INSTITUT NATIONAL DES SCIENCES APPLIQUÉES LYON

X-ray in situ tomography with extreme conditions

C. Le Bourlot, E. Maire, J.Y. Buffière, W. Ludwig,
J. Adrien, J. Lachambre, S. Dancette, D Fabrègue,
and much more…

[1]MATEIS, INSA, 20 avenue A. Einstein 69100 Villeurbanne, France

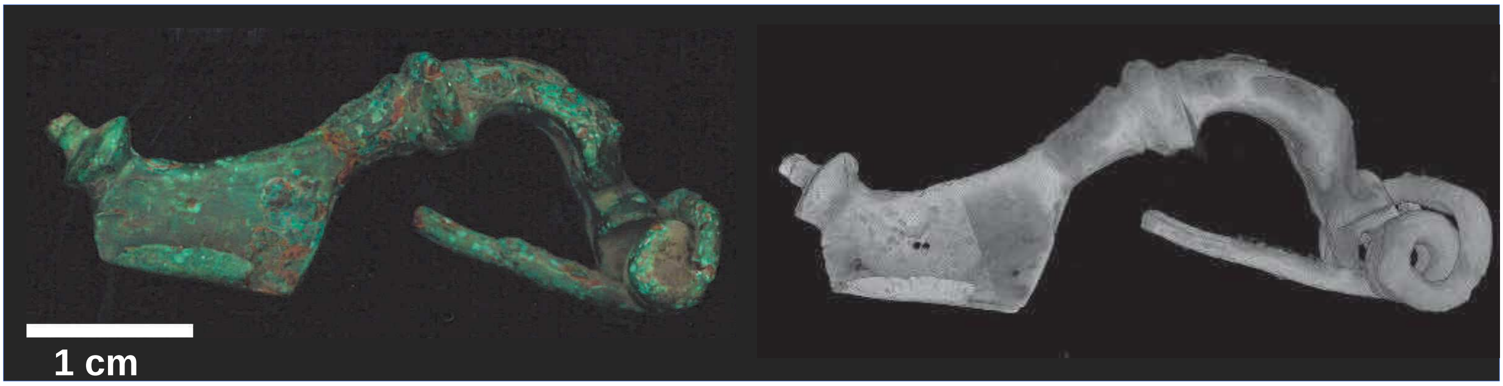
"couplages oxydation-mécanique"
Compiègne les 5 et 6 Juin 2019

membre de UNIVERSITÉ DE LYON

Tomography – full field approach

from Ancient Greek

τόμος tomos: slice, section
γράφω graphō: to write

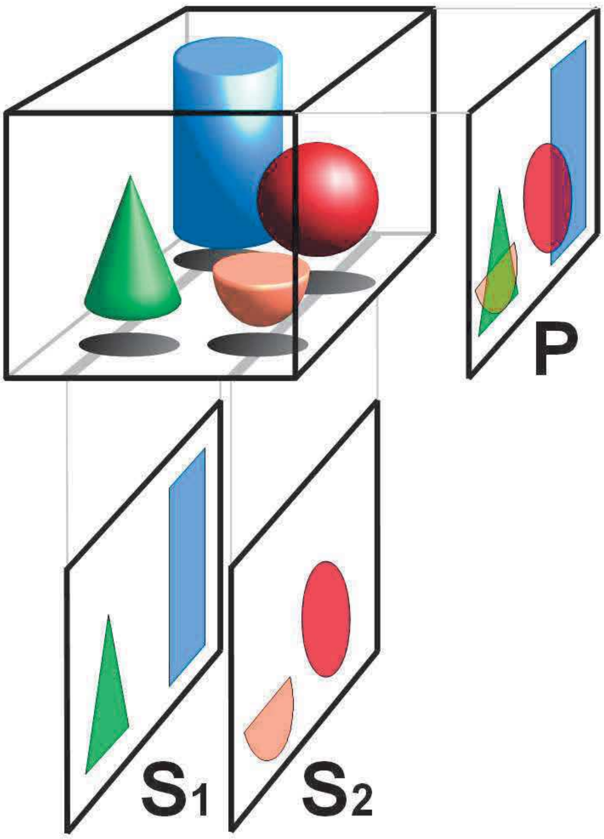


Allan M. Cormack (South Africa)
Sir Godfrey N. Hounsfield (United Kingdom)

"for the development of computer assisted tomography" (CT)
1979 Nobel Prize for Physiology or Medicine

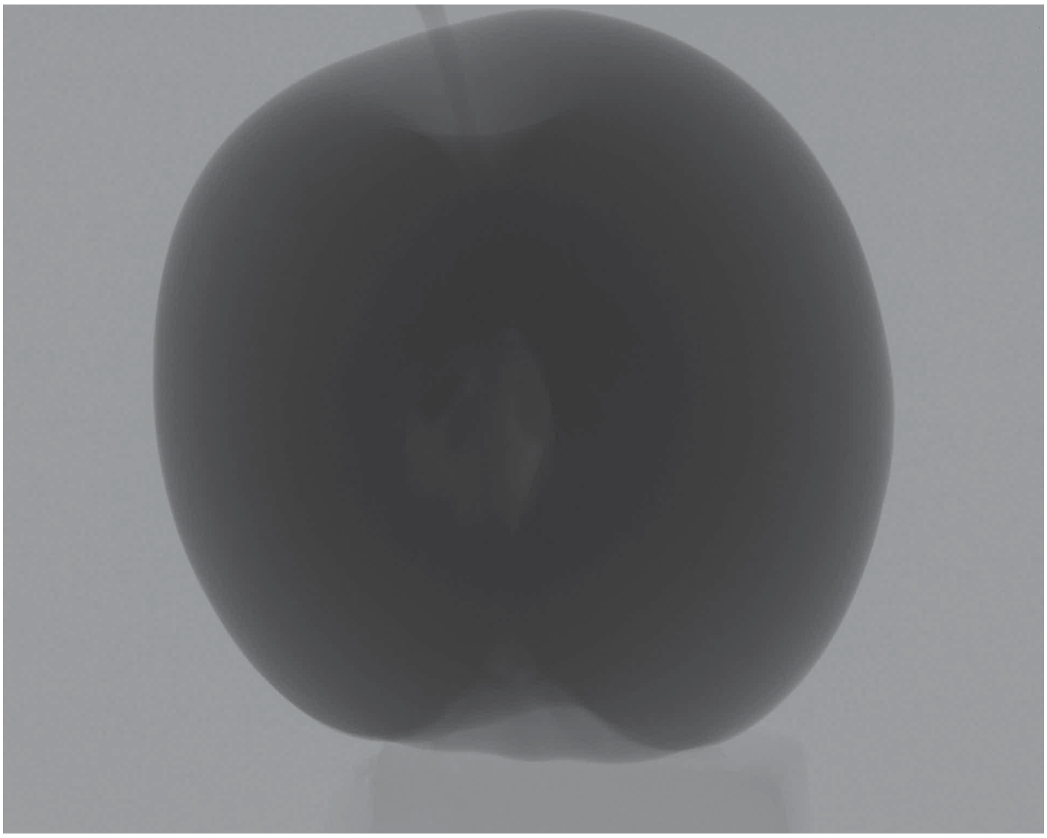
Tomography

refers to imaging by sections or sectioning, through the use of any kind of penetrating wave.

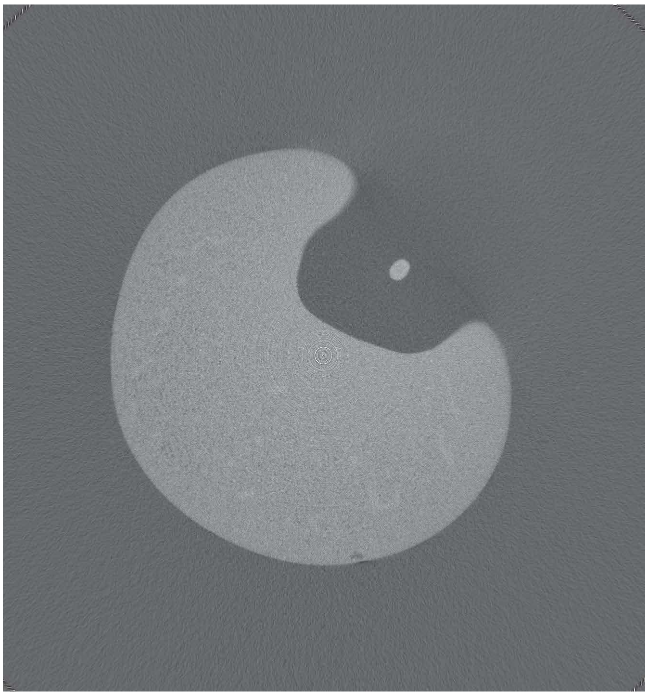


"Tomography Principle Illustration" by Dtrx. (wikipedia)

