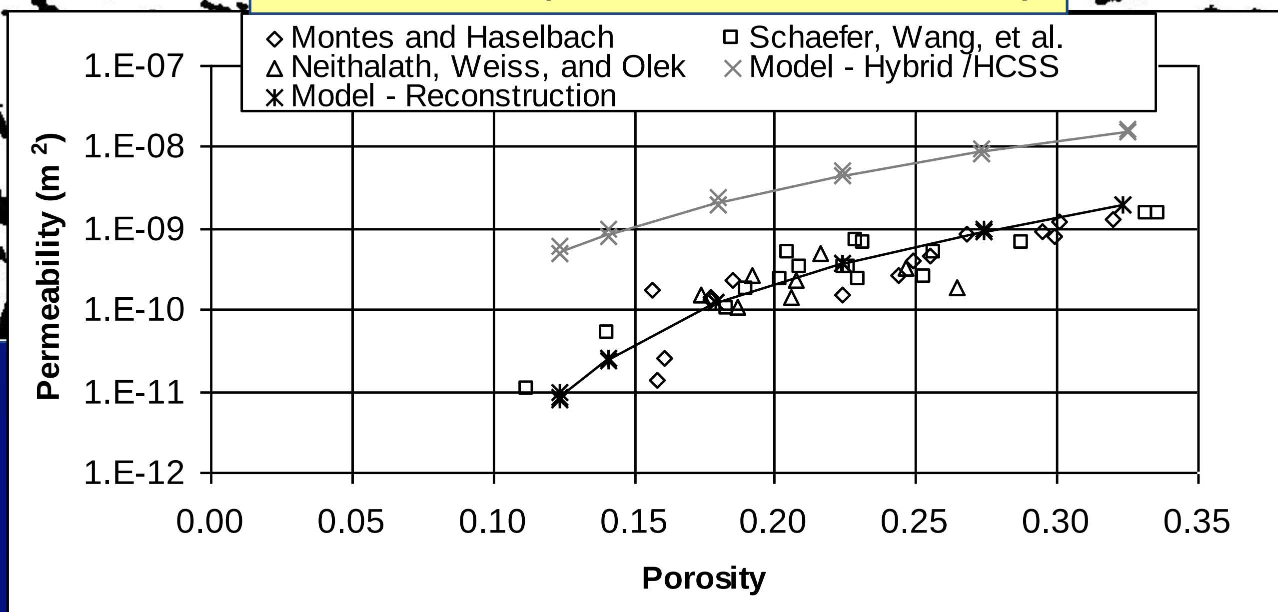


Image from 3-D Reconstruction



Model vs. Experimental Permeability



Neutron Tomography

Quantifying sorption in mortars exposed to $CaCl_2$

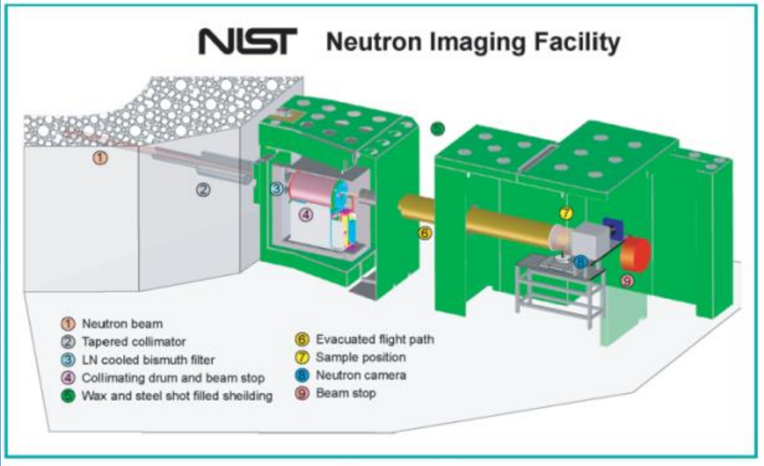
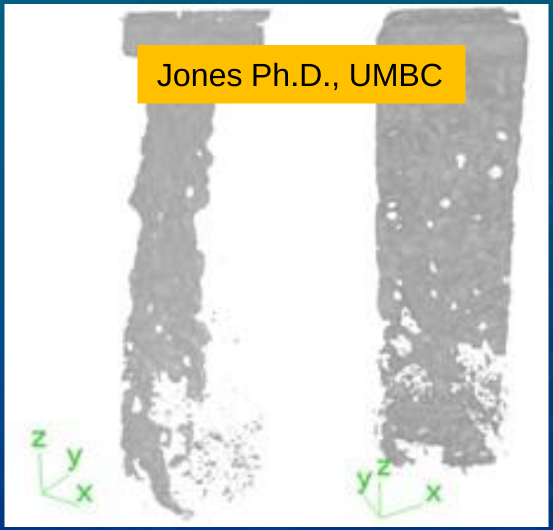
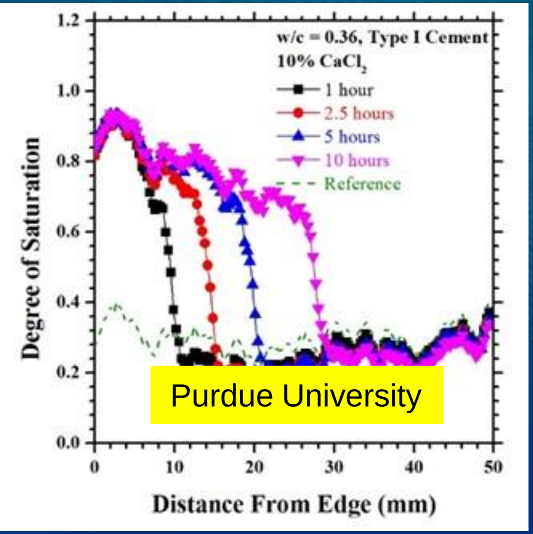


