X Ray 3D imaging of construction materials

Mateis activities in XRCT on construction materials

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Outline

1. Introduction - Background (history, some old static CT reconstructions)
2. What can be done with the static images : 3D Image processing
3. Dynamic measurements (Marco)

- Plaster burning
- Plaster setting
- FE simulation, DVC measurements (François)

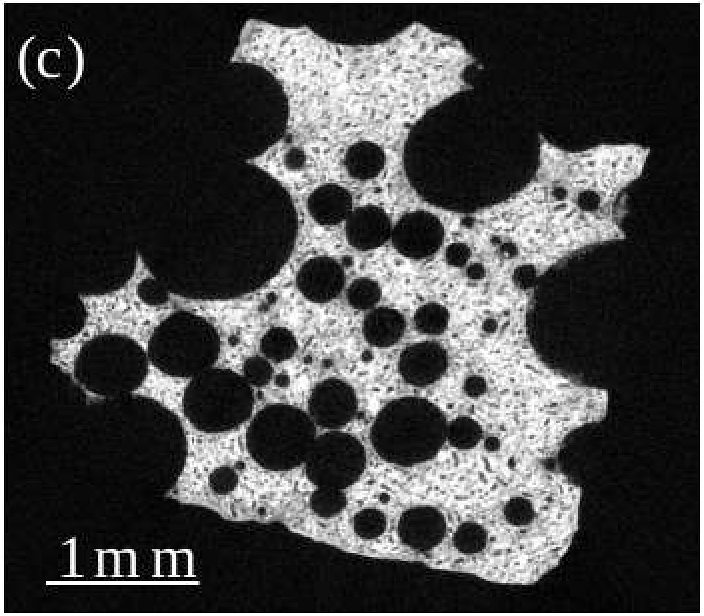
Conclusion

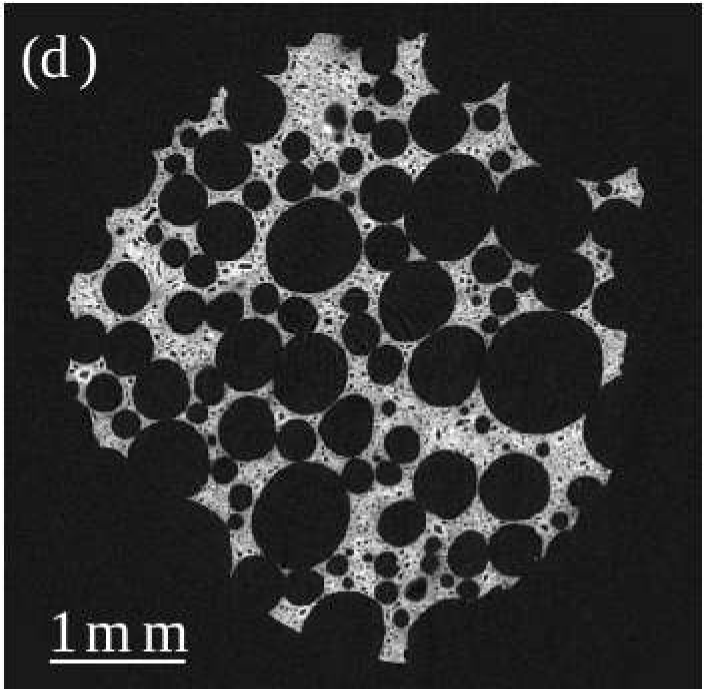
3D X Ray imagers : Tomographs used

- ESRF ID19
- ESRF ID22NI – ID16B
- Lab CT at Mateis (GE V|TomeX + easytom RX Solutions)
- SLS Tomcat

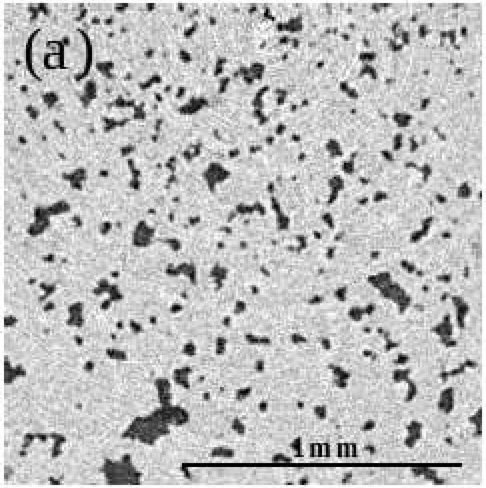
Mateis / construction materials

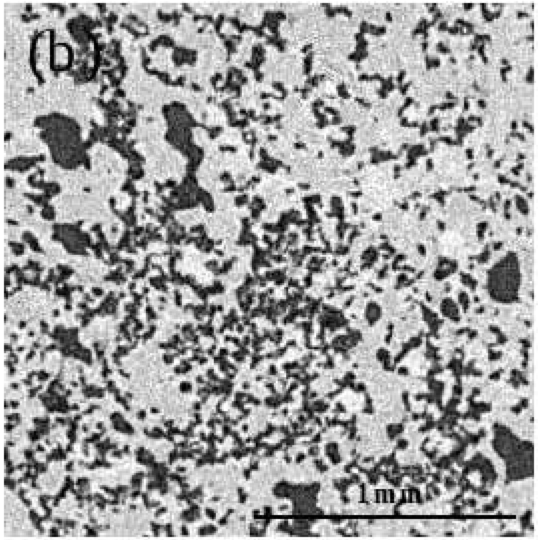
Plaster

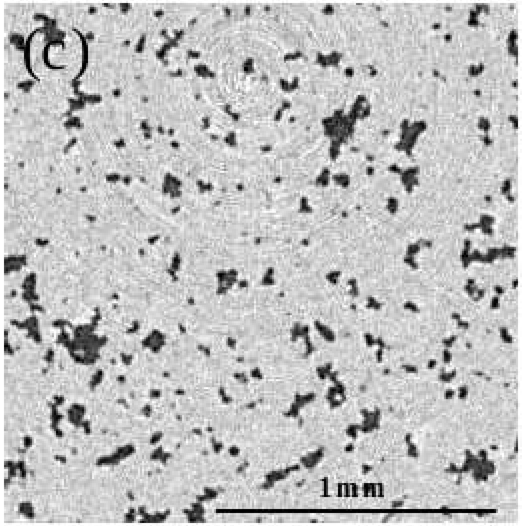




Post doc
A. King 2005
For Lafarge
F Thoué
S Meulenyzer



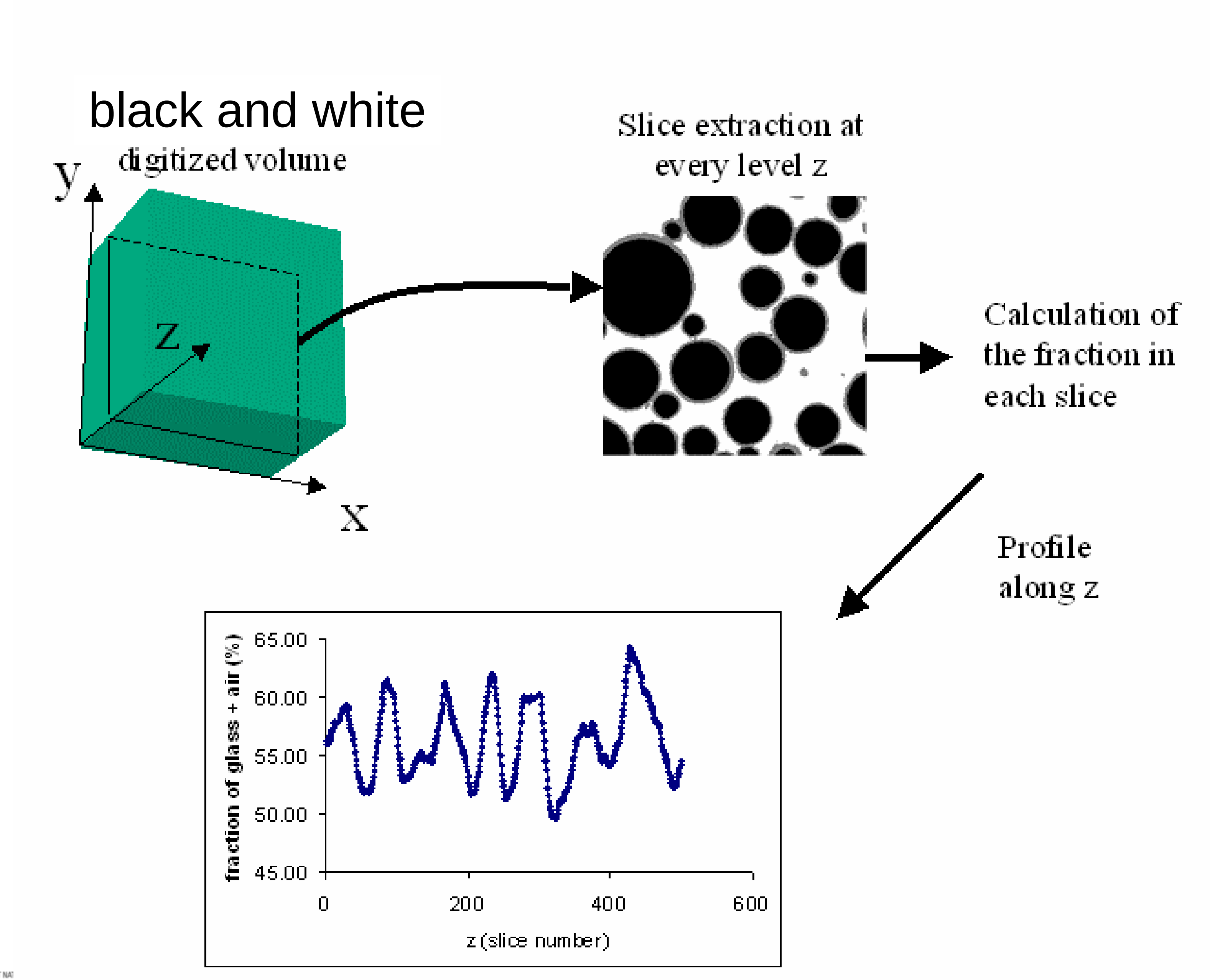




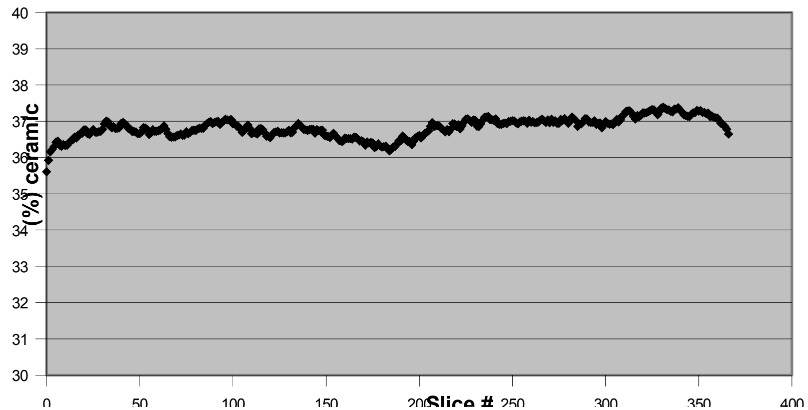
Clinker

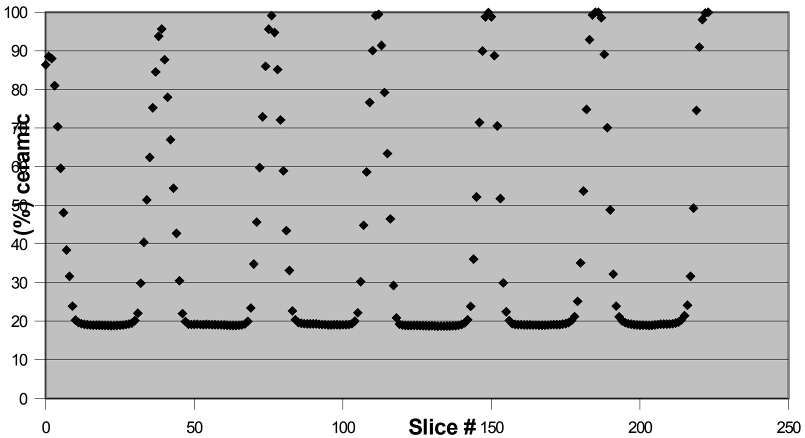
Quantification of the microstructure

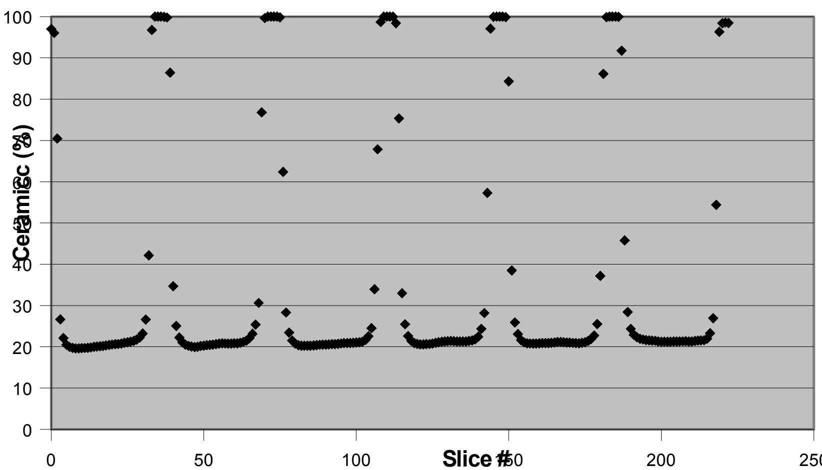
1. Volume fraction (and its spatial distribution)