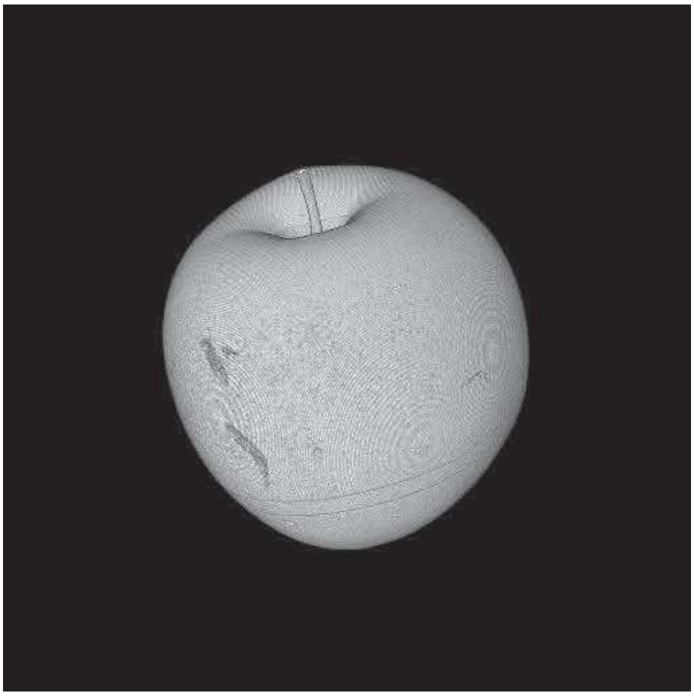
Tomography - example



Tomography - example

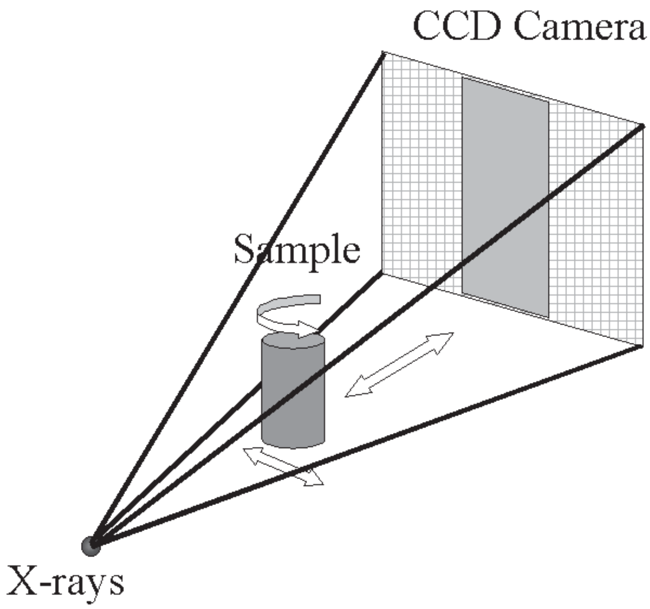


Reconstruction

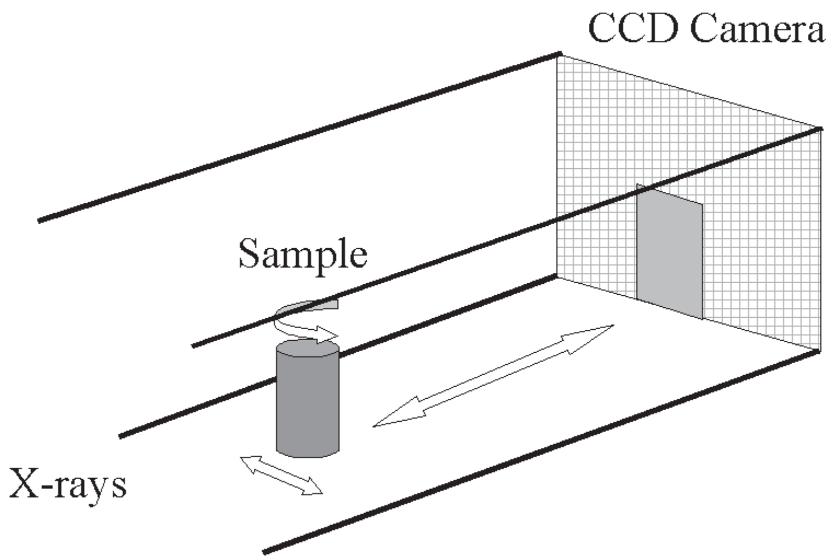


Segmentation

About tomography - geometries

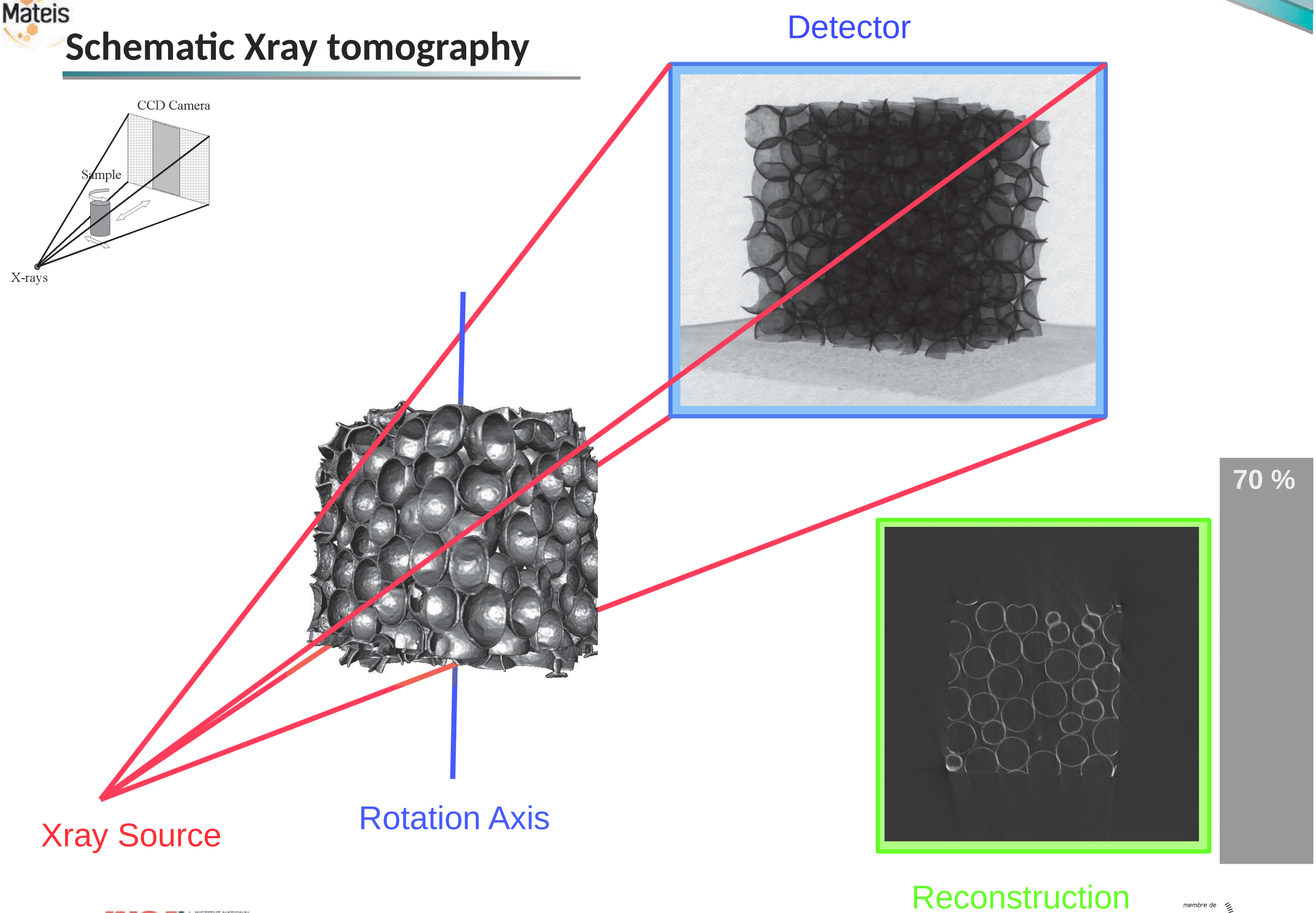


Laboratory

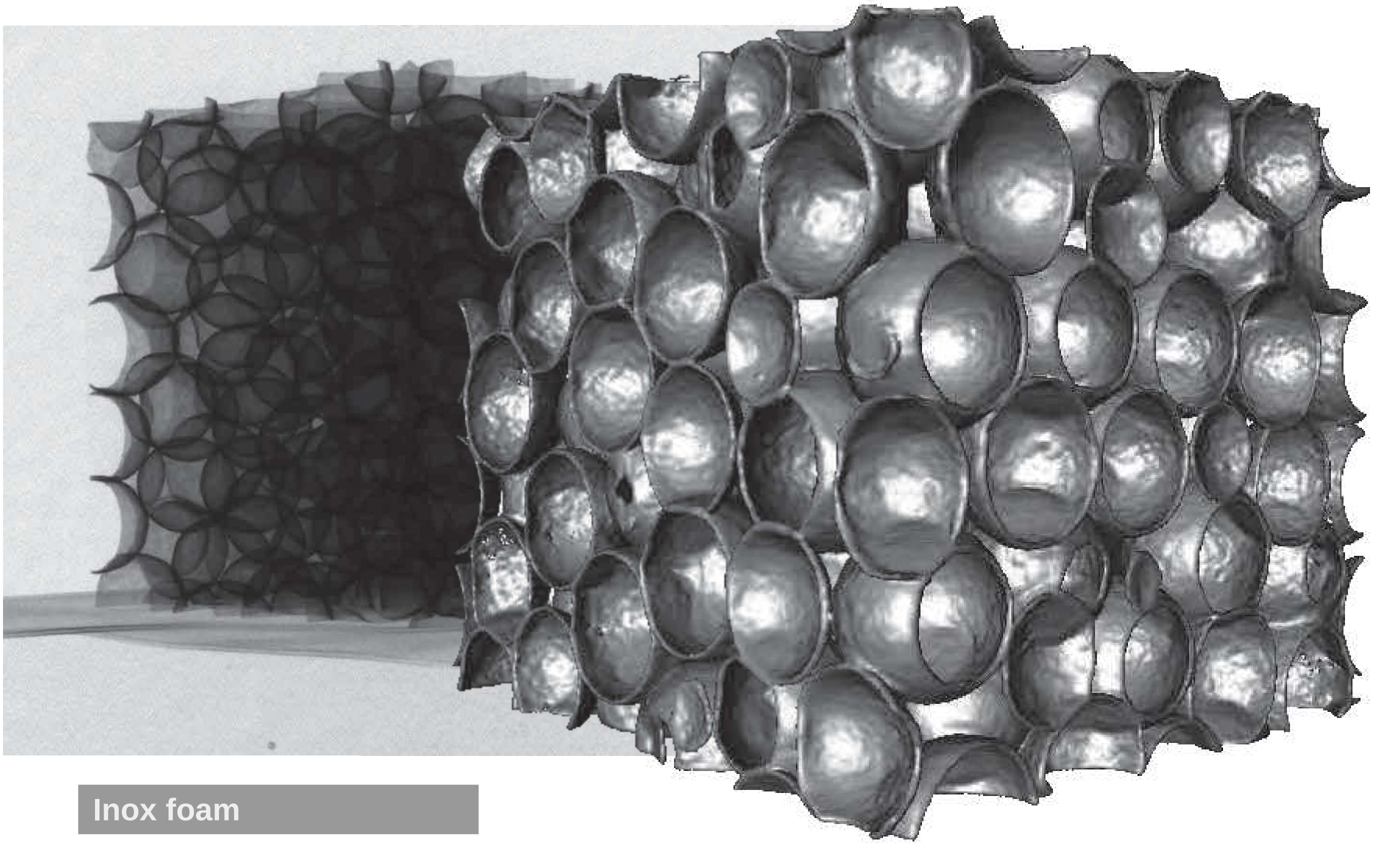


Synchrotron

Schematic Xray tomography



Xray tomography reconstruction



Inox foam

Mateis

2D reconstruction : simple case

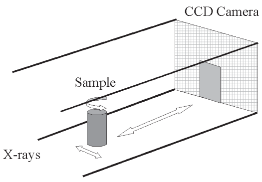
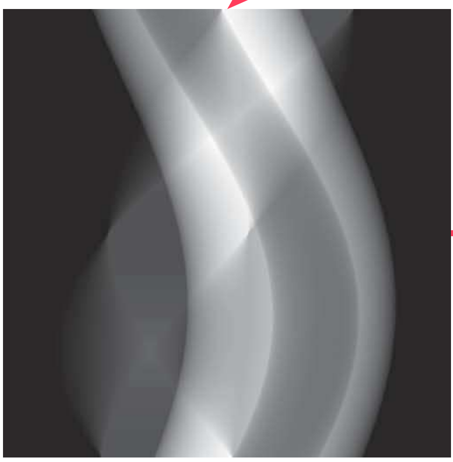
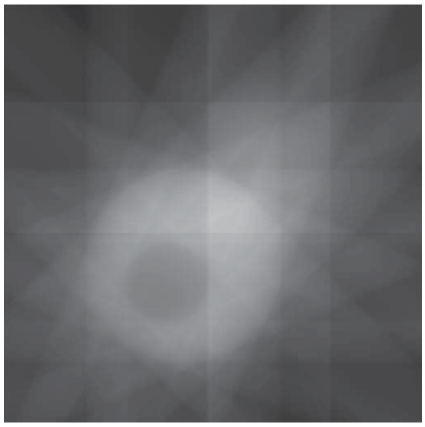