Fig. 1. The new thermal neutron imaging facility at the NCNR.

David Jacobson and Daniel Hussey
Available to the public, apply for beamtime at :
ncnr.nist.gov/call/current_call.html

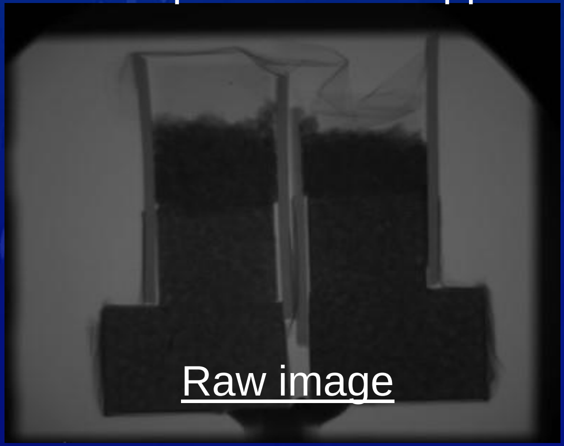


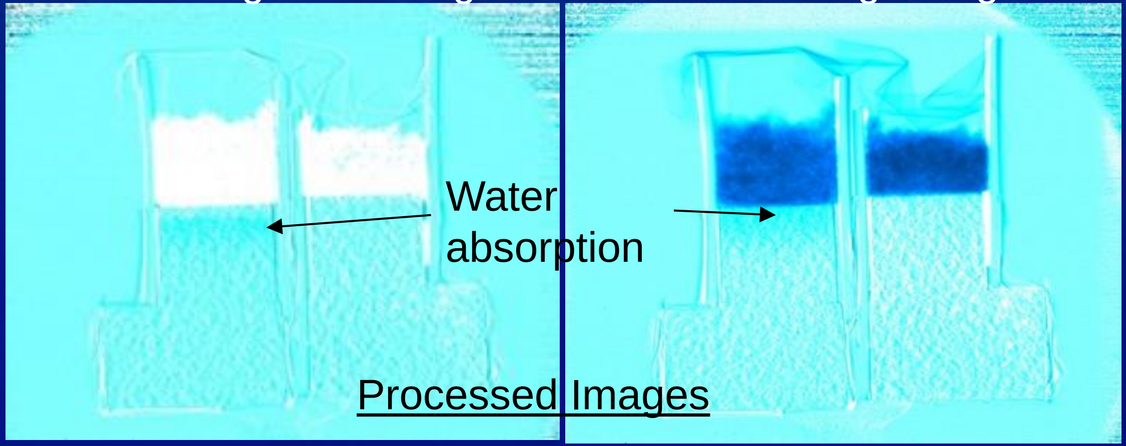
3-D imaging of methacrylate crack filler