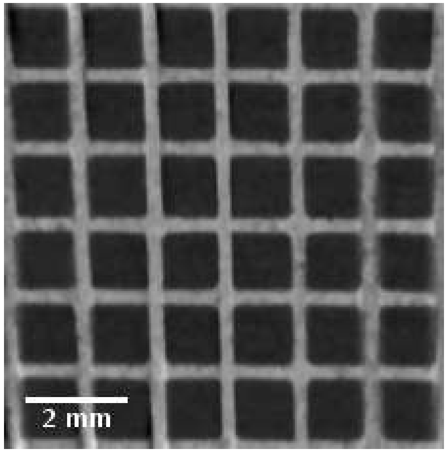


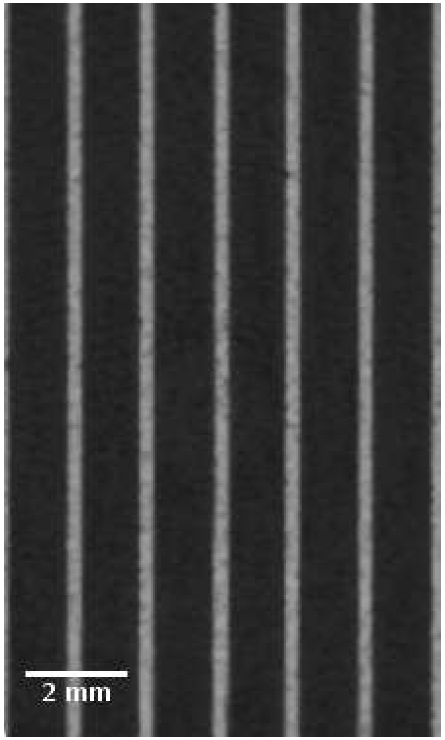
Periodic material



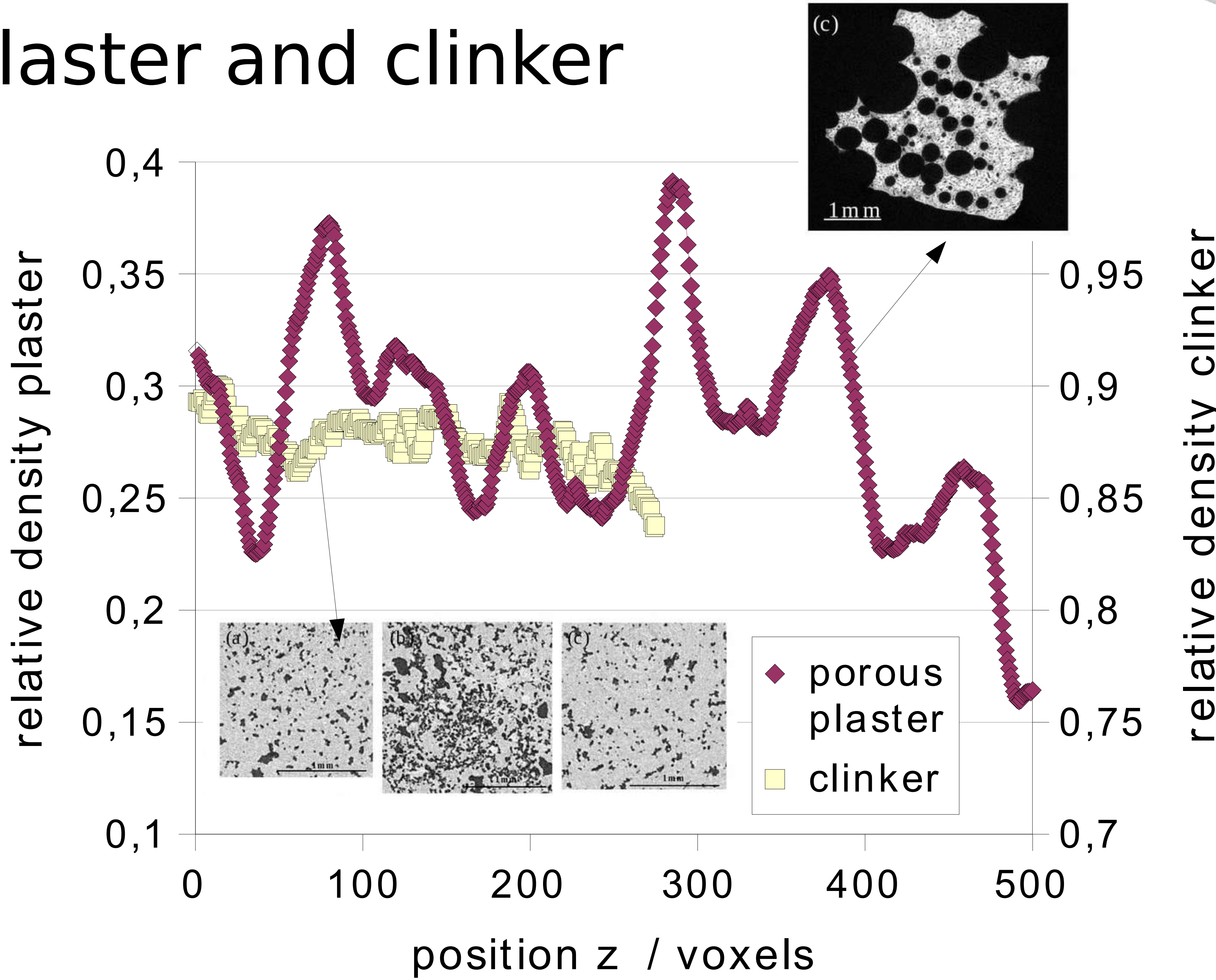








Plaster and clinker

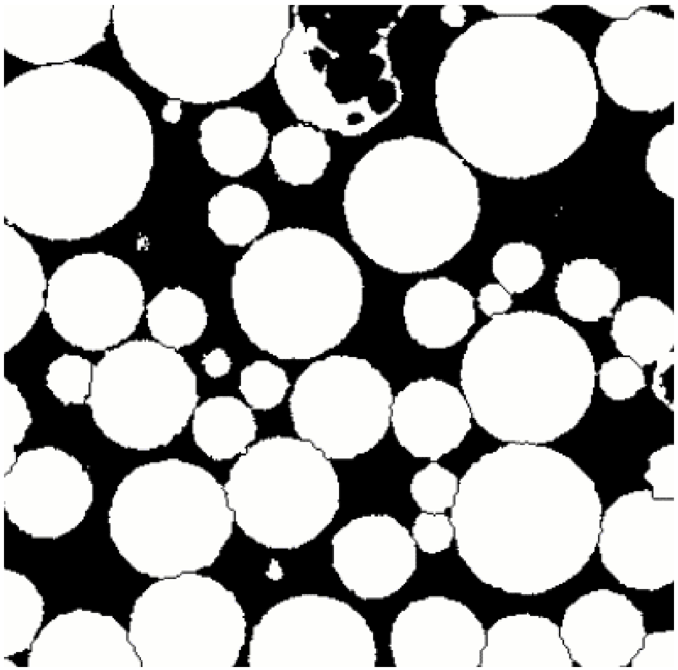


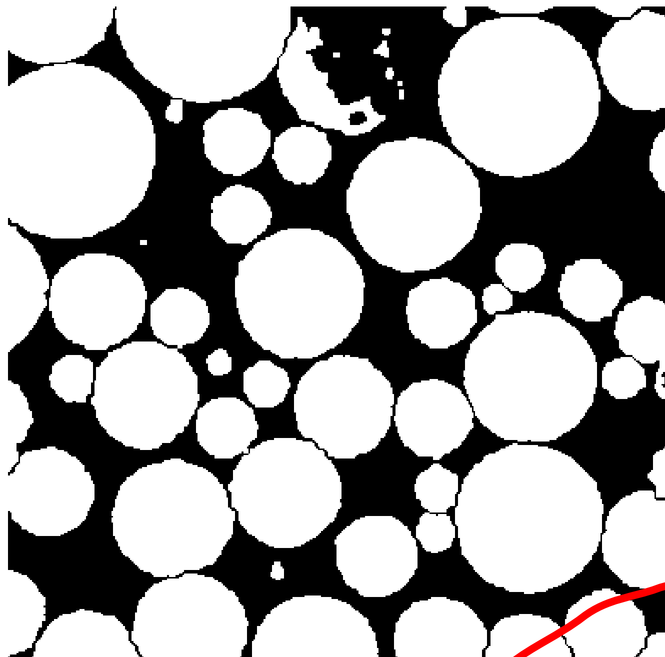
2. Size
3D granulometry=sequencial openings