Image slice

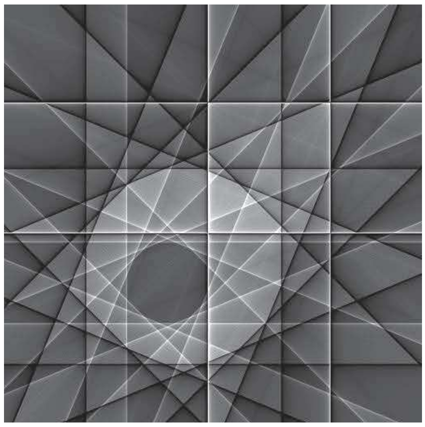


sinograme

Back projection



Filtered back projection

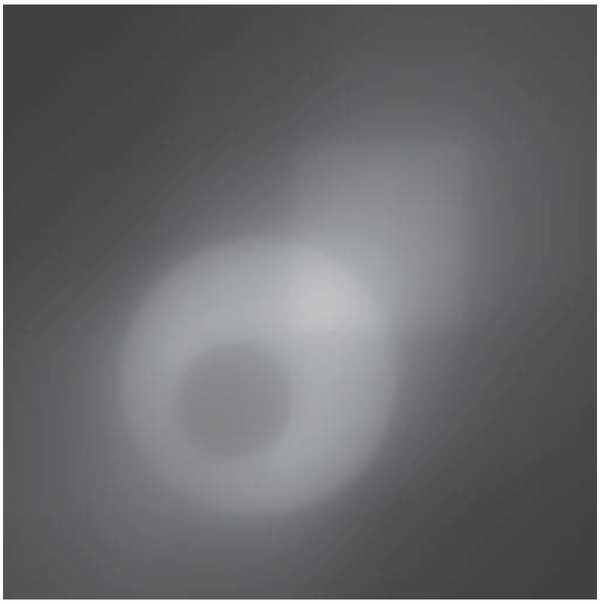


Courtesy of J. Lachambre @MATEIS

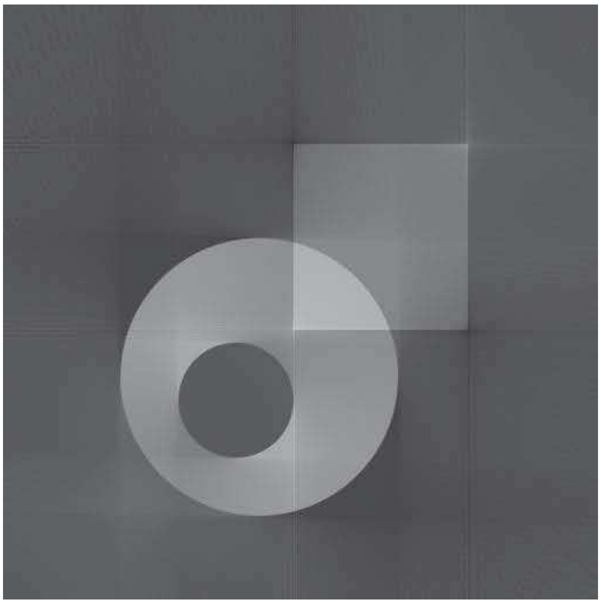
2D reconstruction : simple case

Image slice

Back projection

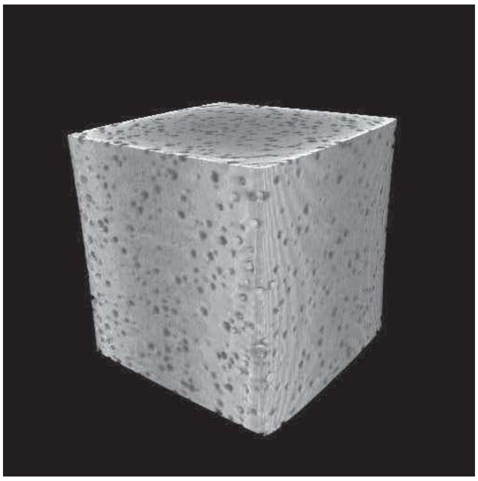


Filtered back projection



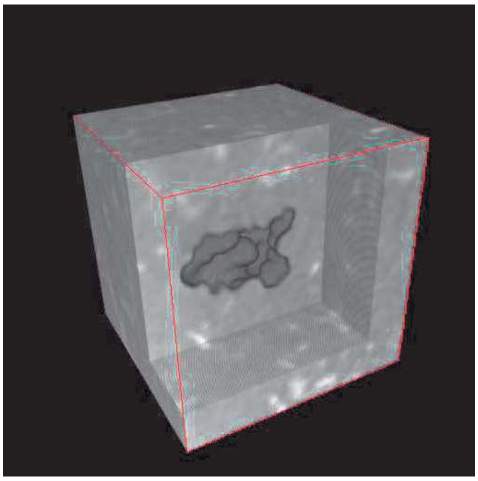
Courtesy of J. Lachambre @MATEIS

Xray tomography - examples



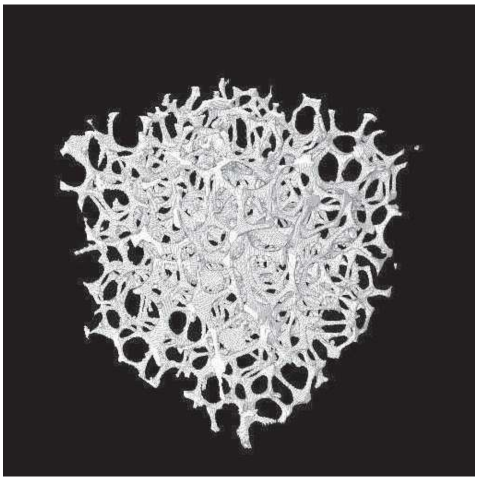
Cast iron

Graphite inclusion



Cast Al

Large porosity

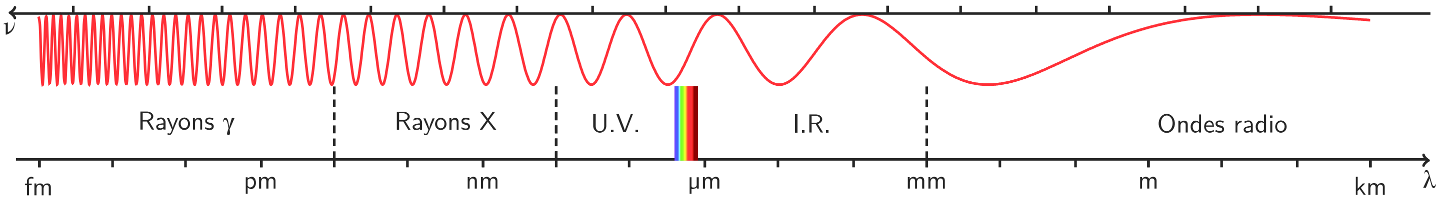


Al foam

Open cell porosity

Courtesy of J. Lachambre @MATEIS

About X rays



> Wikipedia - Spectre électromagnétique

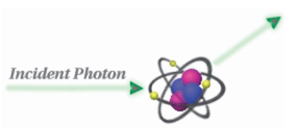
W. C. Röntgen : Über eine neue Art von Strahlen [a]
Comptes-rendus des réunions de la Société physico-médicale de Würzburg, 28 décembre 1895.
Der Kürze halber möchte ich den Ausdruck "Strahlen" und zwar zur Unterscheidung von auderen den Namen "X-Strahlen" gebrauchen. [b]

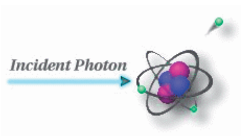
a. Sur une nouvelle sorte de rayons
b. Afin d'être bref, j'utiliserai le terme "rayons", et pour les distinguer d'autres du même nom, je les appellerai "rayons X"

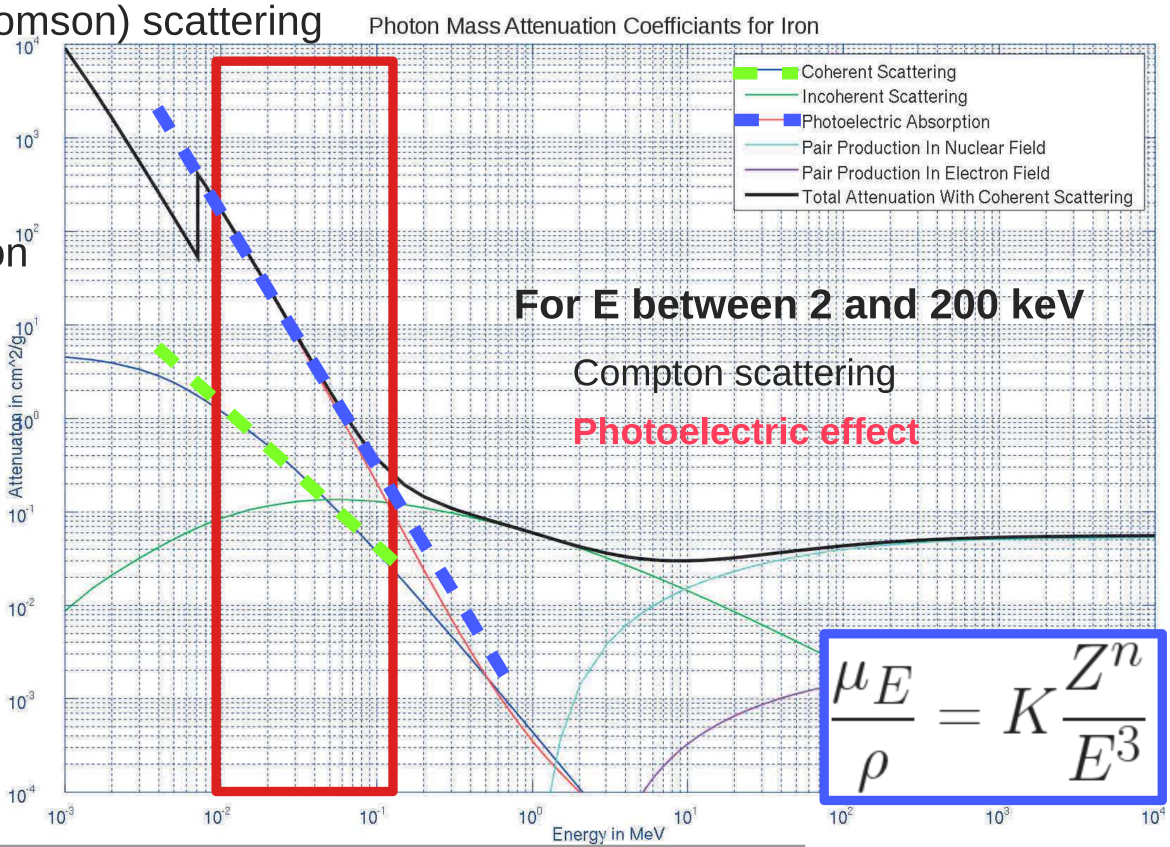
> X-ray radiograph of Wilhelm Conrad Roentgen's wife (1895)

Physical origin of attenuation

- Rayleigh (Thomson) scattering
- Compton scattering
- Photoelectric effect
- Pair anihilation







$$\frac{\mu_E}{\rho} = K\frac{Z^n}{E^3}$$

http://www.nist.gov/pml/data/xraycoef/index.cfm

Case of neutrons…

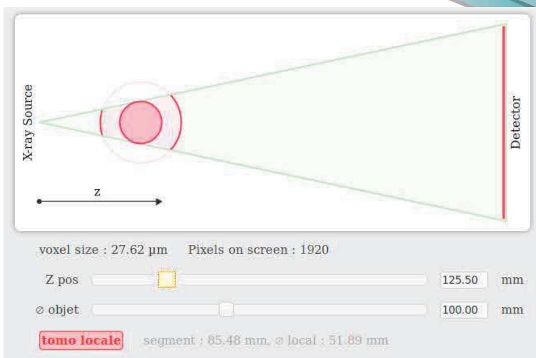
Mateis

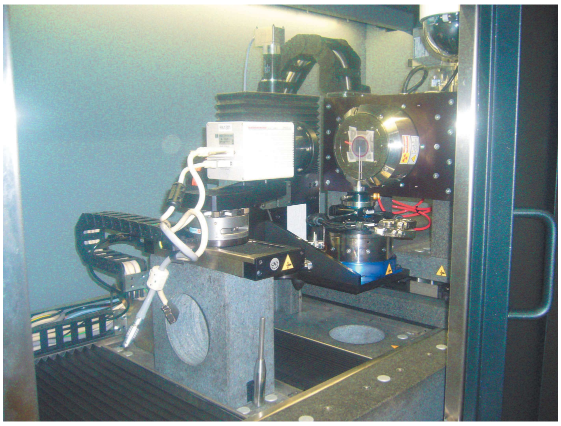
Tomographes – Laboratoire vs. Synchrotrons

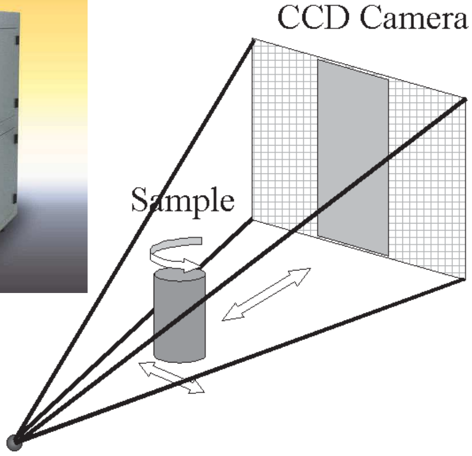
- Phase contrast, high flux, availability …

Tomographes – Laboratoire vs. Synchrotrons

- Résolution mini : 0,3 µm
- Conic beam
- Acquisition time : from few minutes to few hours