Monitoring of moisture movement in repair materials

After repair material applied | 25 h image/ 1 h image | 25 h image/ original



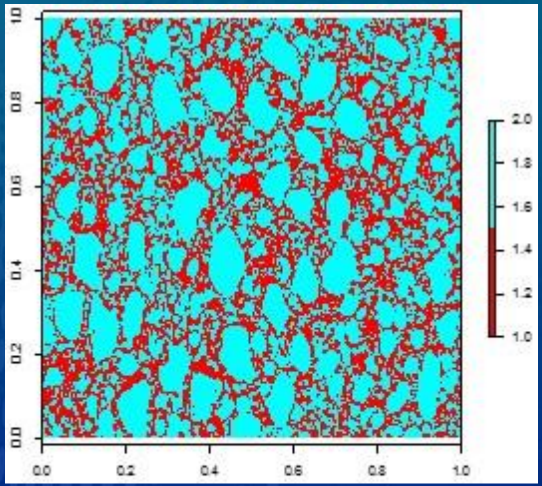


Left- dry; Right - SSD

White – water loss Cyan – water gain

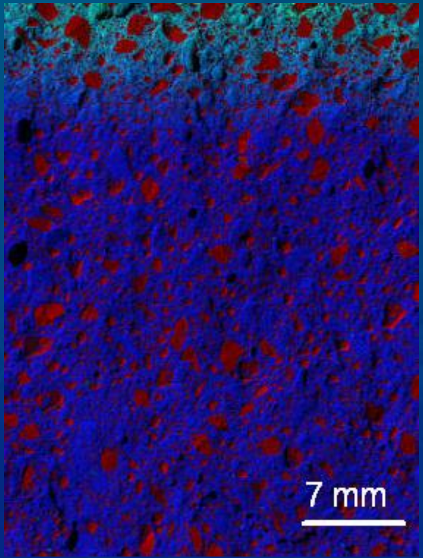
Microbeam X-ray Fluorescence Imaging

Identification of paste and aggregates in mortar

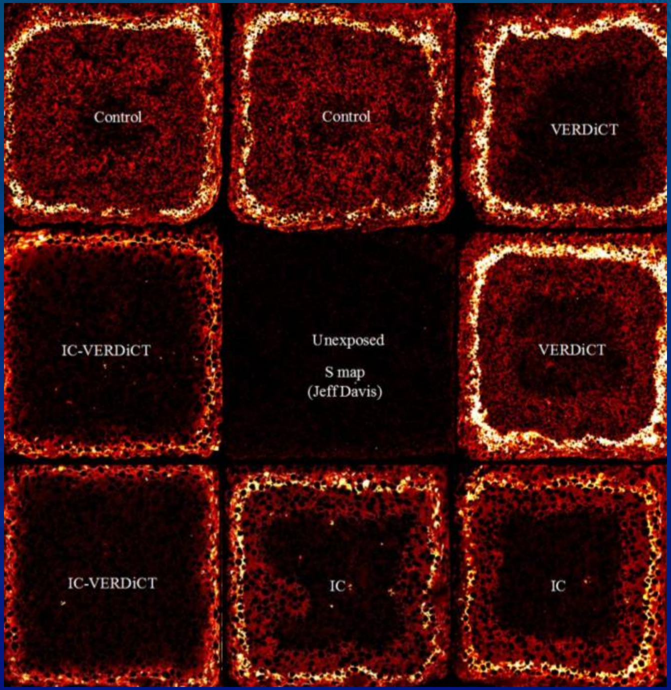


Jones Ph.D., UMBC

Quantifying Cl^- ingress
R=Si, G=Cl, B=Ca



Quantifying sulfate ingress (ASTM testing) in mortars with various additives and/or internal curing