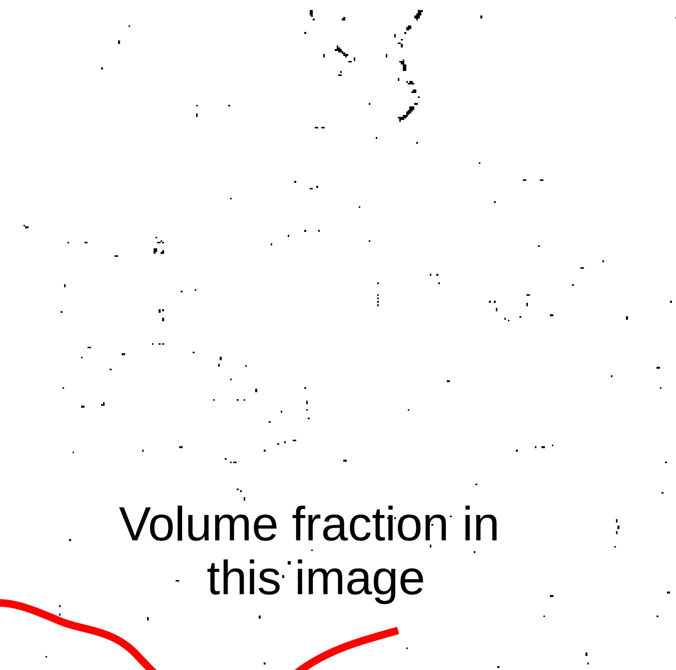
A = Initial

B = Initial + erosion + dilation

Difference between A and B

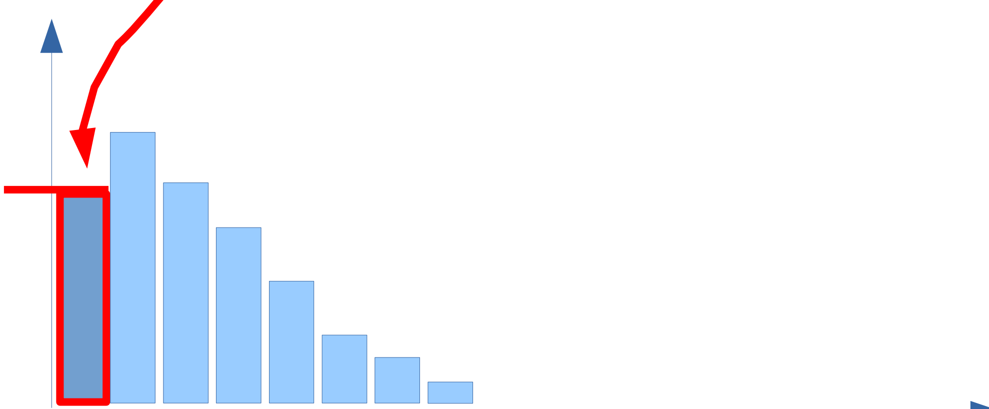






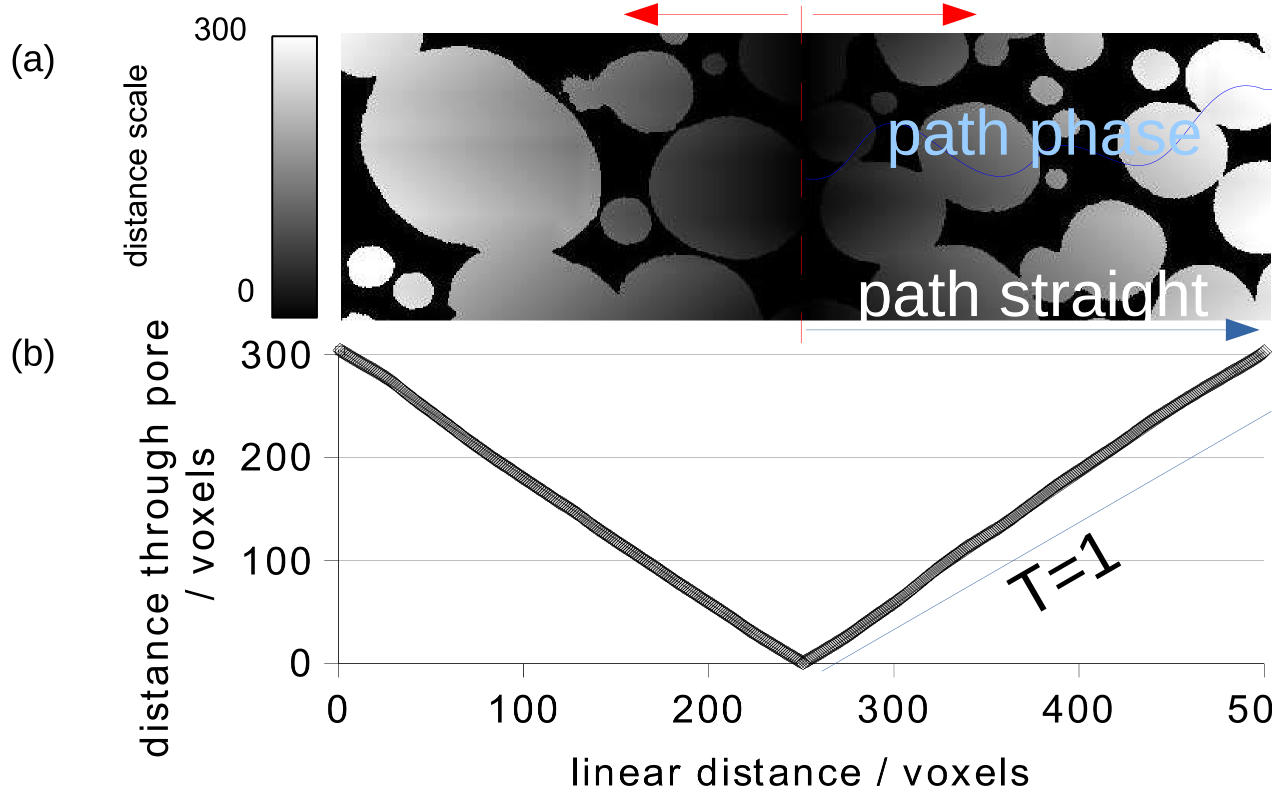
Volume fraction in this image

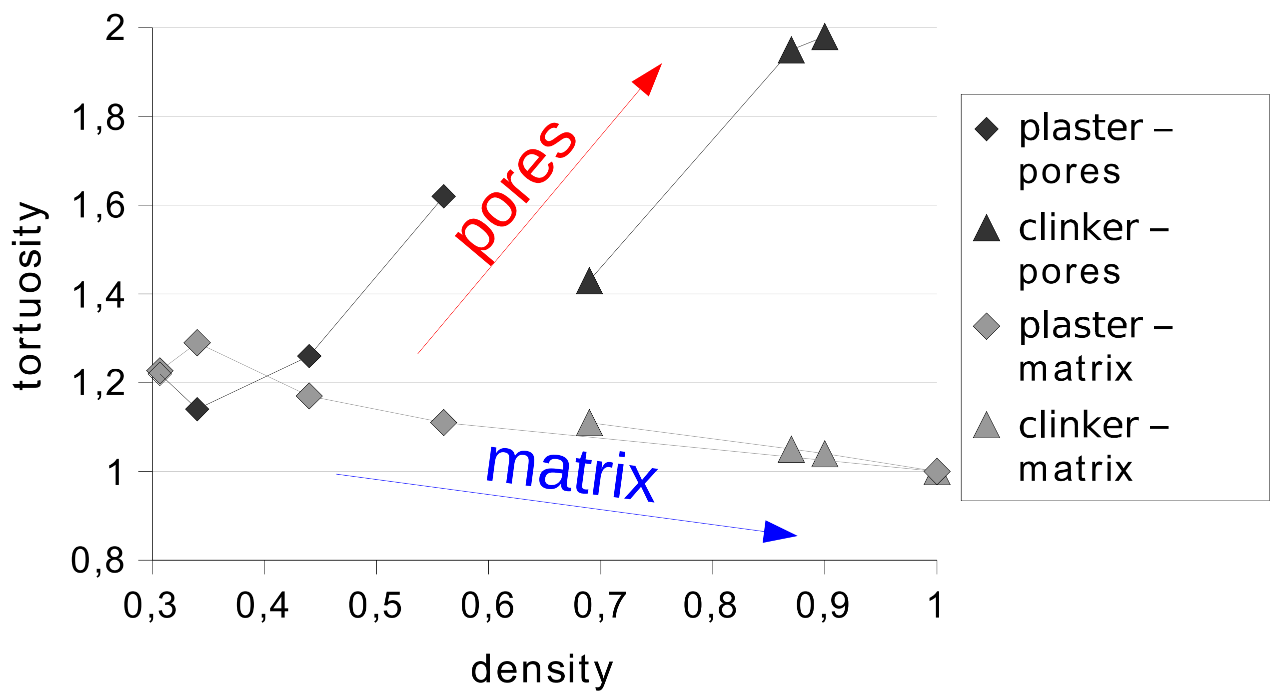
Openings with different shapes (cube, octa, spheres) give different results



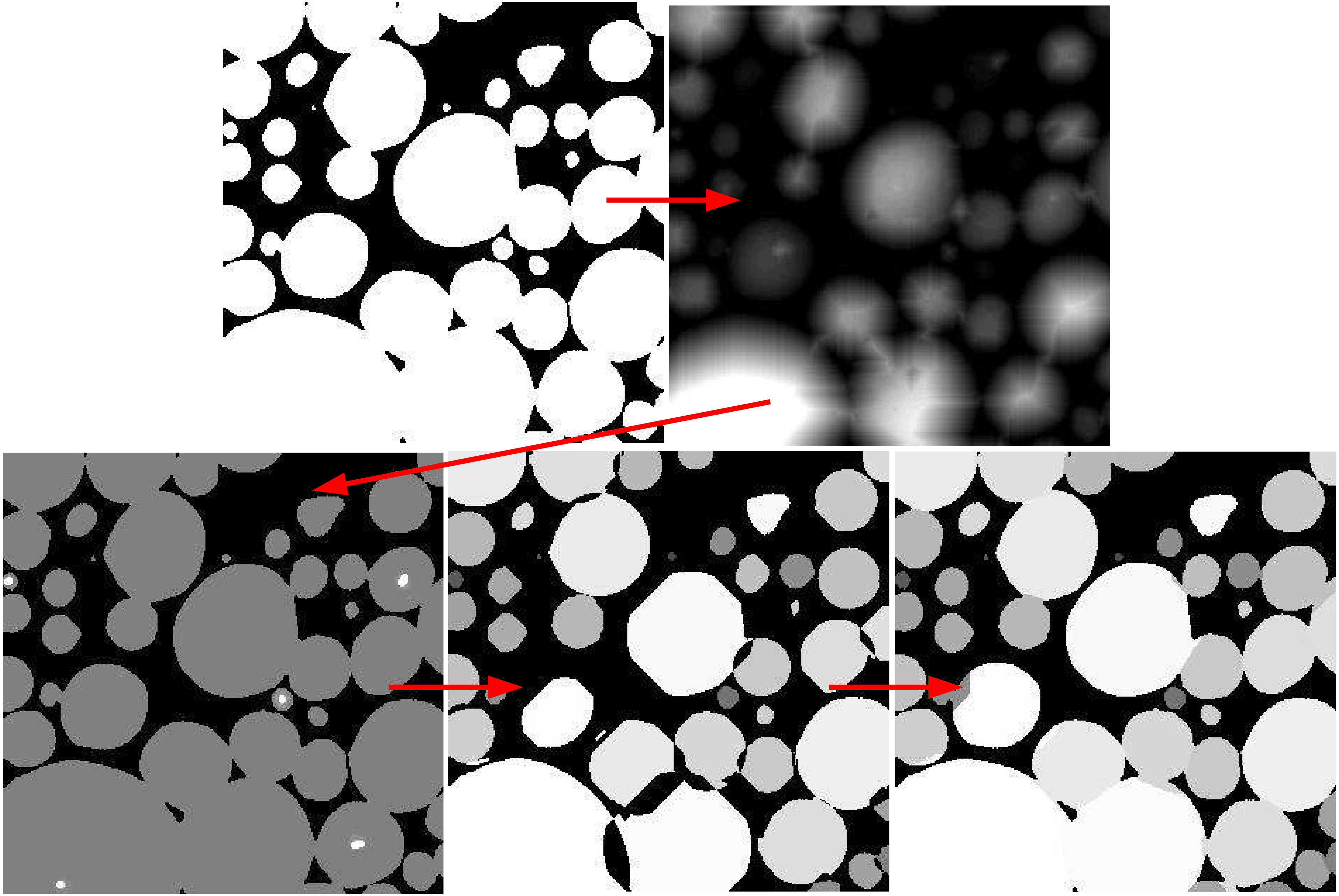
Tortuosity

- $T = \frac{Path_{phase}}{Path_{straight}}$
- Useful for transfer properties (thermal, acoustic)