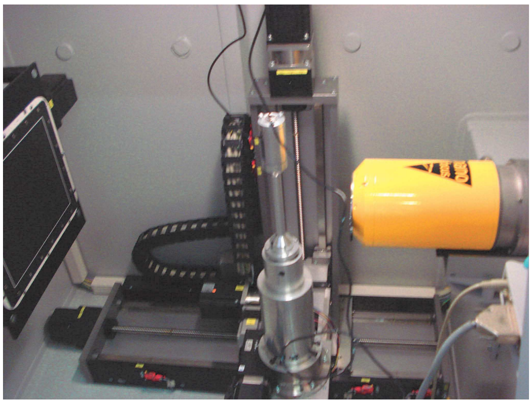




> Plateforme tomographique, laboratoire MATEIS

in situ machines

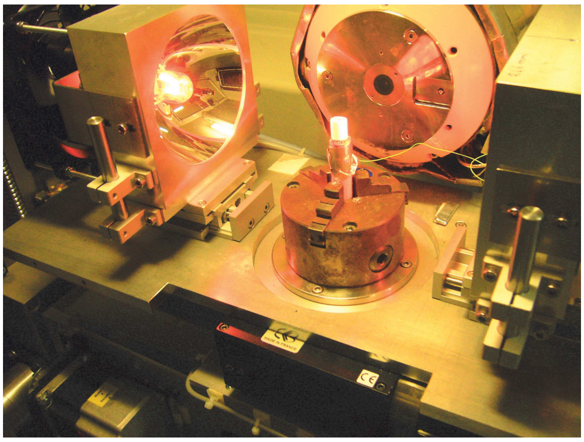
> Traction/Compression



> Hight speed

> Hot T° Traction

> Ovens

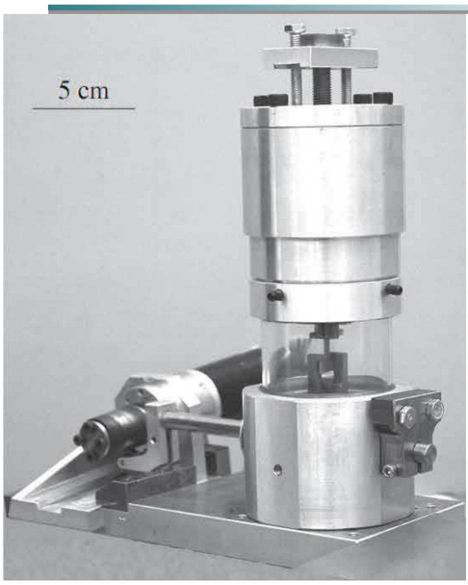


> Cryo

> Peltier

in situ machines

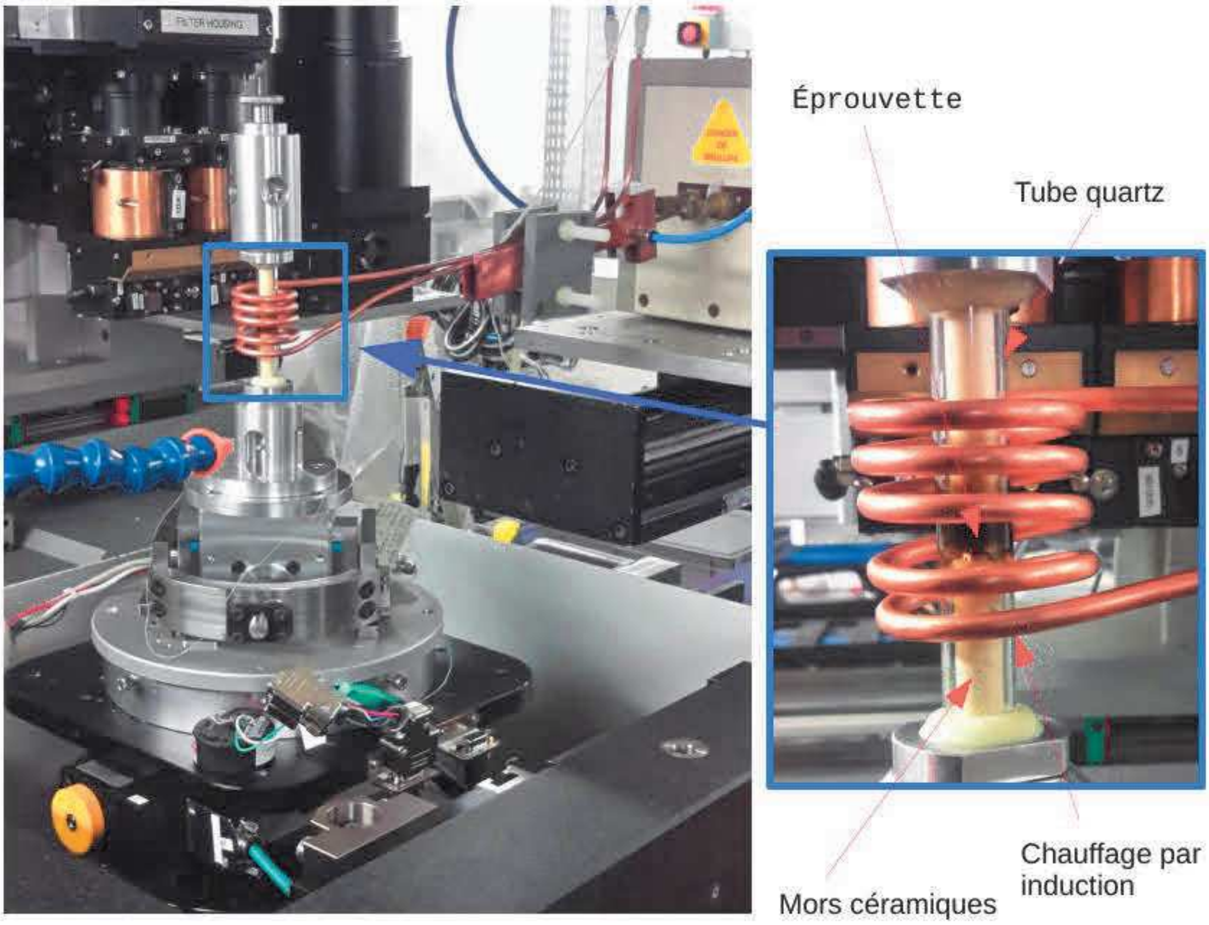
> Fatigue



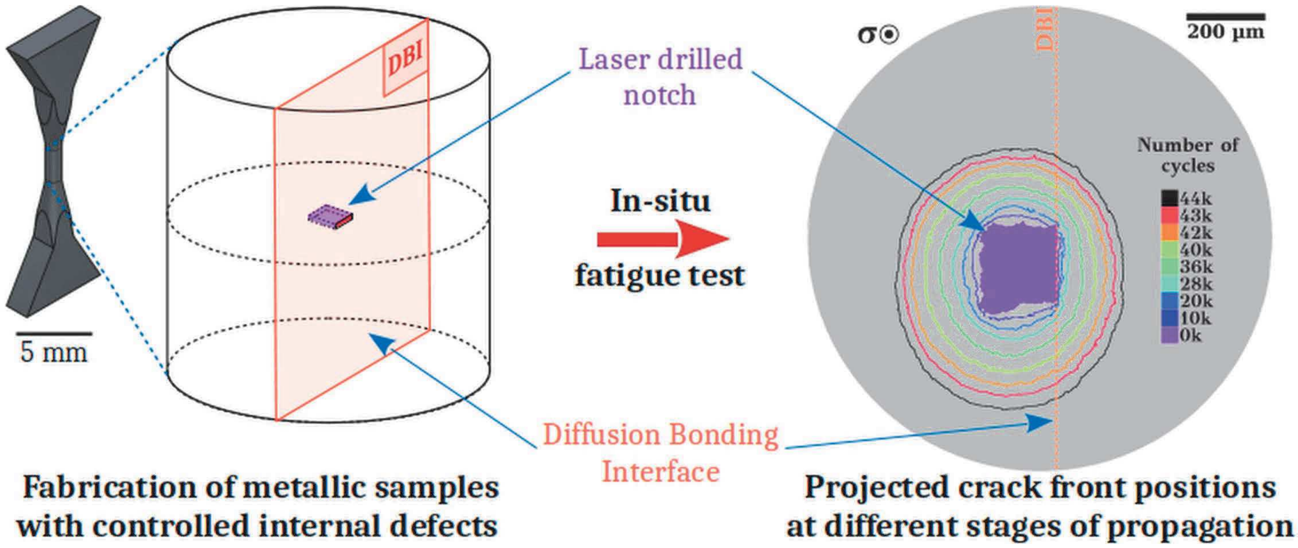
[Buffière et al. Mat.Sc. Tech. 2006]

[Lachambre *et al.*
Proc. Soc. for Exp. Mech. Springer 2013]

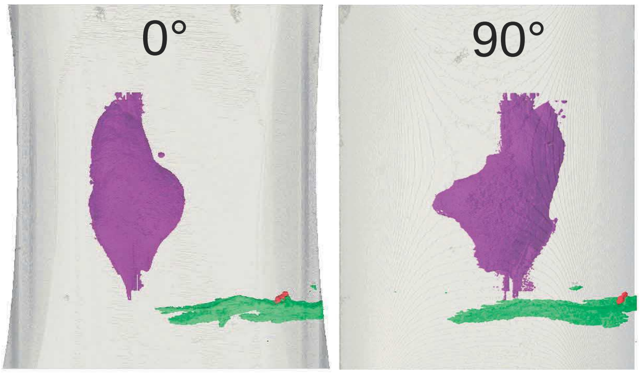
in situ machines



in situ experiment

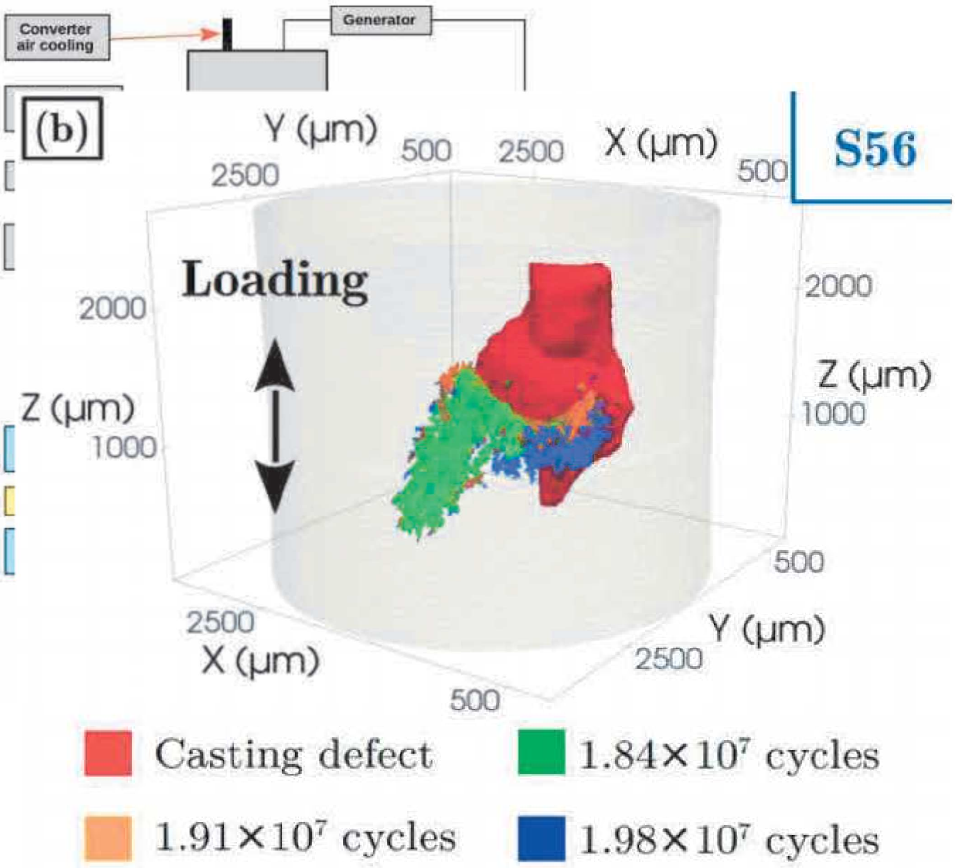


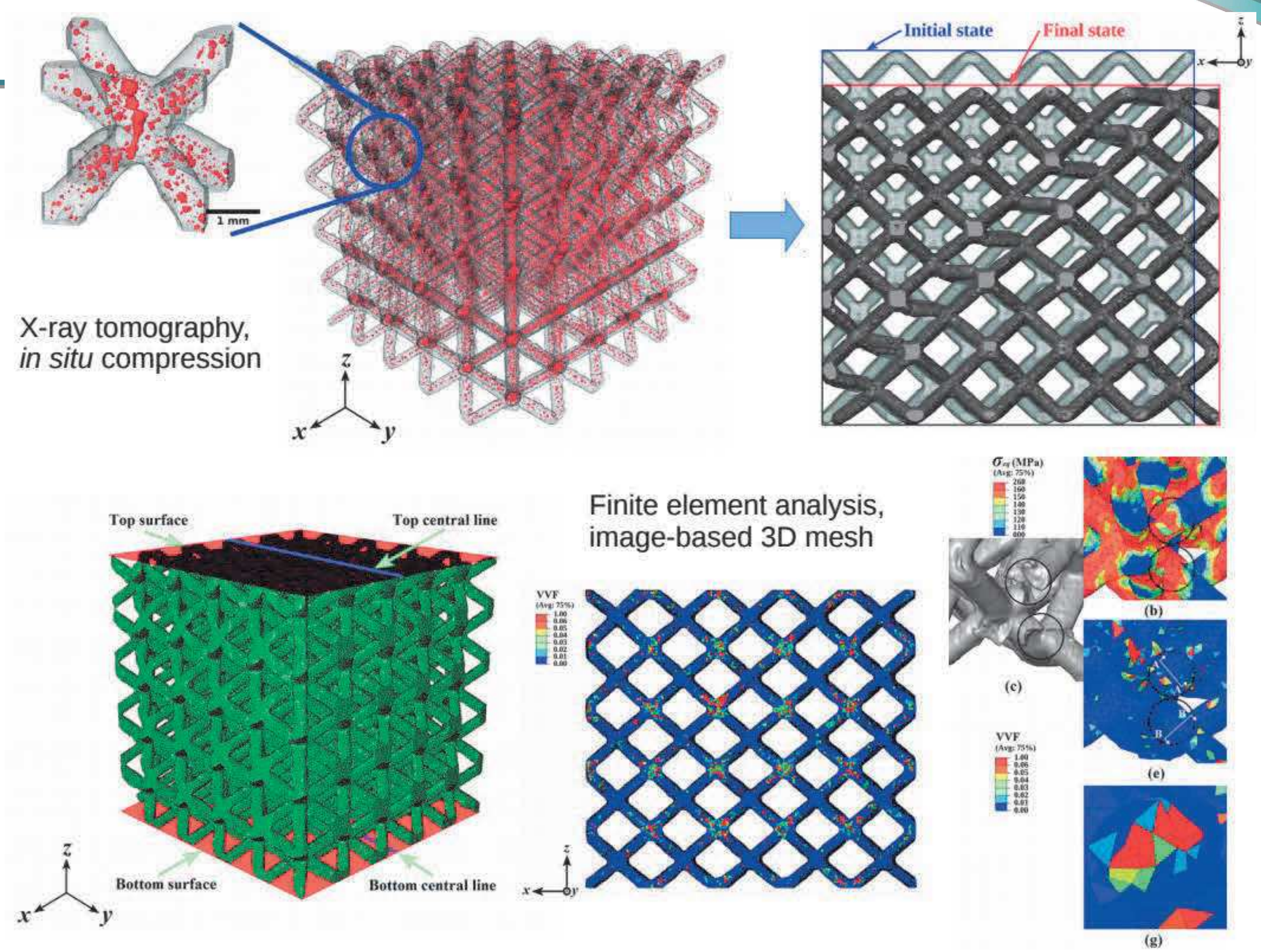
370 000 cycles



Mateis

Fatigue giga-cycles




1 mm
X-ray tomography,
in situ compression
Initial state
Final state
Finite element analysis,
image-based 3D mesh
Top surface
Top central line
Bottom surface
Bottom central line
VVF
(b)
(c)
(e)
(g)