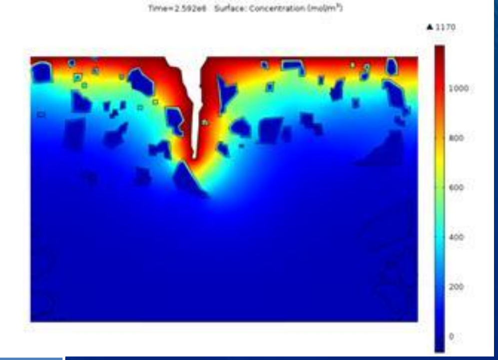
Jeffrey Davis
PNDetector

Can easily image specimen fracture surfaces with minimum sample preparation

Recent Work - Images of Cracks as Inputs to Computational Models

Impact of a small (50 μm wide by 20 mm deep) crack on service life

Variable		Service life (years)	Change from uncracked case
$C_{rebar}/C_{ext} = 0.1$	51 mm cover (34 yrs)	23	(32 %)
	76 mm cover (76 yrs)	65	(14 %)
	102 mm cover (137 yrs)	126	(8 %)
	5% silica fume (51 mm;103 y)	69	(33 %)
	7% silica fume (51 mm;172 y)	113	(34 %)
$C_{rebar}/C_{ext} = 0.3$	Corrosion inhibitor (51mm;86)	67	(22 %)
$C_{rebar}/C_{ext} = 0.5$	Epoxy-coated rebar (51;203)	167	(18 %)

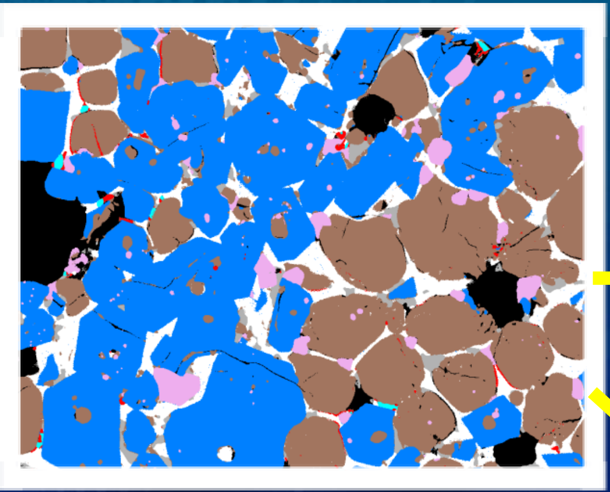


Impact of a large (500 μm wide by 40 mm deep) crack on service life

Variable		Service life (years)	Change from uncracked case
$C_{rebar}/C_{ext} = 0.1$	51 mm cover	1	(97 %)
	76 mm cover	26	(66 %)
	102 mm cover	83	(39 %)
	5% silica fume (51 mm)	2	(98 %)
	7% silica fume (51 mm)	4	(98 %)
$C_{rebar}/C_{ext} = 0.3$	Corrosion inhibitor (51 mm)	4	(95 %)
$C_{rebar}/C_{ext} = 0.5$	Epoxy-coated rebar (51 mm)	16	(92 %)

Jones, S., Martys, N., Lu, Y., and Bentz, D., "Simulation Studies of Methods to Delay Corrosion and Increase Service Life for Cracked Concrete Exposed to Chlorides," *Cement and Concrete Composites*, **58**, 59-69, 2015.