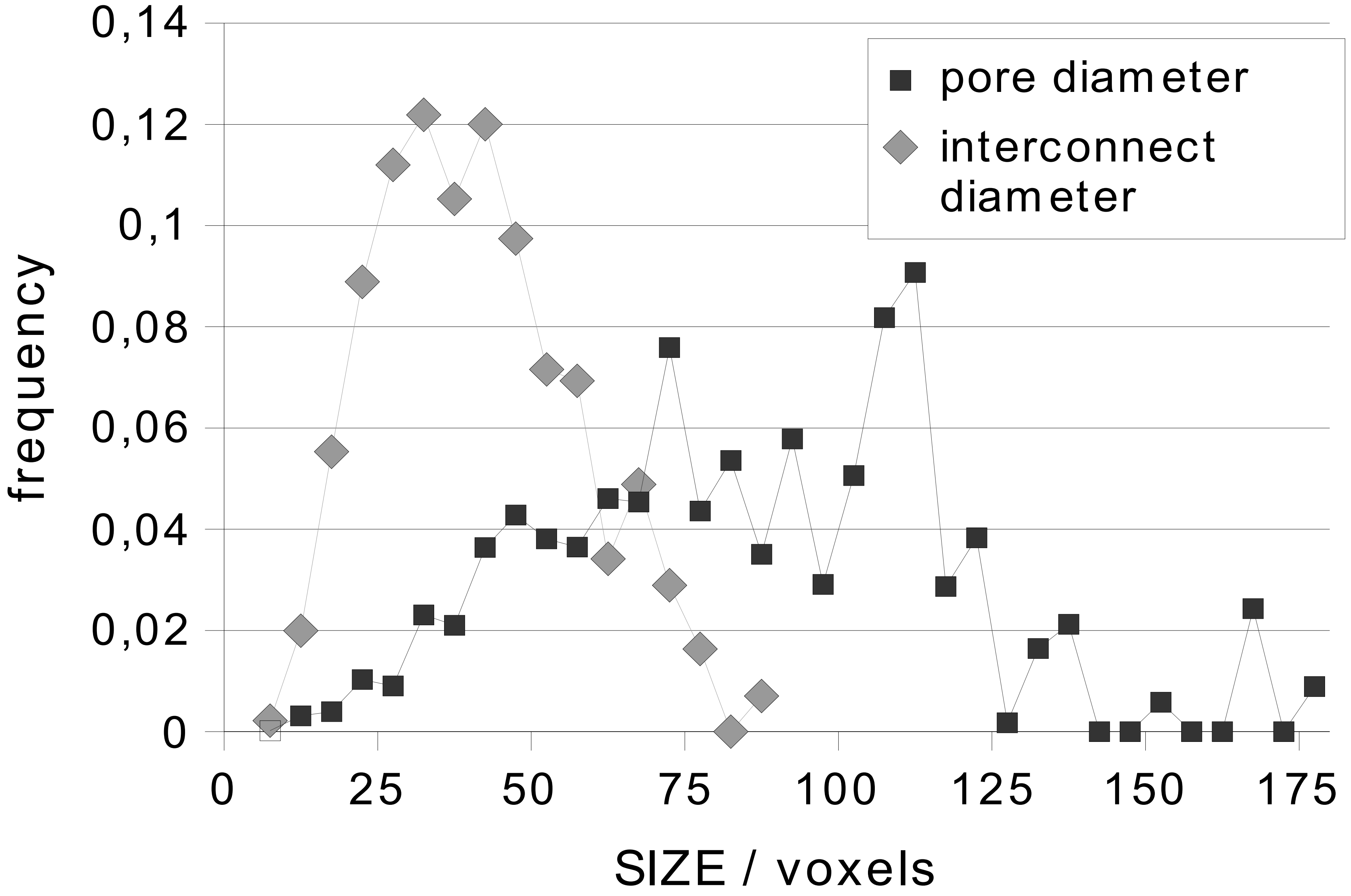




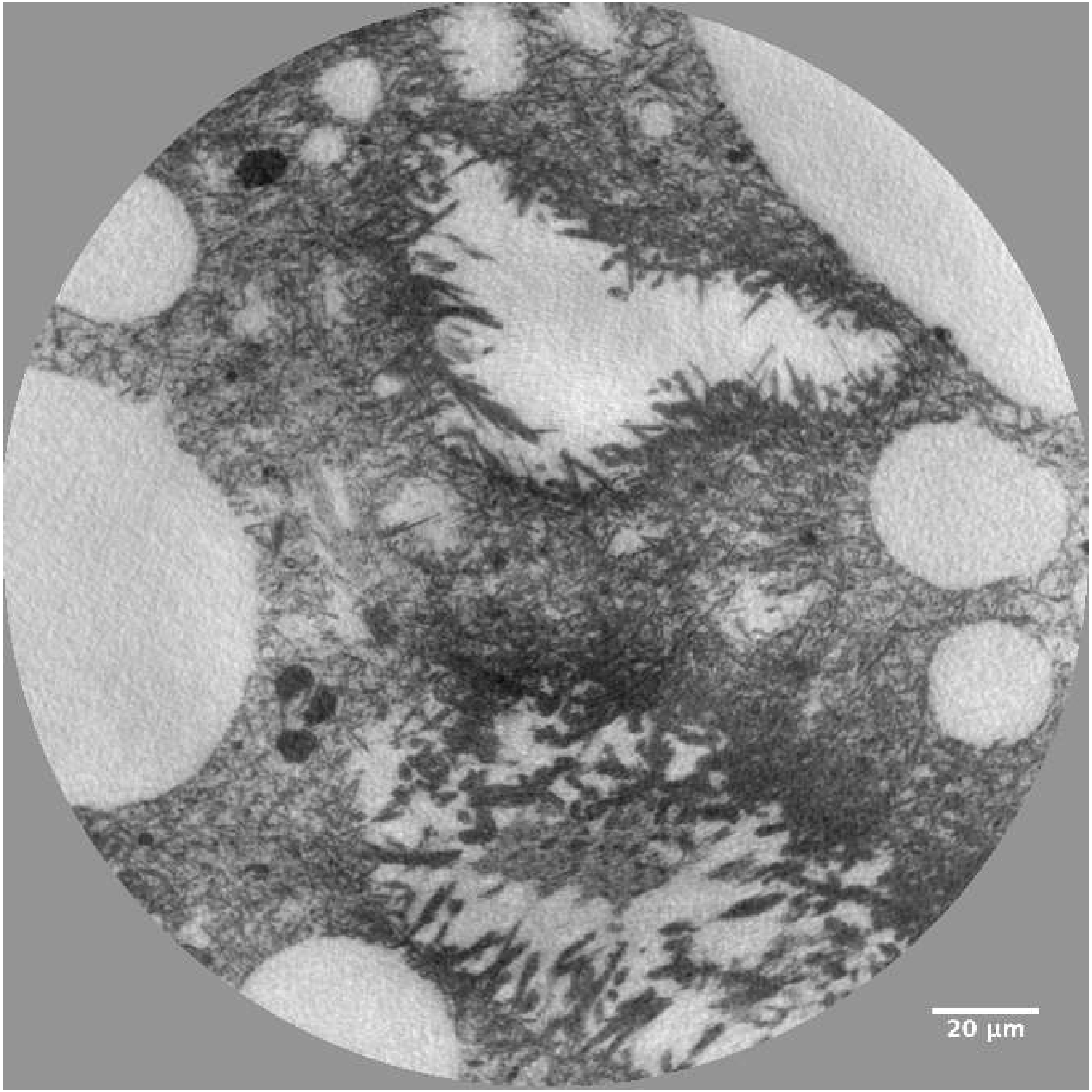
Interconnections



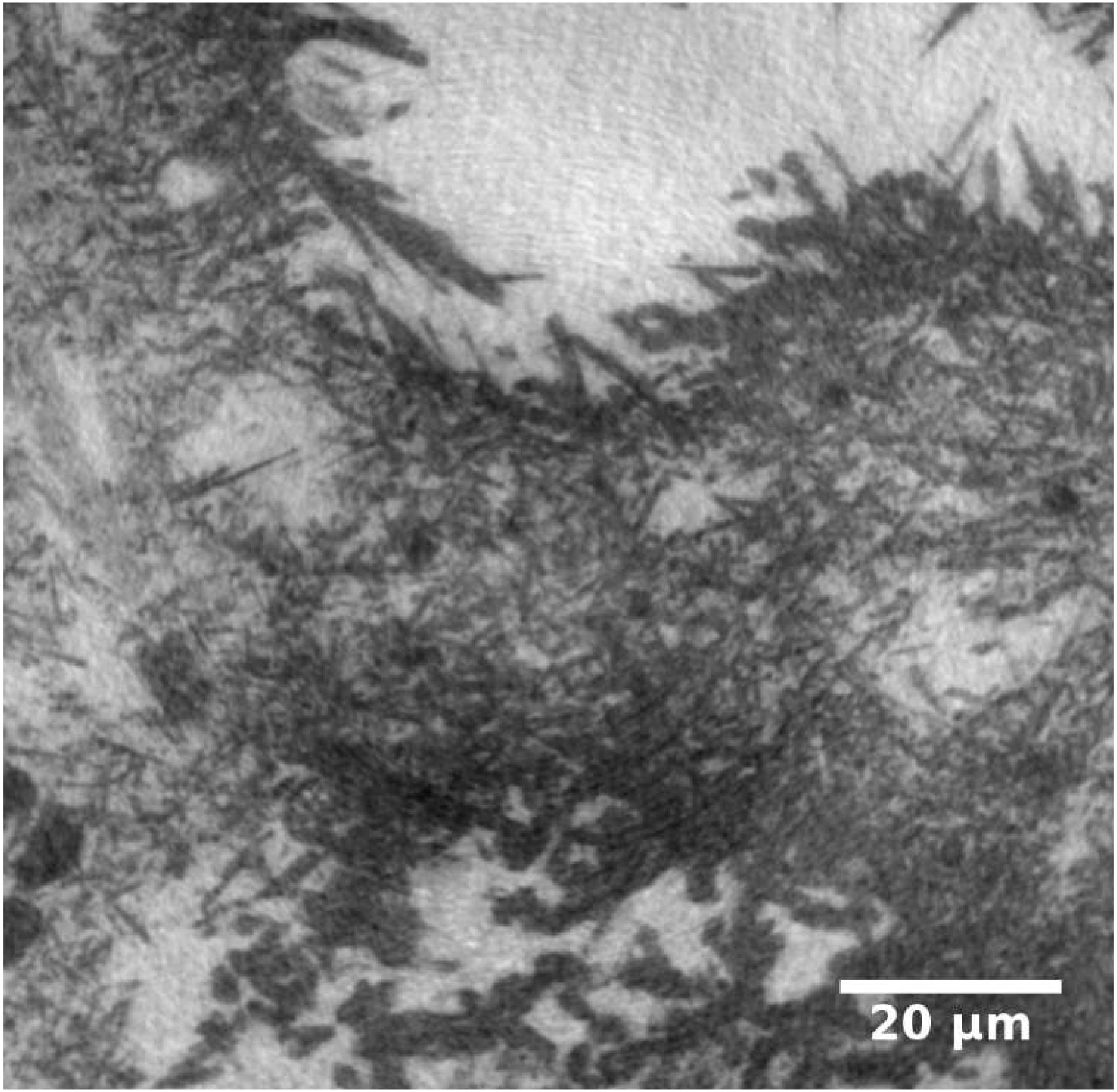
Results on plasterboard



Pixel size 100 nm (ID22NI ESRF)



Pixel size 100 nm (ID22NI ESRF)



Conclusion (partial)

- Static 3D imaging of constrcution materials is well established
- + 3D image processing
- Detailed information about the microstructure
- Covers a large size range
- Non destructive
- Can be complemented with destructive
  - 3D FiB
  - 3D TEM
  - 3D atome probe

Dynamic studies

High temperature transformation of plaster

FIRE AND MATERIALS
Fire Mater. 2016
Published online in Wiley Online Library (wileyonlinelibrary.com). DOI: 10.1002/fam.2357

In situ observation of plaster microstructure evolution during thermal loading

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2 µm resolution
ESRF ID19 – 2 minutes for a scan

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SEM in siotu observations

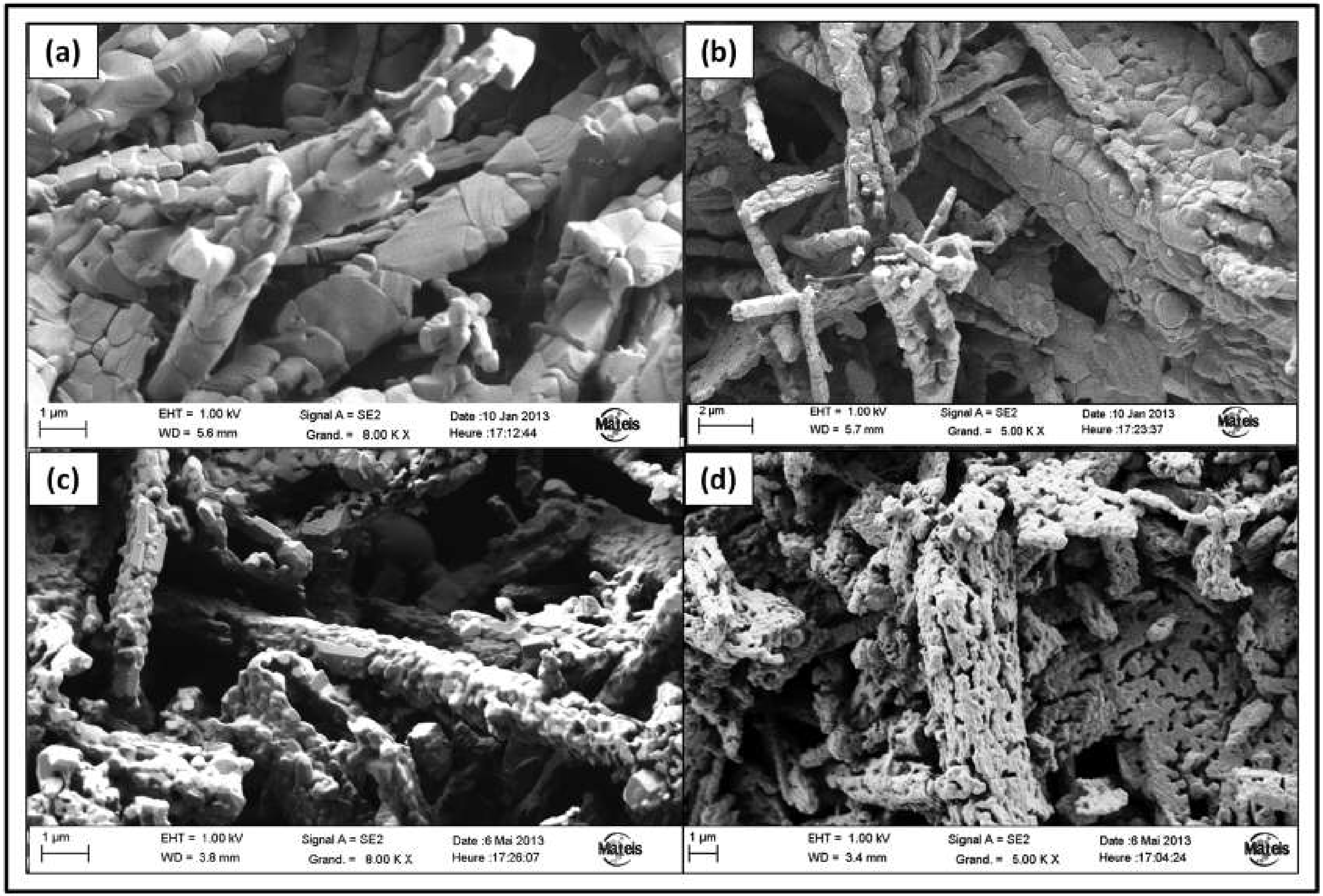

Figure 4. Scanning electron microscope images of a sample heated at (a) and (b) 900 °C in a conventional furnace and rapidly cooled down and (c) and (d) at 950 °C in the environmental scanning electron microscopy.

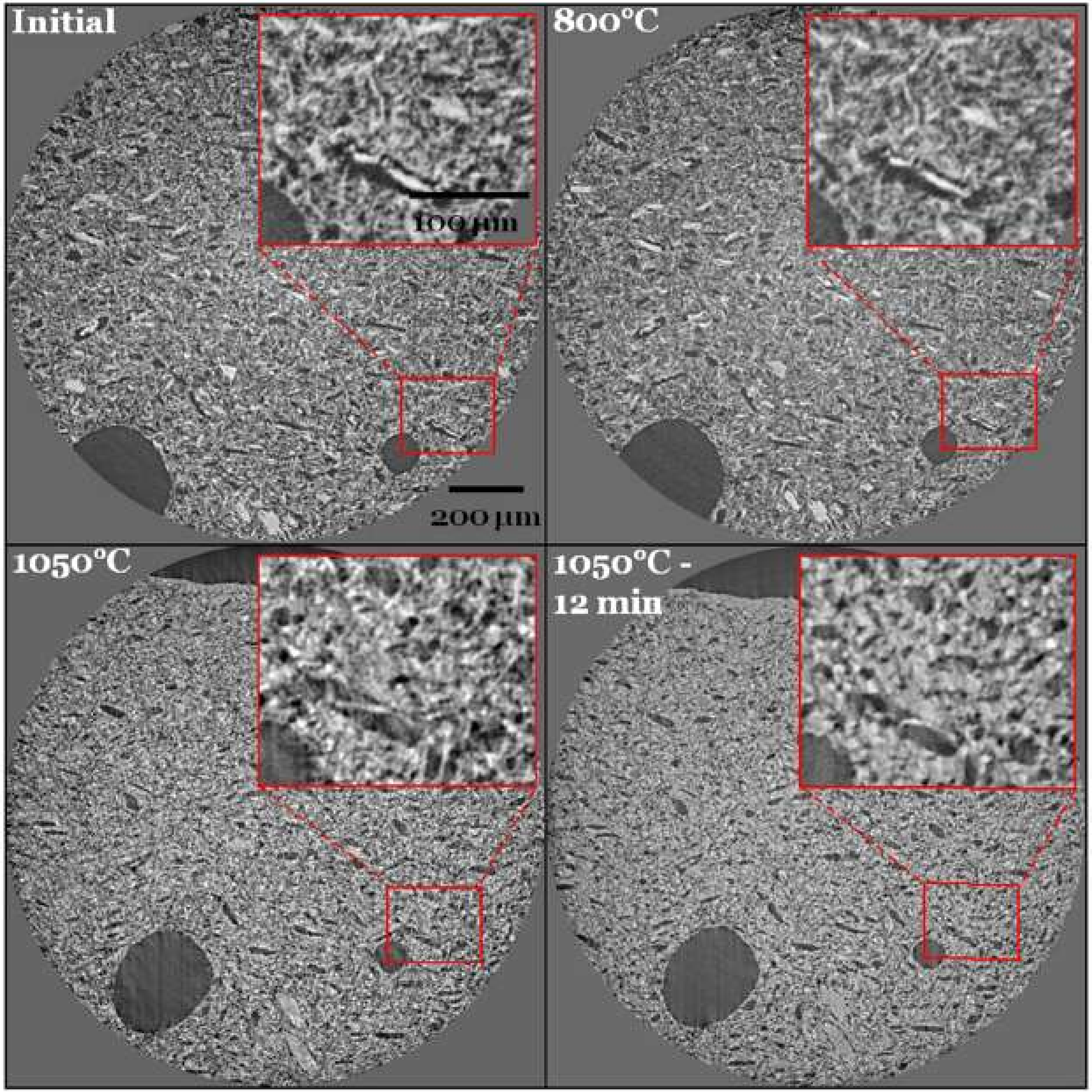

Figure 5. Reconstructed slices from synchrotron X-ray tomography of the same area of a foamed plaster heated *in situ* up to 1050 °C showing microstructural evolutions with thermal loading. Inserts show magnified views of the micropores.