Porous metals

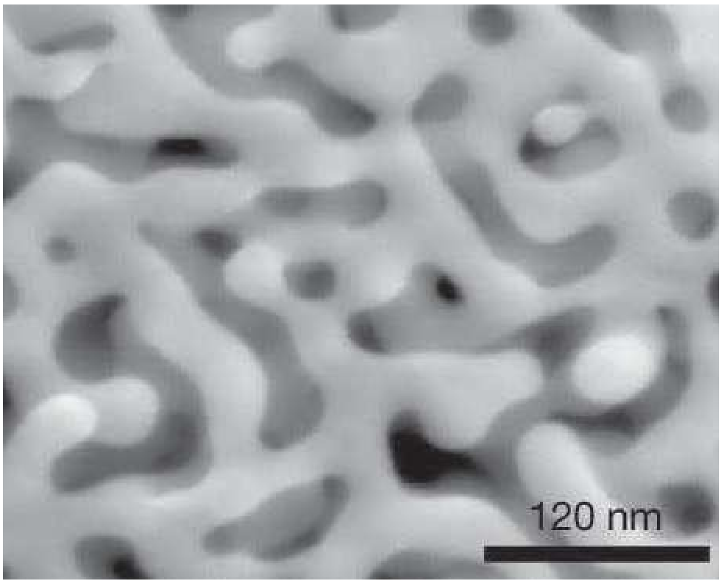
Morgane Mokhtari PhD

Mechanicals properties

Applications : light-weighting

High specific surface

Applications : catalyst, sensor, filtration, electrode for battery and capacitor, energy harvesting



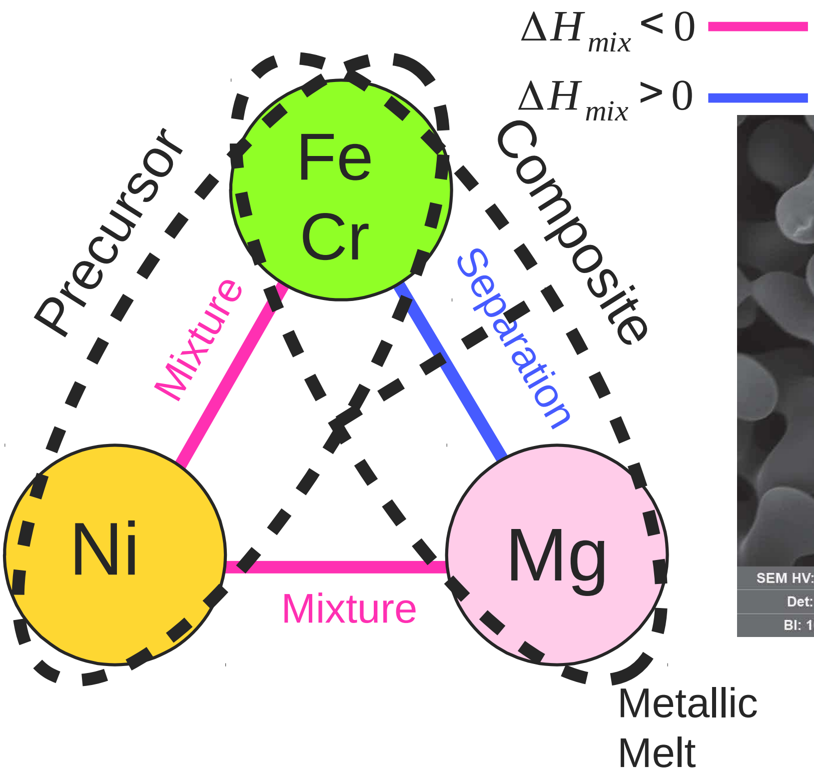
Nanoporous gold elaborated from $Au_{26}Ag_{74}$

J. Erlebacher et al., Nature 410 (2001) 450-451

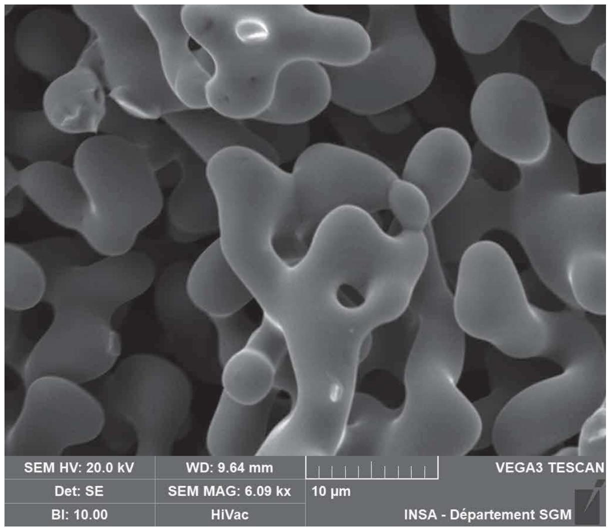
How can we deal with common metals ?

Liquid metal dealloying

Liquid metal dealloying principle

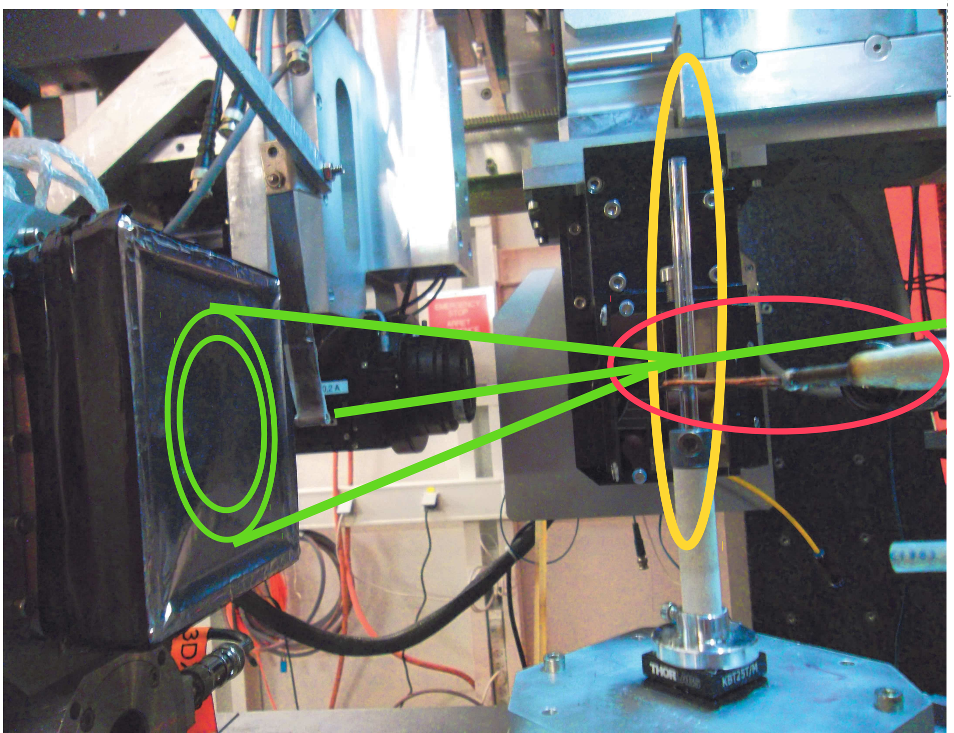


Dealloying Triangle

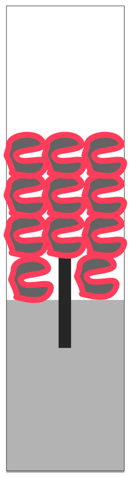


In situ dealloying – X-ray diffraction

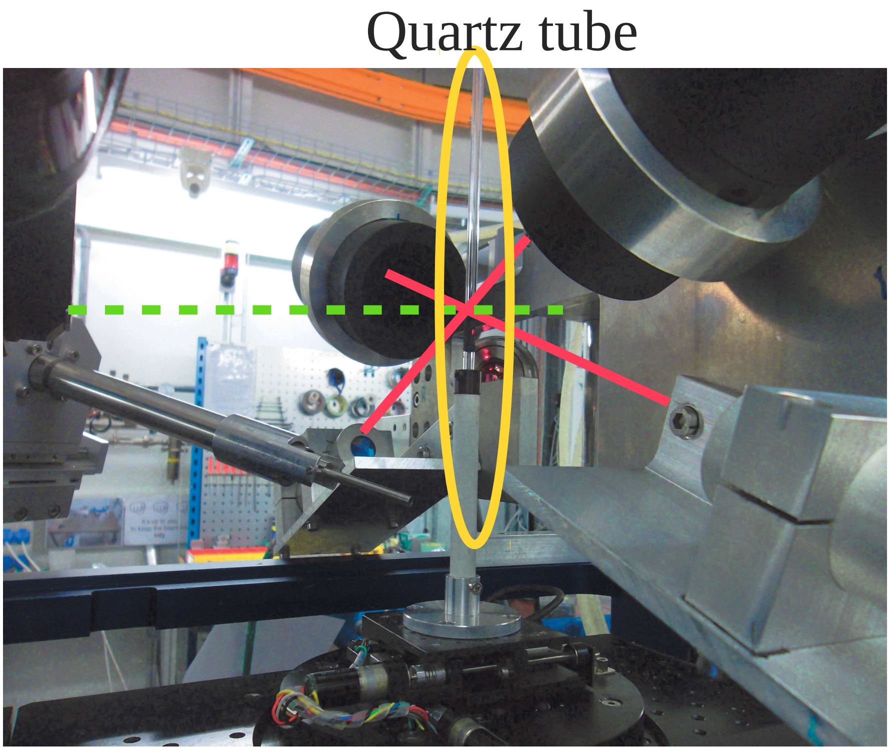
Quartz tube



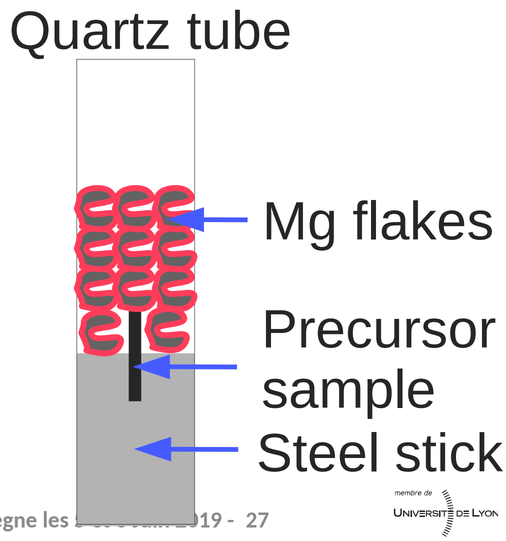
Experimental parameters
E=35.1 keV
ID 11 ESRF