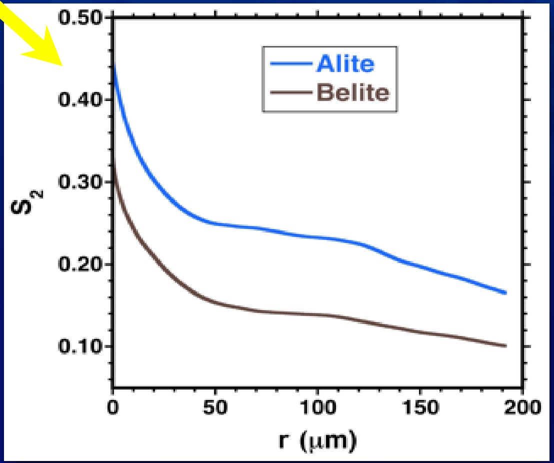
Recent Work - Quantitative Phase Analysis of Clinker by Scanning Electron Microscopy (SEM)



Alite | Periclase
Belite | Arcanite
C_3A | Aphthitalite
C_4AF | Void

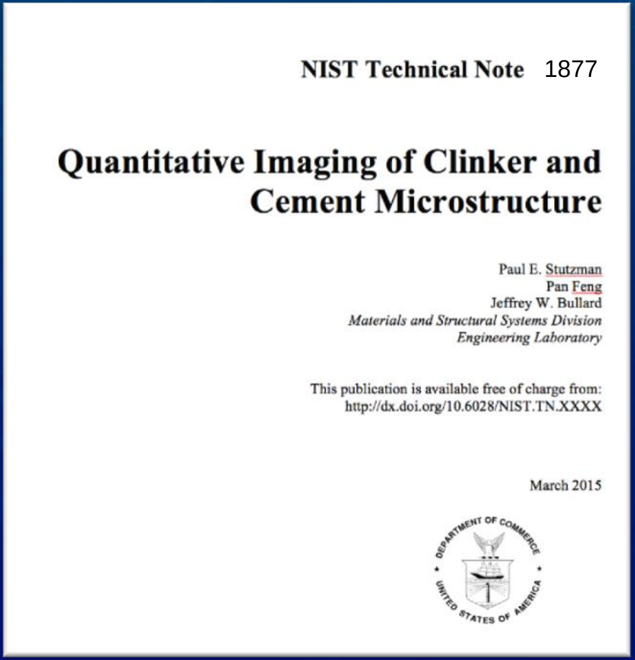
SRM2686.txt.pfc — Edited

NAME	MASS	VOLUME	AREA
Alite	0.4609	0.4741	0.2245
Belite	0.3479	0.3503	0.2971
Aluminate	0.0243	0.0264	0.0974
Ferrite	0.1202	0.1064	0.2617
Arcanite	0.0039	0.0049	0.0412
Thenardite	0.0017	0.0021	0.0077
Periclase	0.0411	0.0359	0.0705



Correlation functions again!

NIST Tech Note 1877
Stutzman, Feng, & Bullard



NIST Technical Note 1877

Quantitative Imaging of Clinker and Cement Microstructure

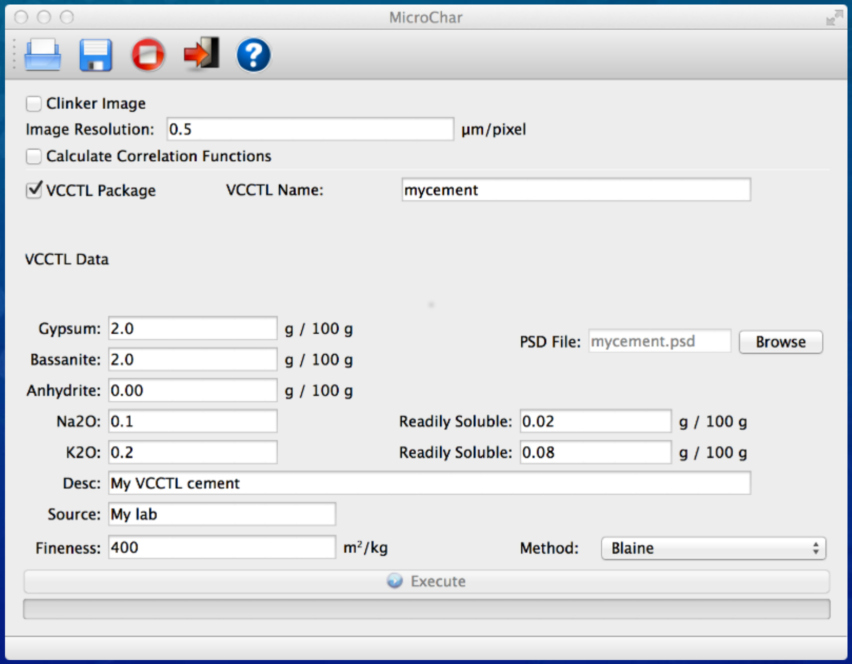
Paul E. Stutzman
Pan Feng
Jeffrey W. Bullard
Materials and Structural Systems Division
Engineering Laboratory