Grey levels

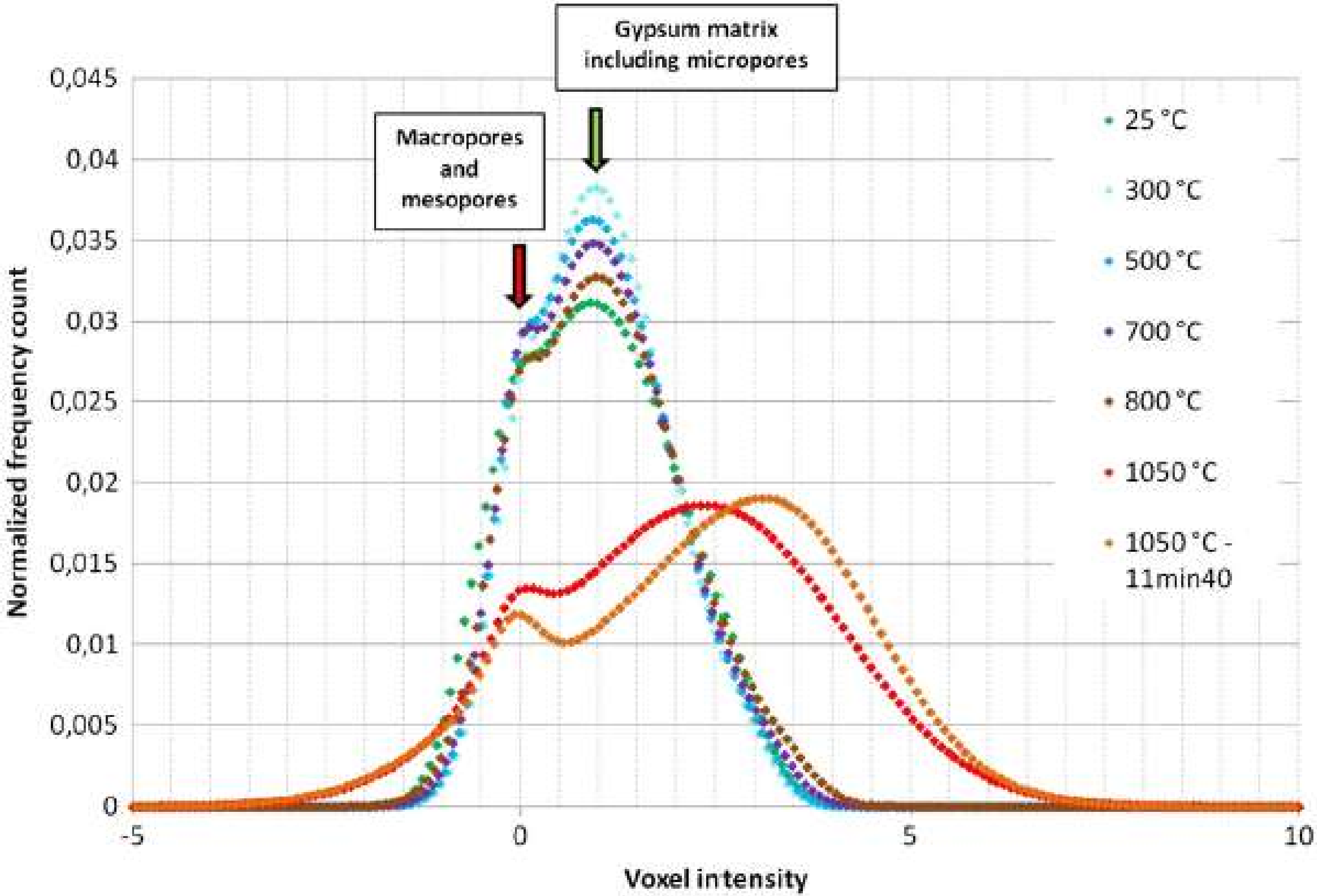

Figure 7. Evolution of the normalized distribution of voxel intensities (arbitrary unit) on X-ray tomography reconstructions with thermal loading.

Shrinkage

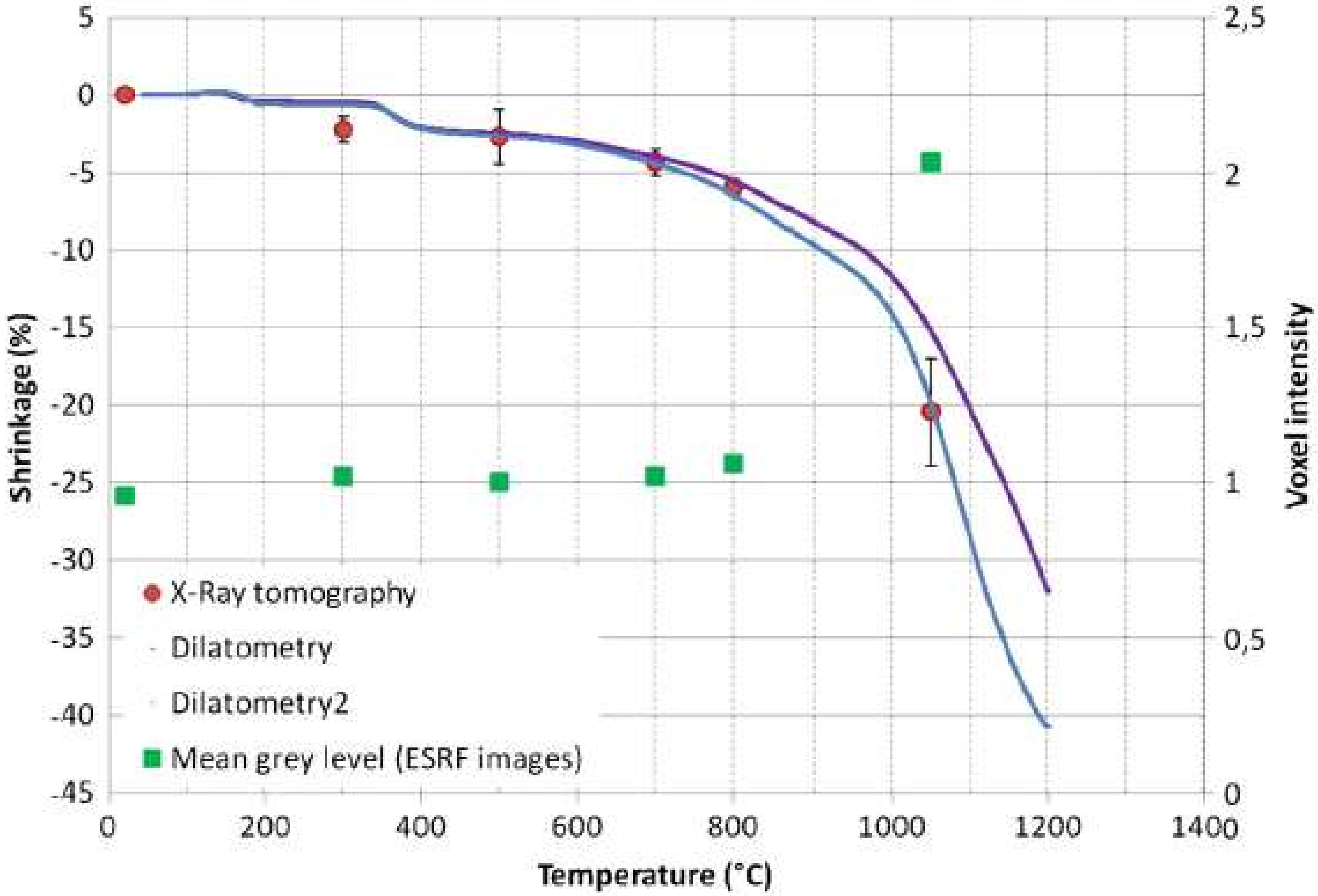

Figure 6. Shrinkage measurements evaluated by image analysis of X-ray tomography reconstructions (circles, average of three measurements) and comparison with dilatometry results. Mean grey level values from the European Synchrotron Radiation Facility tomographies are also shown (squares).

In situ monitoring of plaster setting

Cement and Concrete Research 82 (2016) 107–116

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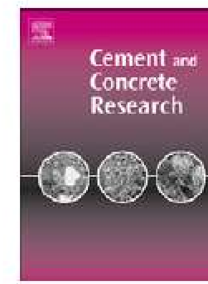
journal homepage: www.elsevier.com/locate/cemconres



In-situ X-ray tomographic monitoring of gypsum plaster setting

CrossMark

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Experimental procedure

In situ monitoring of plaster hydration using X-Ray tomography

Materials

β hemihydrate powder, 96% purity

W/P 0.8, hand mixing

Knife setting time: 1200 s (20’)

$$\beta\, CaSO_4, \frac{1}{2} H_2O$$ + water

Tomography

Spatial resolution: 2.5 µm per voxel

Acquisition time: 3’ per scan (600 projections)

Monitoring from 800 s (13’20”) to 6200 s (1h43’)

Final scan after full hydration and drying (better imaging conditions)

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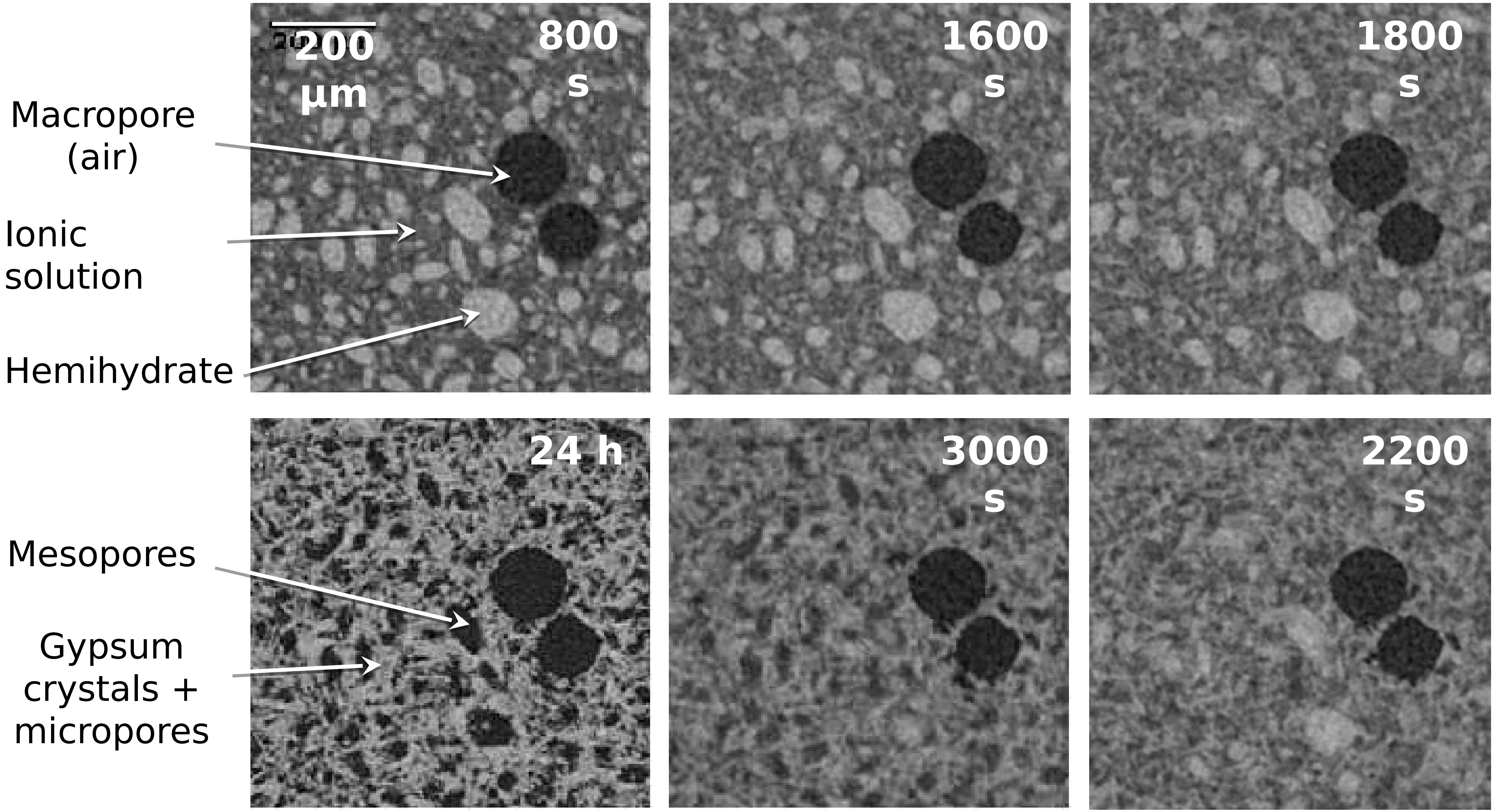
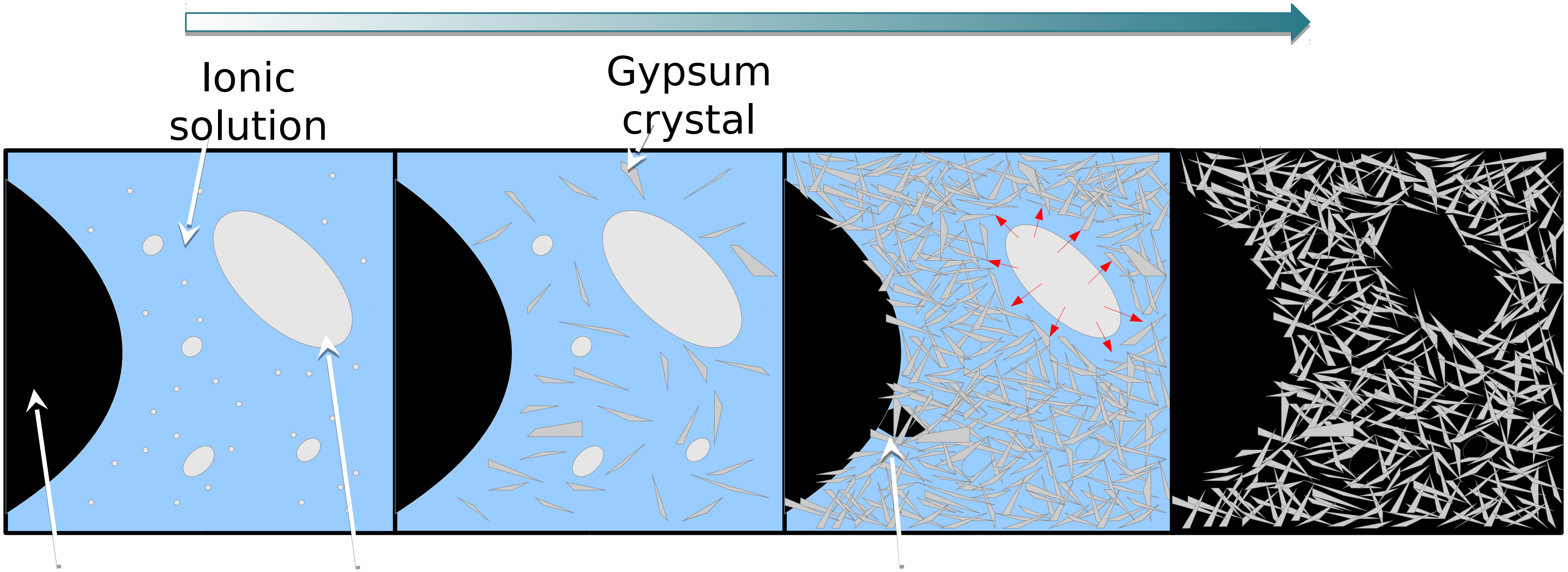
Evolution with time