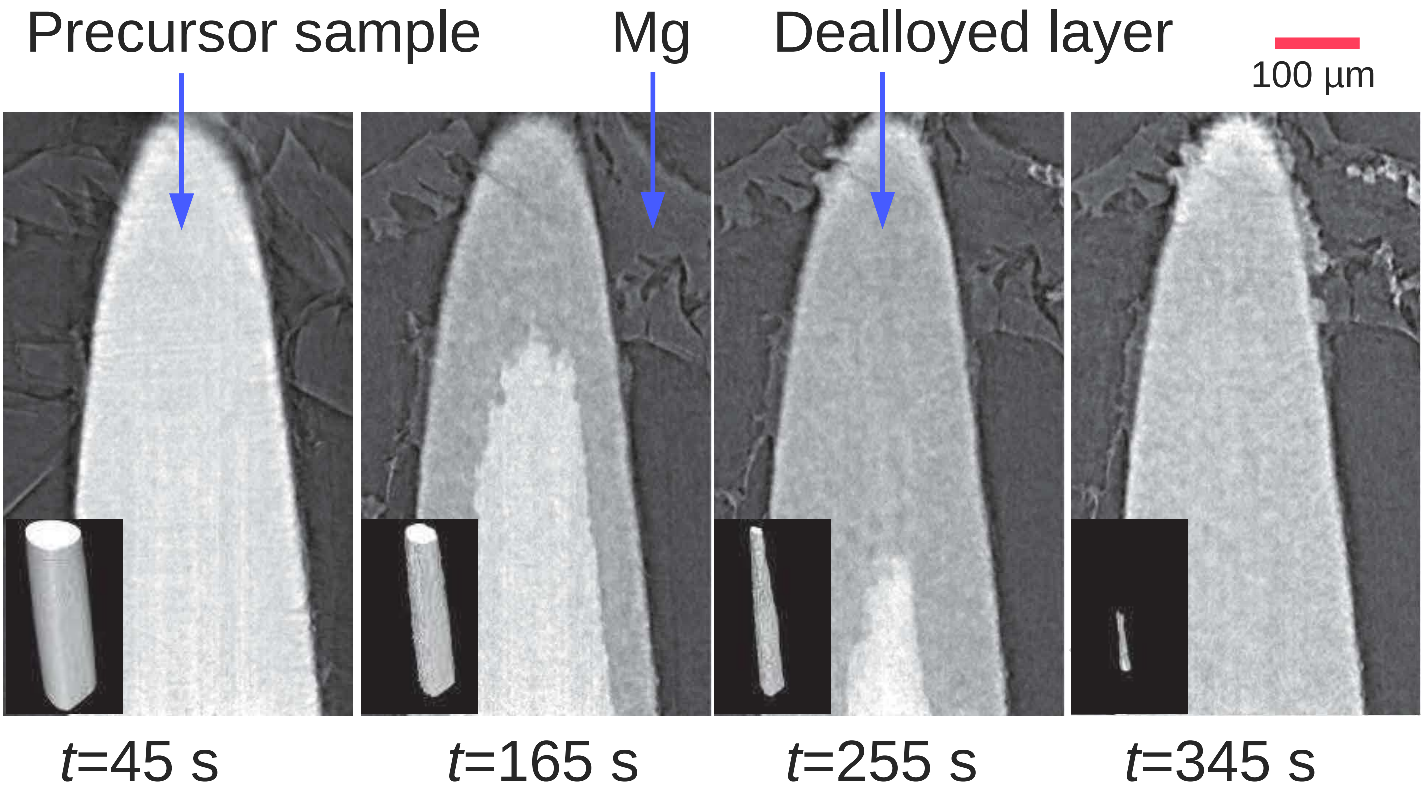
In situ dealloying



Experimental parameters
- E=25keV
- Projections : 1001
- Exposure time : 20 ms
- Voxel size : 0.32 µm

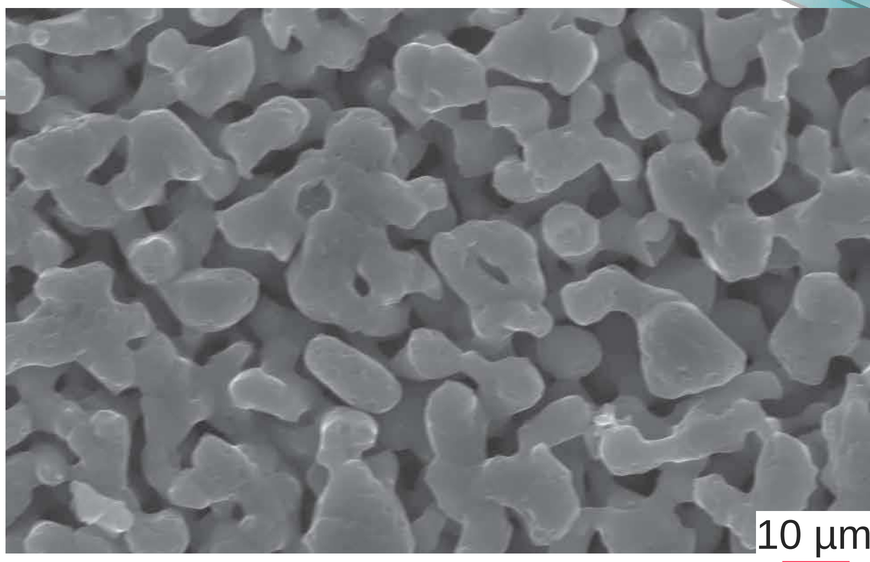


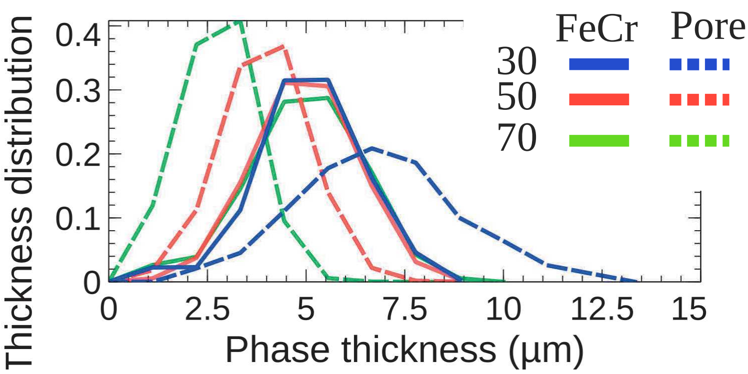
In situ dealloying - Experiment



Foam morphology

Porosity ~ at% Ni
in the precursor





Specific surface
S ~4. 10^5 m²/m3

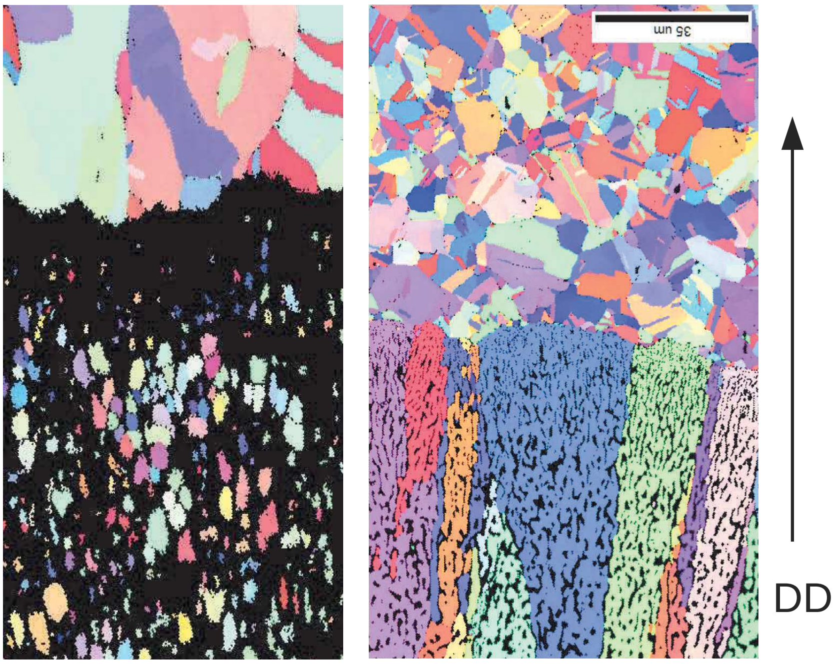
$$S = \frac{C}{\rho * d}$$

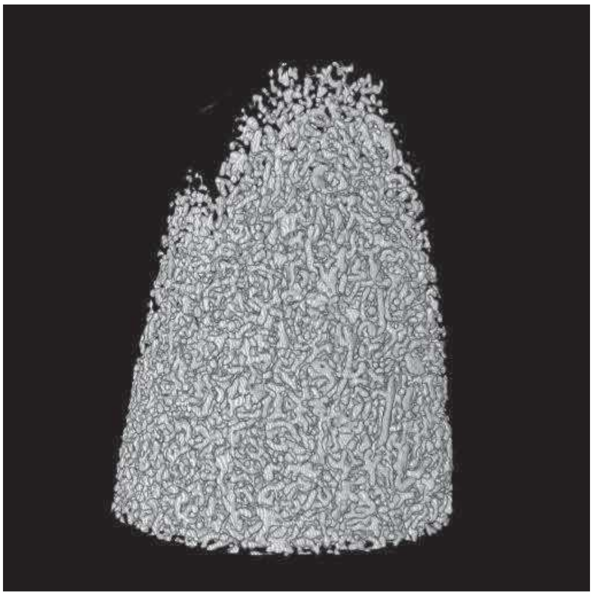
C= 3.7

E. Detsi et al. Act. Mater. 59 (2011) 7488–7497

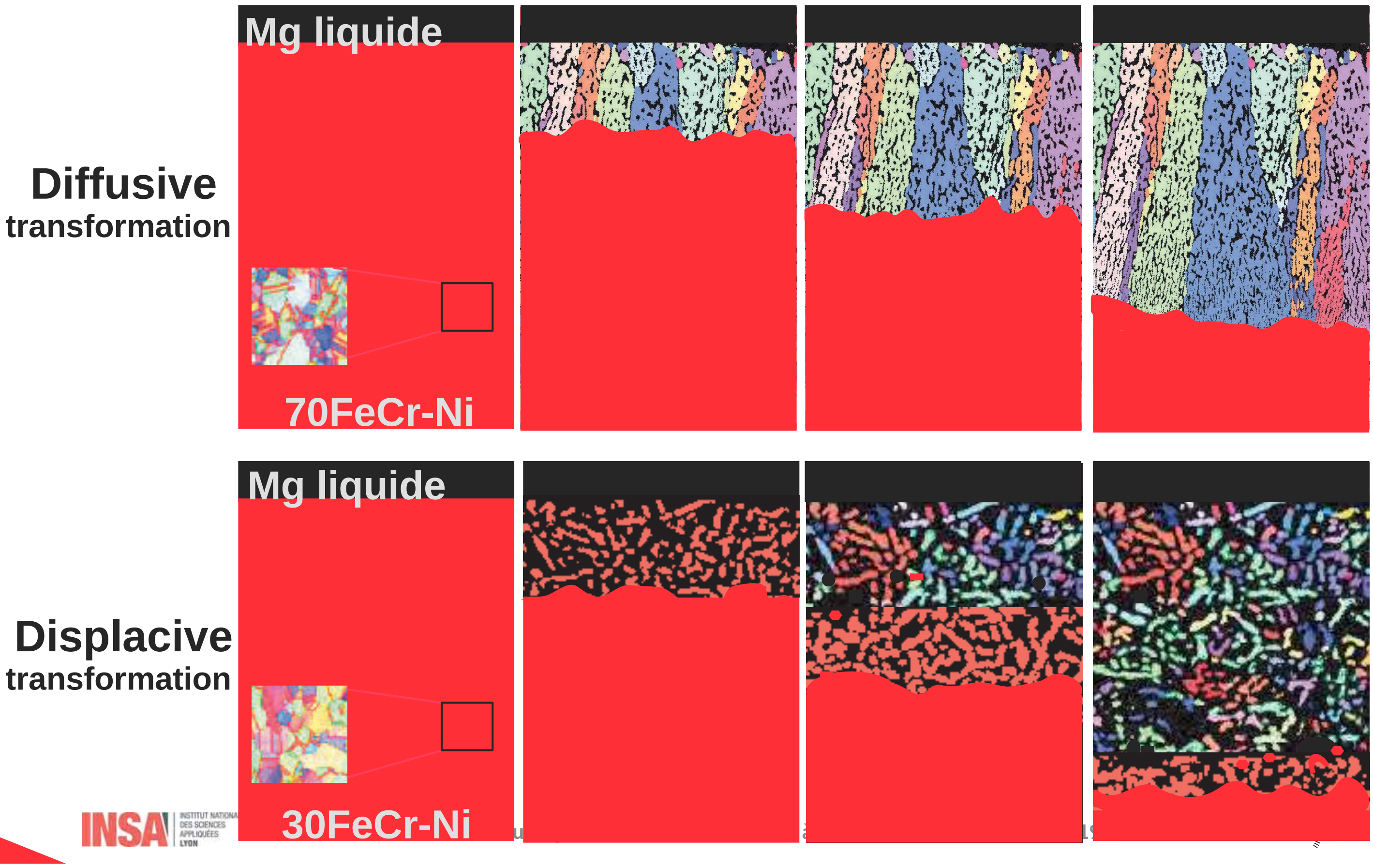
- Ligaments and pore size can be predicted