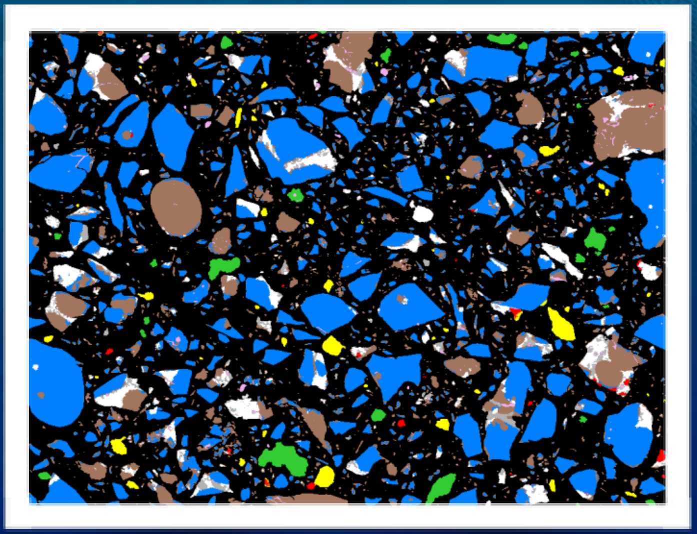
This publication is available free of charge from:
http://dx.doi.org/10.6028/NIST.TN.XXXX

March 2015

Recent Work

MicroChar: New software for semi-automatic quantitative analysis of segmented cement (and clinker) microstructures





Alite, Calcite, Belite, Arcanite, C_3A, Gypsum, C_4AF, Void

- Volume and area fractions
- Two-point correlation functions
- Formats data for VCCTL upload
- Available for Windows and Mac
- Bullard, J.W., NIST Technical Note 1876, April 2015.

Summary and Prospectus

- Imaging has a wide & ever-growing range of applications to building materials
  - Quantitative analysis for property and failure state characterization
  - Inputs for and comparison to models
    - Digital-image-based modeling
- Freeware for imaging is ubiquitous and powerful
  - NIH ImageJ and Purdue MultiSpec as examples, along with NIST products
- Multi-spectral imaging and other combinations of signals/sources (concurrent X-ray and neutron imaging)
  - NIST neutron imaging facility recently upgraded to include X-ray
- Exciting future awaits us as researchers in this field

Collaborators

Martin Batts, Jeff Bullard, Jim Clifton, Tate Coverdale, Jeffrey Davis, Ned Embree, Xiuping Feng, Geoff Frohnsdorff, Ed Garboczi, Mette Geiker, Abraham Grader, Claus Haecker, Phil Halleck, Kurt Hansen, Daniel Hussey, David Jacobson, Hamlin Jennings, Ole Jensen, Scott Jones, Larry Kaetzel, Mark Levenson, Yang Lu, Pietro Lura, Catherine Lucero, Jonathan Martin, Nick Martys, Mary McKnight, Narayanan Neithalath, Pierre Pimienta, Daniel Quenard, Sébastien Rémond, John Roberts, Tony Roberts, Erik Schlangen, Larry Schwartz, Ken Snyder, Paul Stutzman, Milani Sumanasooriya, Franck Vallee, Jason Weiss, et al.

Thank you for your attention!

Questions ????