Same section of a reconstructed 3D volume



Conclusions

Schematic representation of calcium sulfate hydration



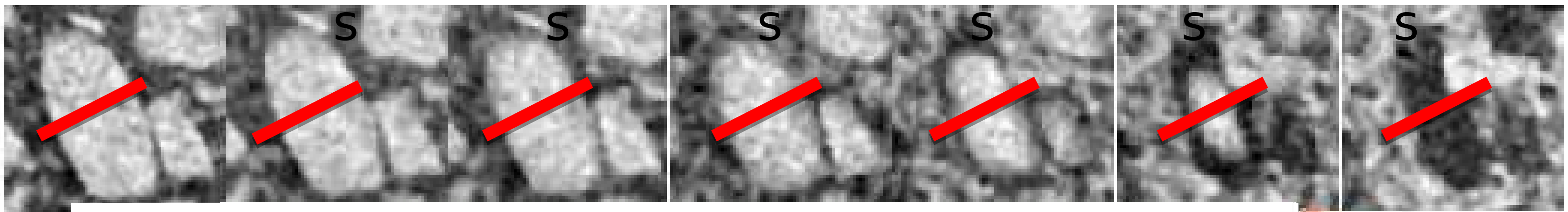
Possible control of final microstructure with initial particle size distribution

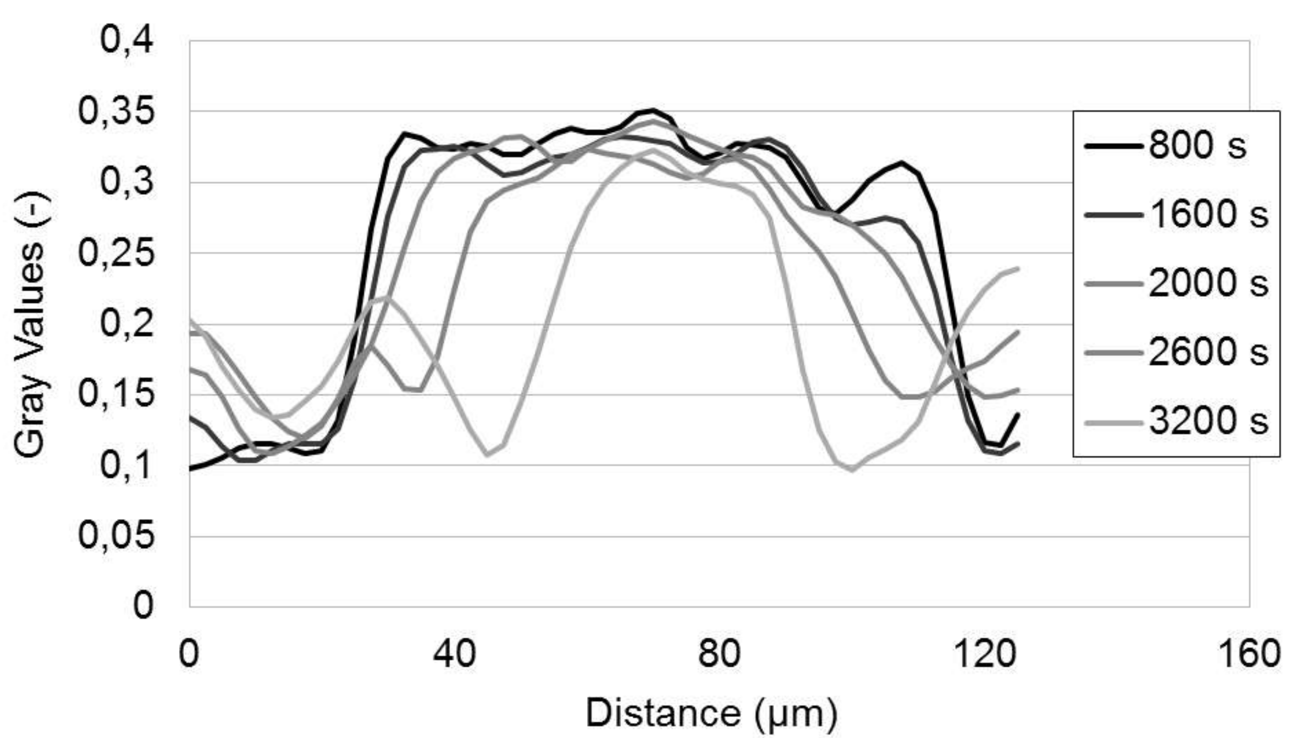
Gypsum is NOT a simple material!

Individual particles

Mesopores: at the initial location of large hemihydrate particles (not water voids!)

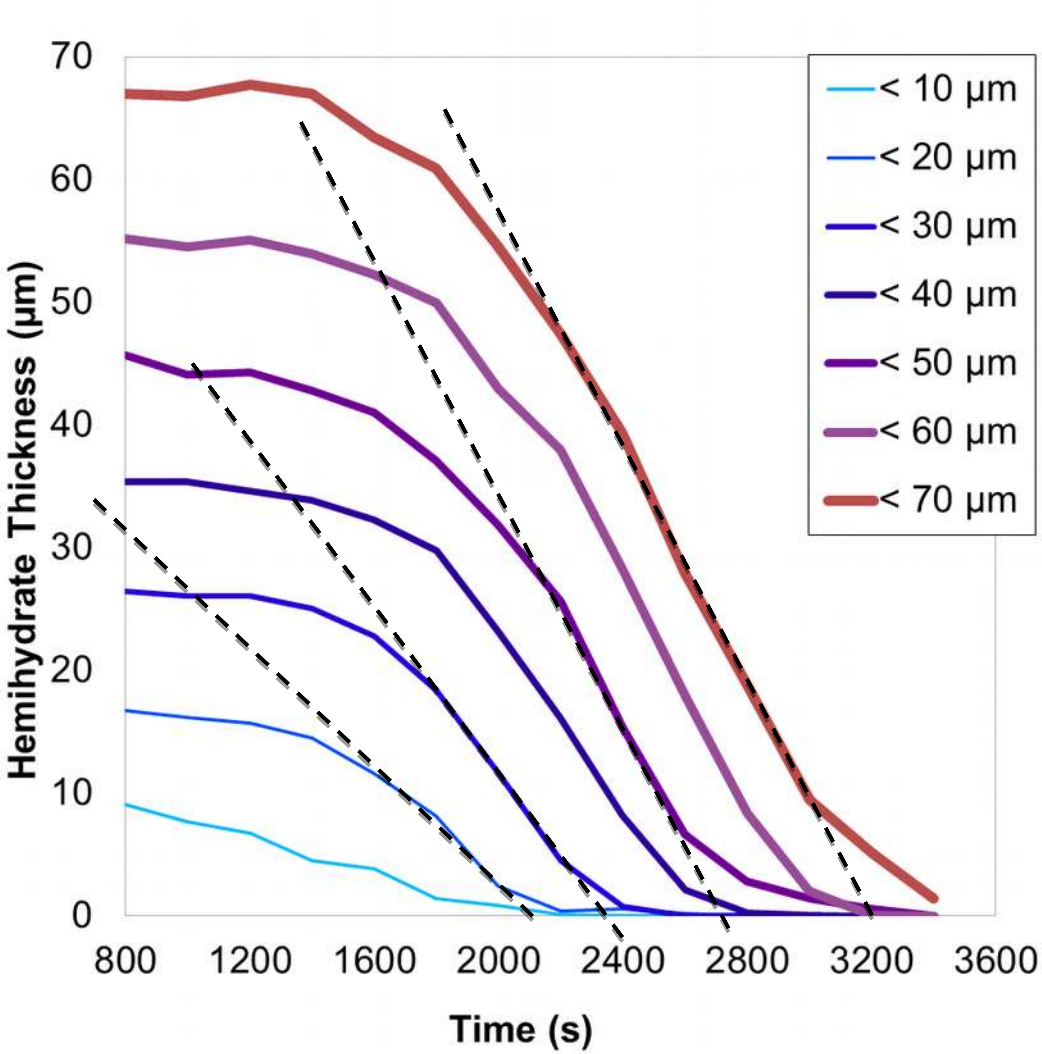
800 s 1200 s 1600 s 2000 s 2600 s 3200 s 6600 s





Influence of HH particle size

Statistical analysis of the dissolution of HH particles depending on their size (10 particles per size class) – calculation impossible below 20 µm



Thickness at 800s (µm)	dT/dt µm/min
< 20	0.84
< 30	1.08
< 40	1.14
< 50	1.14
< 60	1.5
< 70	1.5

Additional tests: powder granulometry

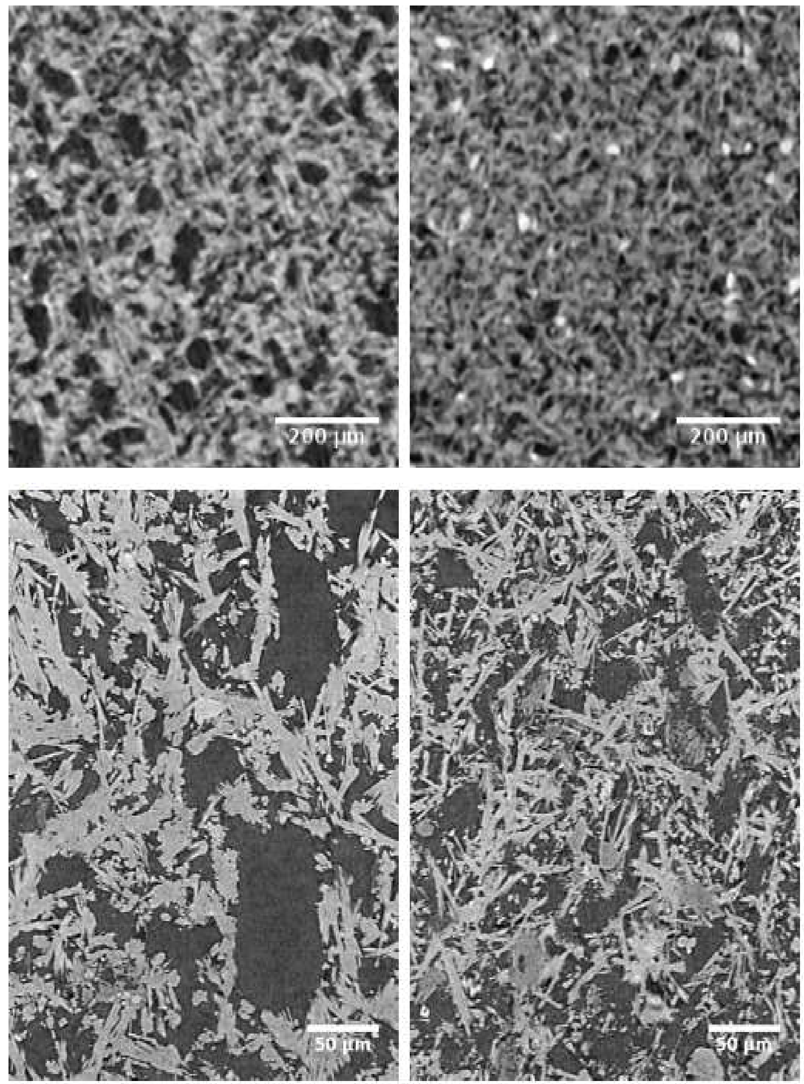
Sieving of HH powder to keep only the smallest particles (below 40 µm)