Partial dealloying for 3 min



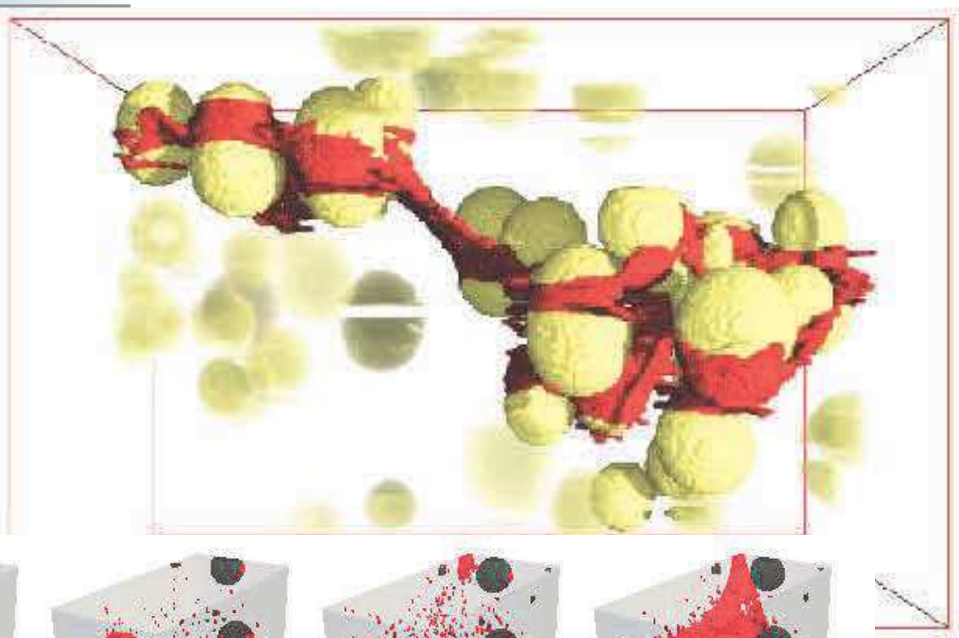


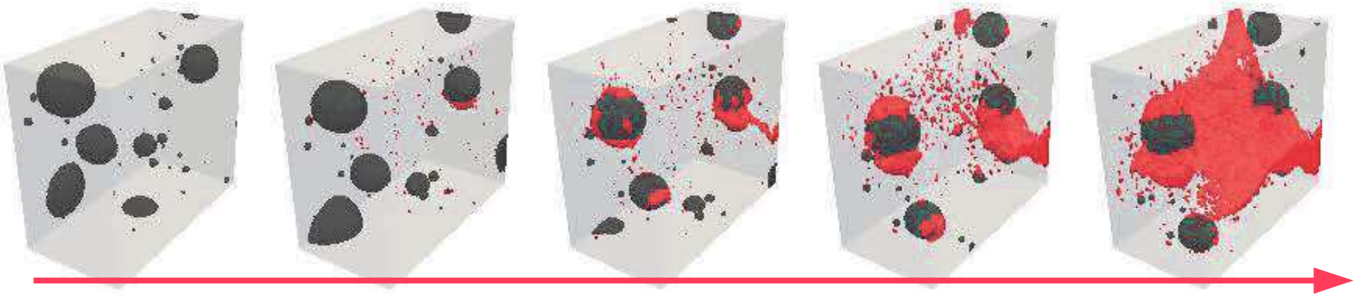
Grain formation mechanism



Xray tomography – examples: Ductile damage

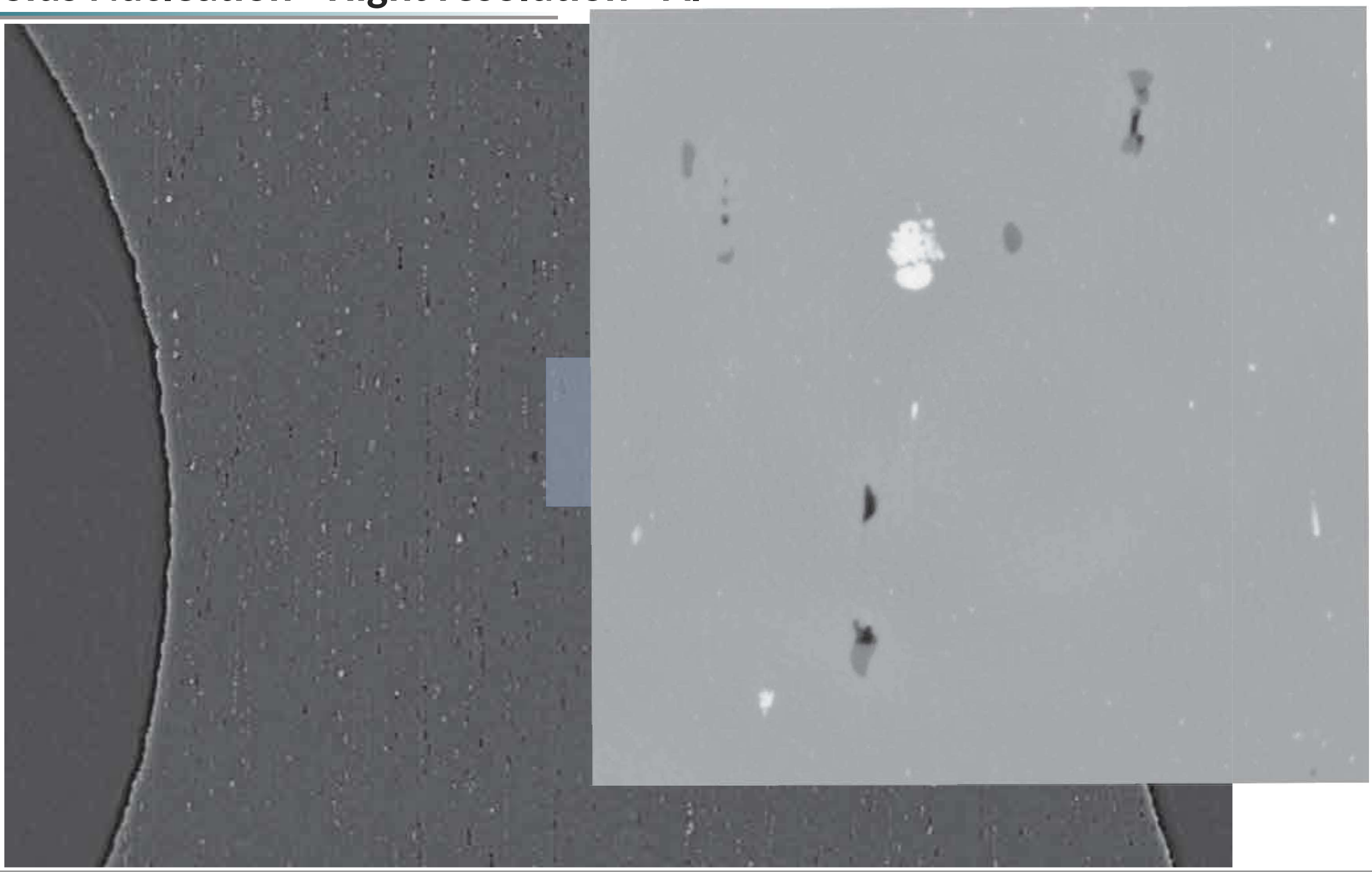
1) Germination
2) Growth
3) Coalescence/percolation
→ **brutal failure**





> Damage in Metals Studied by High-resolution in situ Tomography - ESRF, ID19

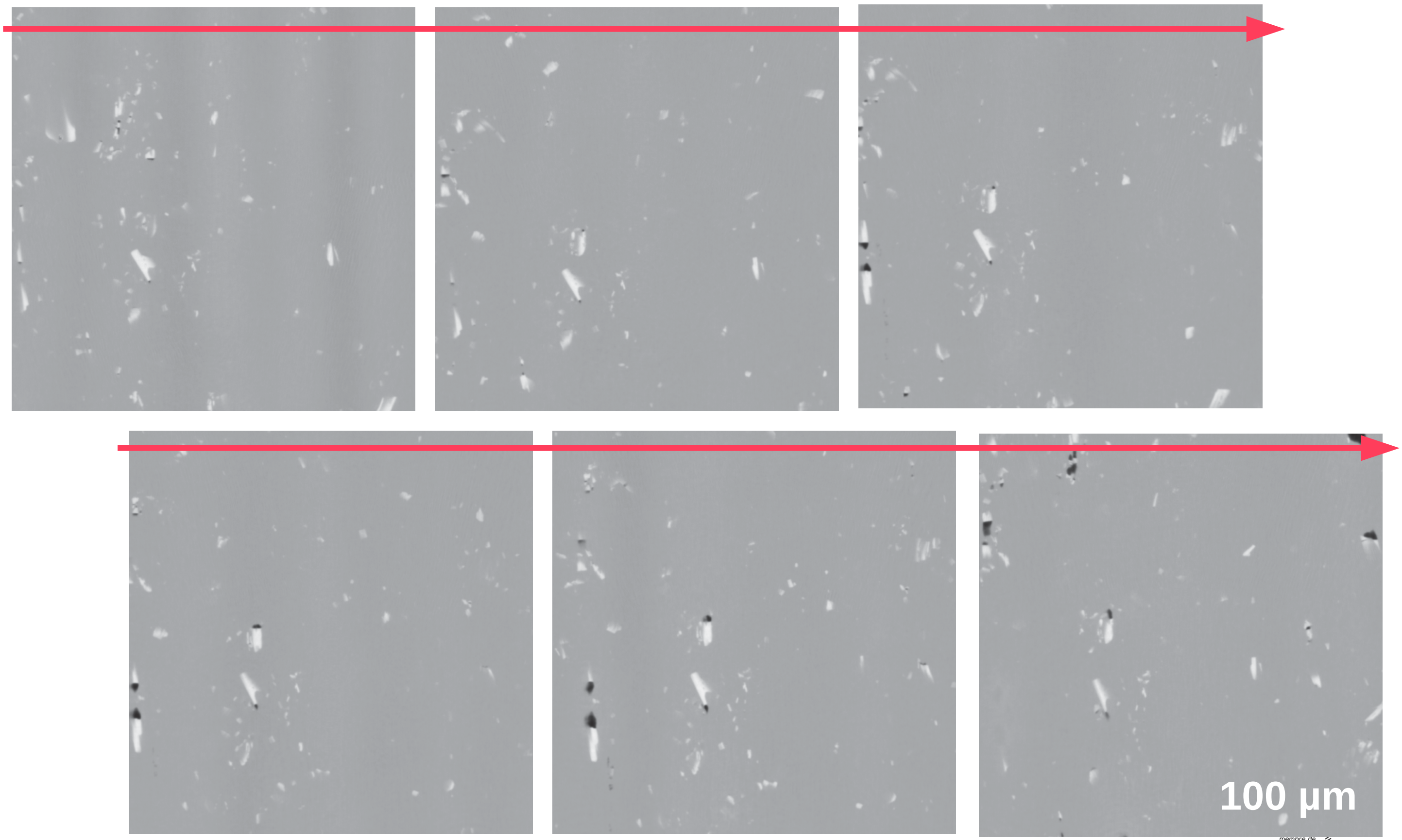
Voids Nucleation - Hight resolution - Al