Microstructure **after hydration**, observed at two resolutions

Raw powder | Sieved powder

2.5 µm/voxel

200 µm



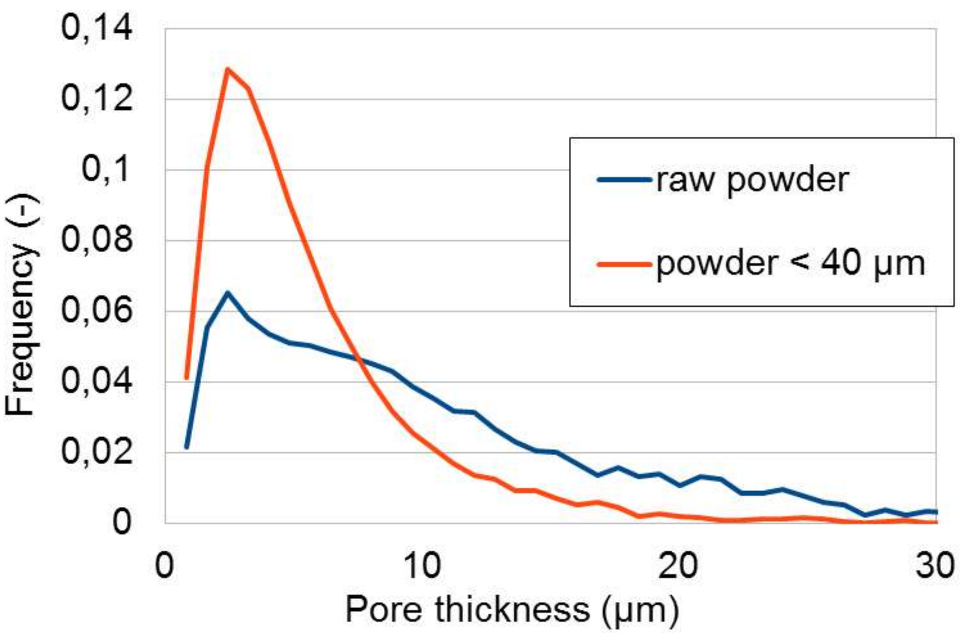
0.7 µm/voxel

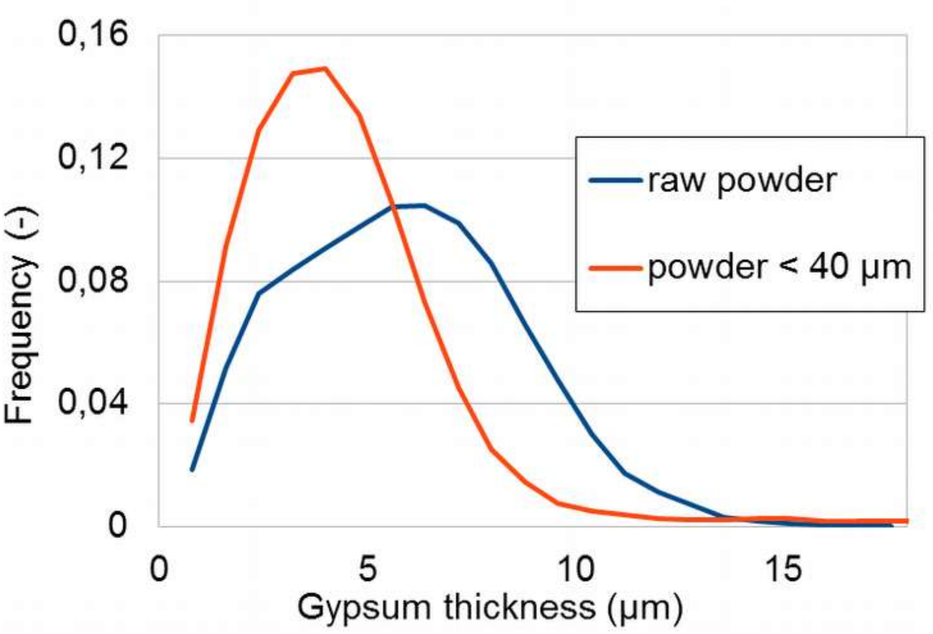
RX solution tomograph

100 µm

Influence of powder granulometry

Distribution of pore and solid phase thickness after hydration (from successive erosion/dilation)



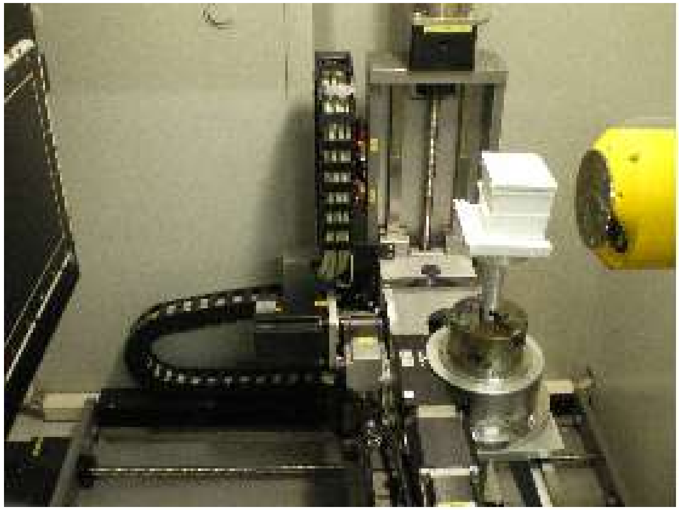


→ Possible tailoring of the microstructure

X-ray tomographs

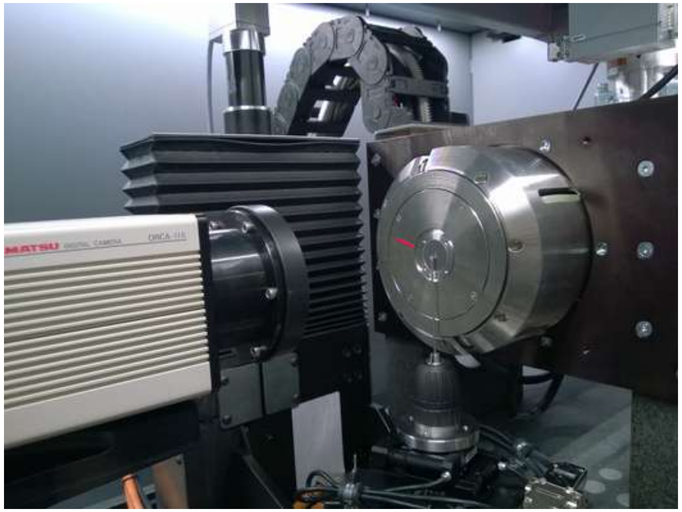
Low flux, conical beam

High acquisition speed
Phoenix V tome X

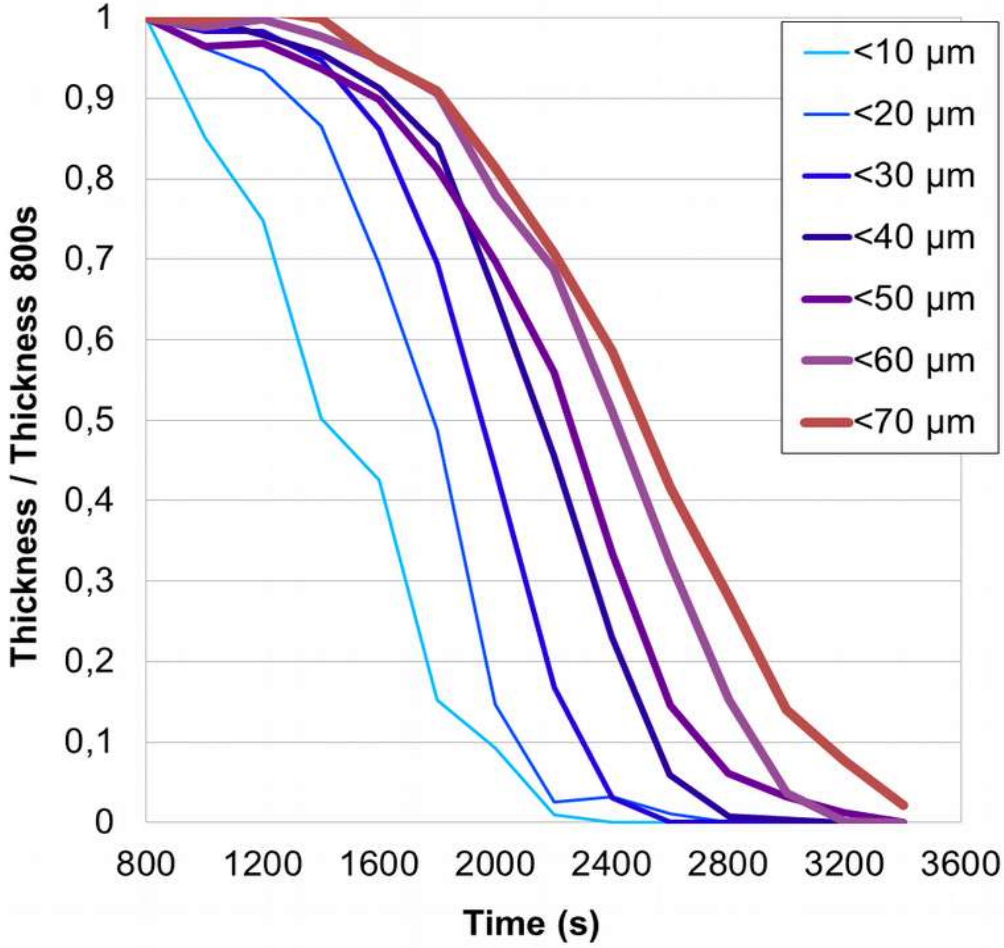


Voxel size from 1 to 100 µm
Acquisition time from hours to several minutes

High spatial resolution
RX Solutions EasyTom Nano

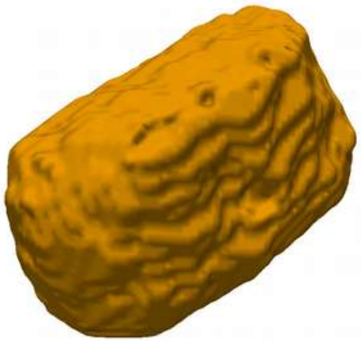


Voxel size min 0,25 µm
Acquisition time : several hours at maximal resolution

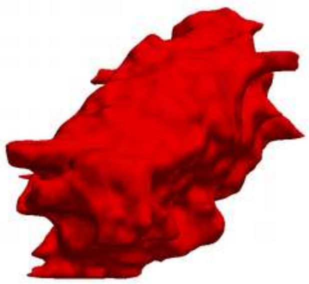


Thickness at 800s (µm)	dT/dt µm/mi n	Incubati on time (s)
< 20	0.84	1500
< 30	1.08	1600
< 40	1.14	1690
< 50	1.14	1780
< 60	1.5	1820
< 70	1.5	1820

Initial HH particle



Final mesopore



Most recent results

Tomcat 0.16 µm pixel size
1 minute acquisition time

β plaster larger particles

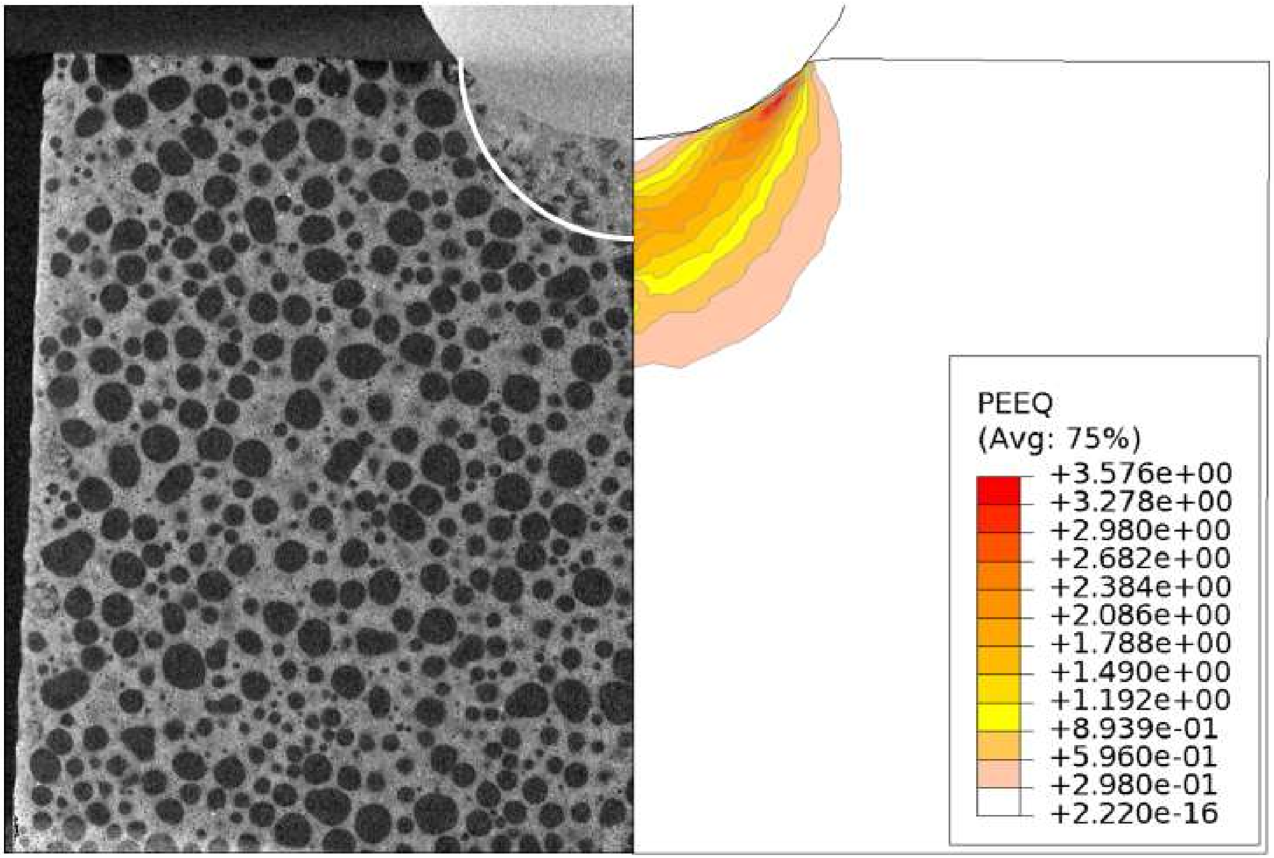
β plaster fine particles

α plaster

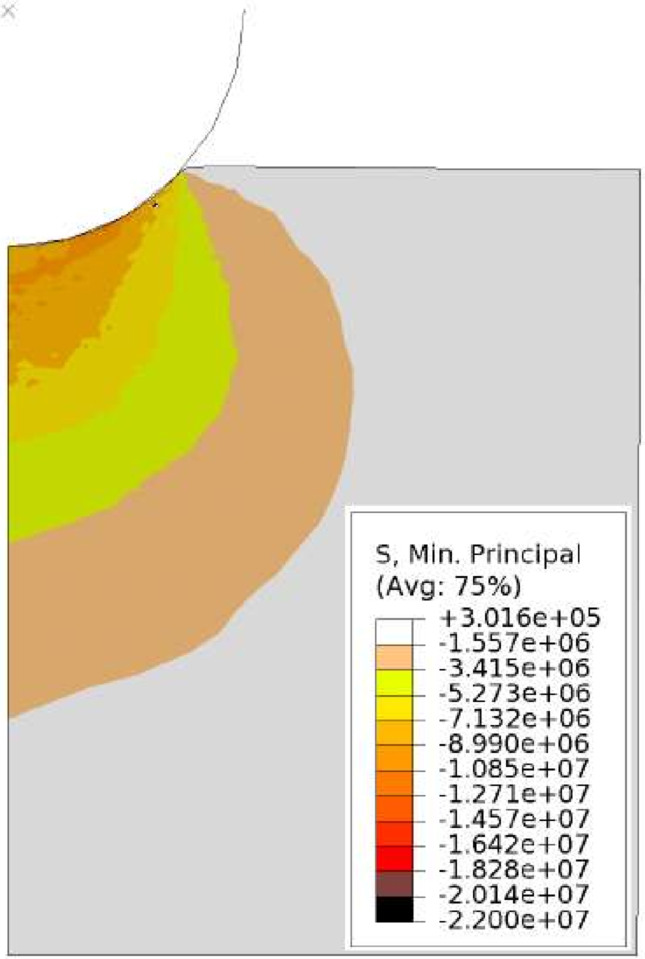
Deformation modes

+DVC
+FE modelling

Bouterf, Adrien, Hild, Roux, Maire



(a) Equivalent plastic strain



(b) Minor eigen stress

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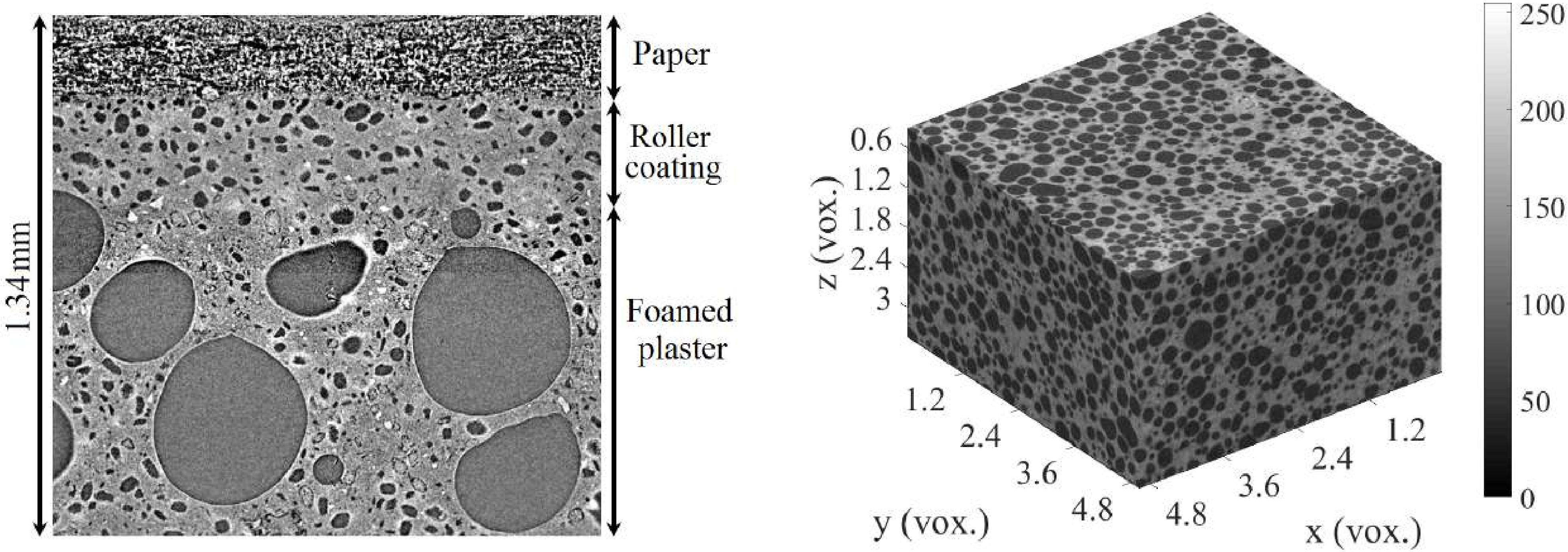
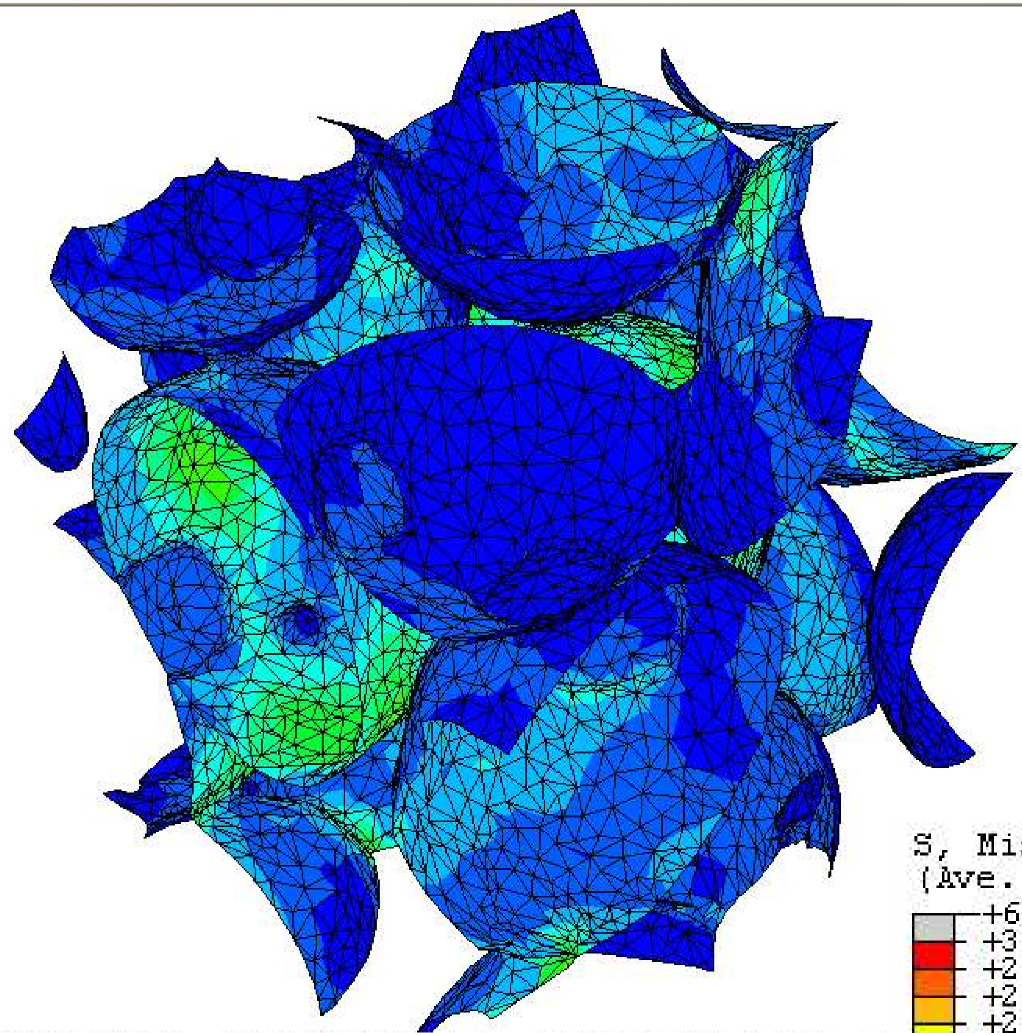


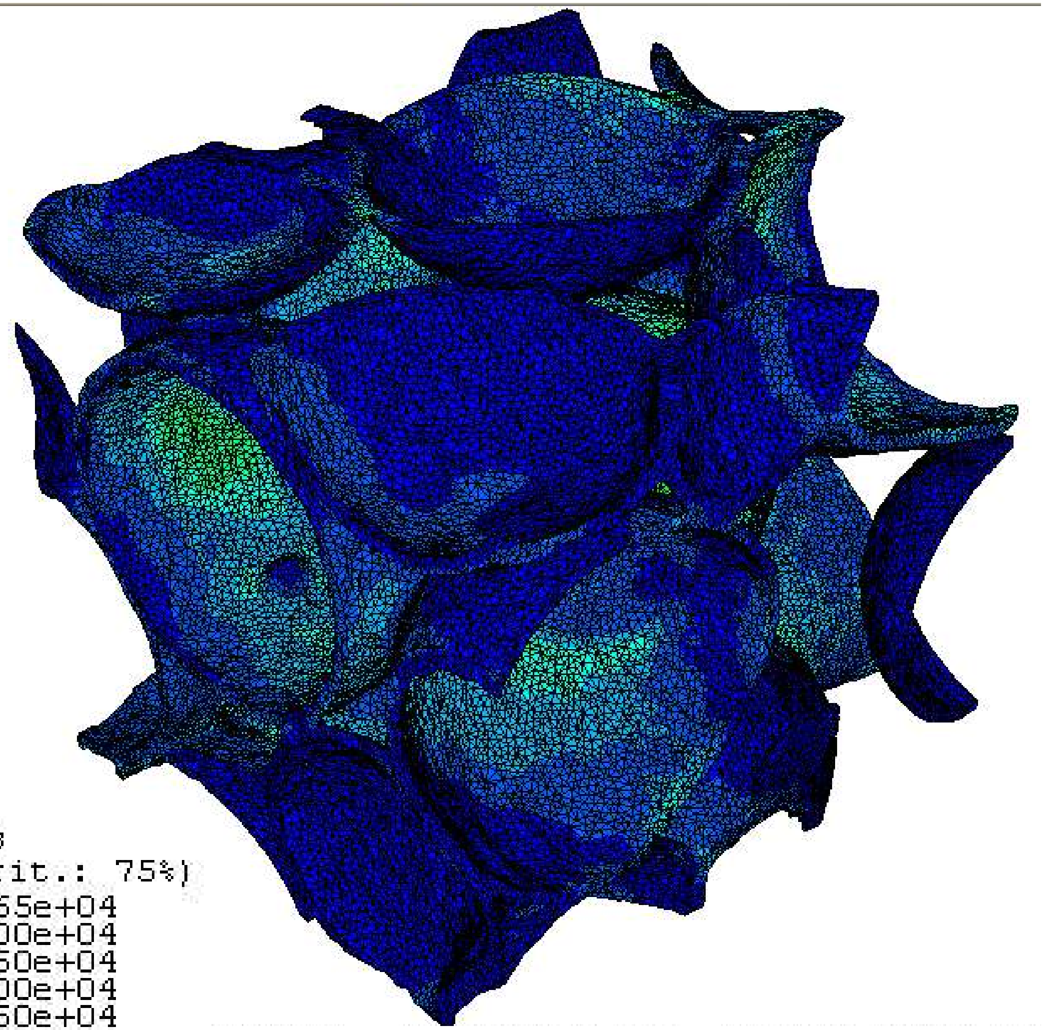
(a) (b)

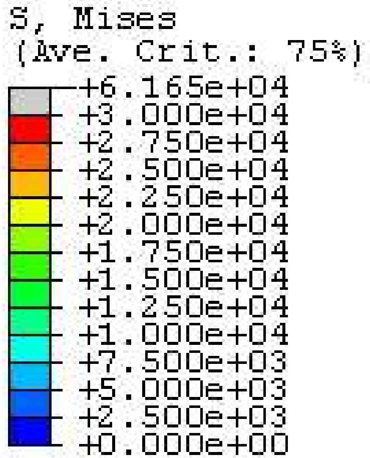
Figure 1: (a) Tomographic section showing the microstructure observed at a resolution of 1.4 μm of the studied plaster whose porosity is of the order of 75 %. The size of the section is 1.34×1.4 mm^2. (b) 3D rendering of microstructure of the core material studied herein observed at a resolution of 12 μm. The size of the section is 4.8×4.8 mm^2

Modelling

Shell elements the thickness of which is dictated by the measurement







- Calcul en élasticité linéaire
- 7490 éléments coques triangulaires linéaires
- Durée calcul 11 s

- Calcul en élasticité linéaire
- 428000 éléments tétraèdriques linéaires
- Durée calcul 114 s

Conclusion

- A lot of new things to learn from 3D X Ray imaging of construction materials
- The materials work well with the technique (pores, phases)
- Dynamic studies (setting, mechanical loading ...)