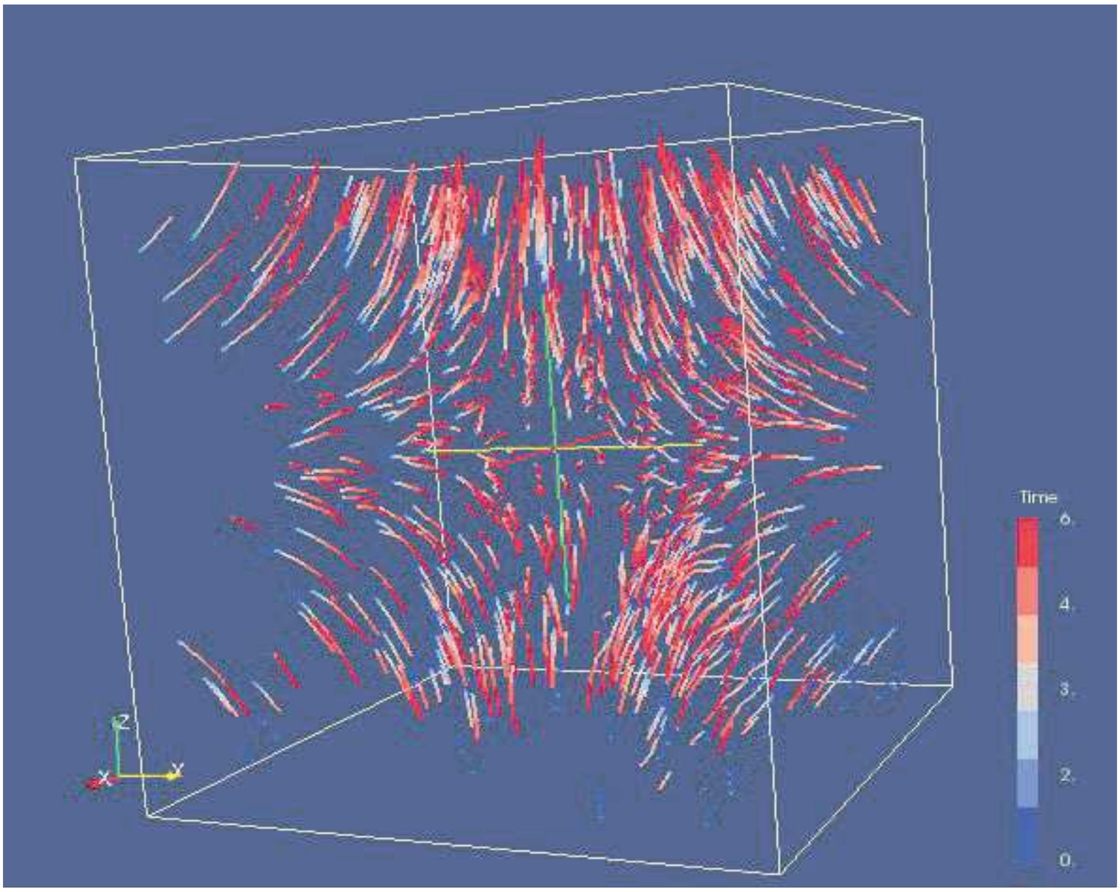
> ESRF, old ID22, now ID16B

Voids Nucleation - Hight resolution - Al

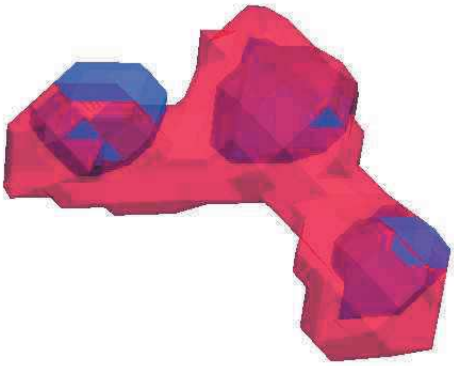
Nano tomo (aluminium 1200)

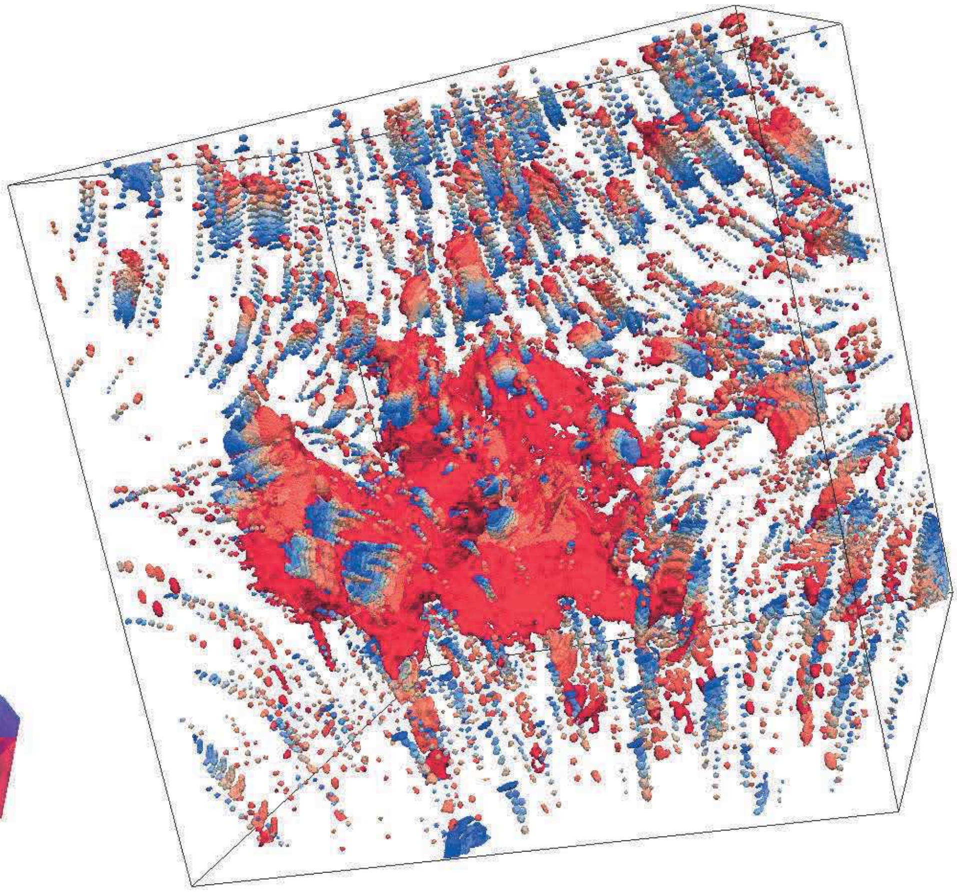


Growth – strain flow and shape evolution



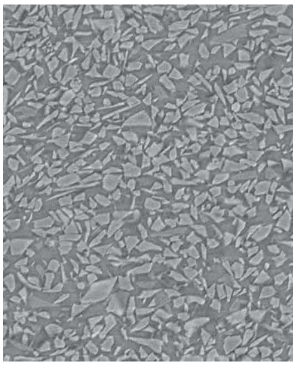
Growth – strain flow and shape evolution



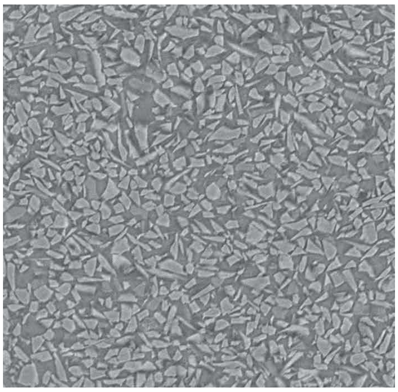


evolution of the acquisition time

> 5 minutes



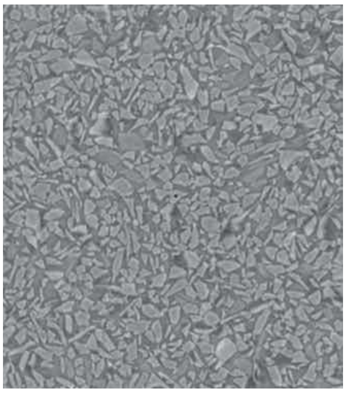
> 20 sec



tensile test

with dε/dt=10^{-3} s^{-1}

> 16 sec

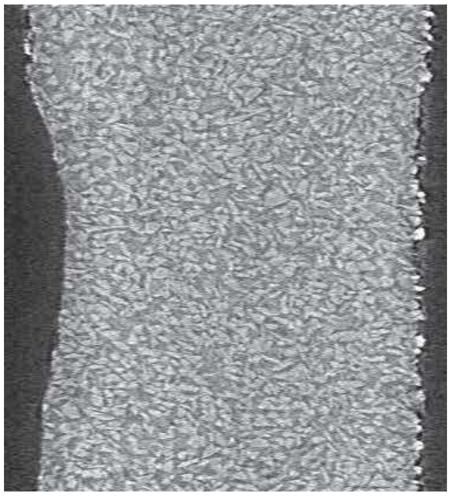


> ESRF ID15

tensile test

with dε/dt=10^{-3} s^{-1}

> 0,05 sec



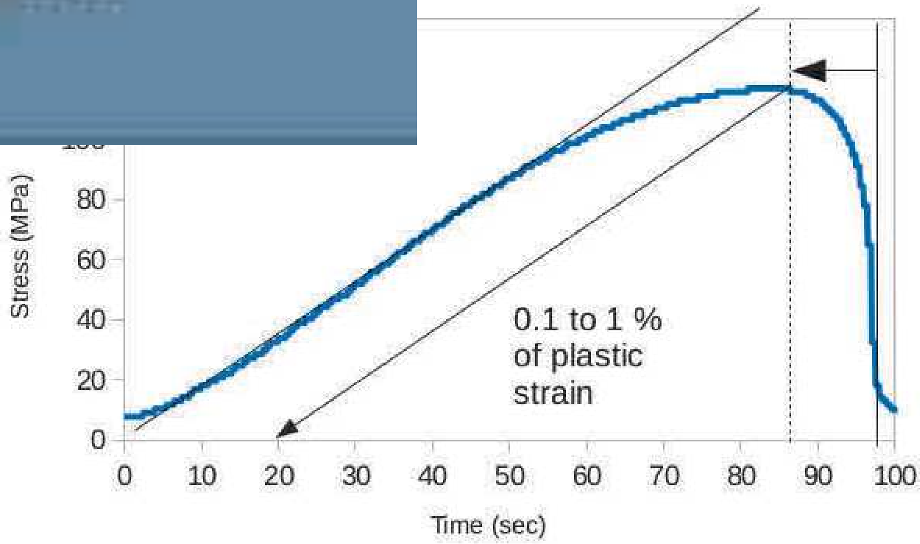
> SLS Tomcat

500 microns

Faillure – Fast tomo

- Rotation : 10Hz
- Acquisition : >10kHz
- 20 volumes/s
- 12s of acquisition (cyclic buffer)

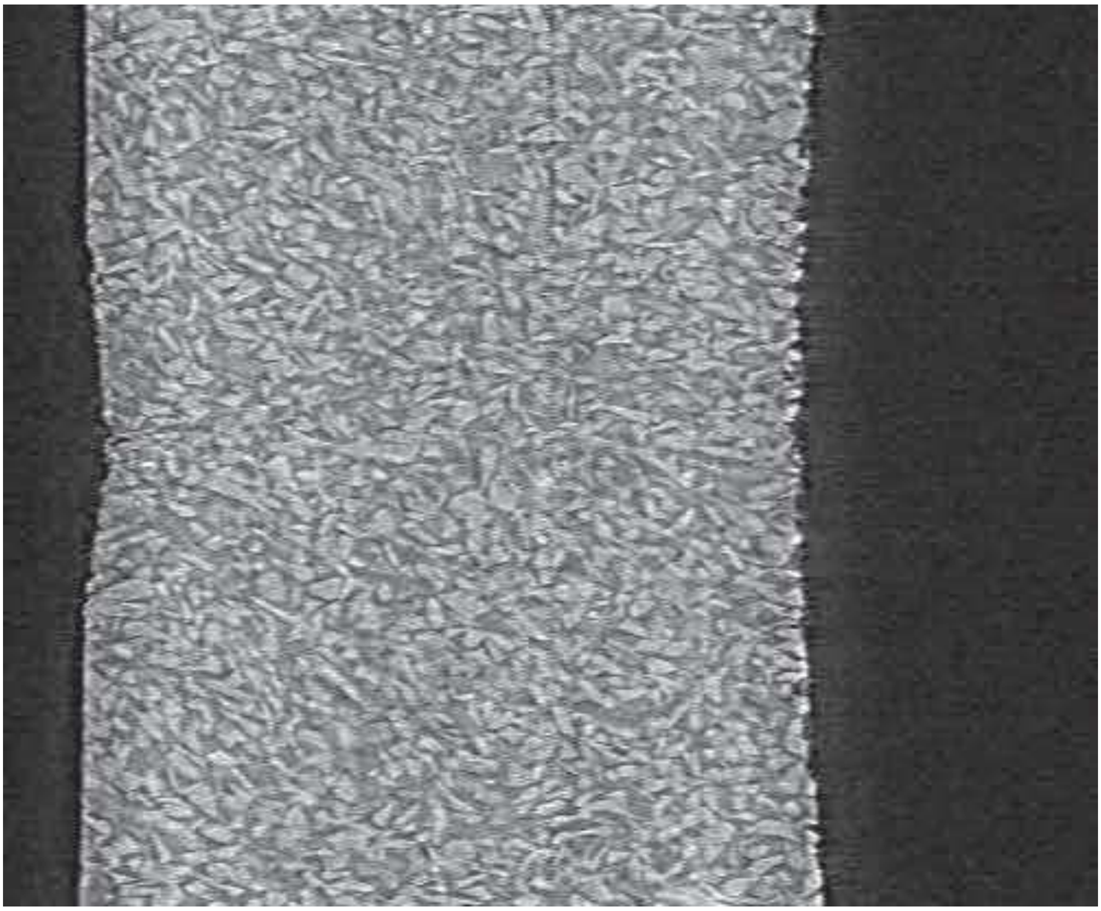
> SLS, TomCat
> traction in situ INSA-MATEIS



Failure – Fast tomo

> Acquisition 20 Hz

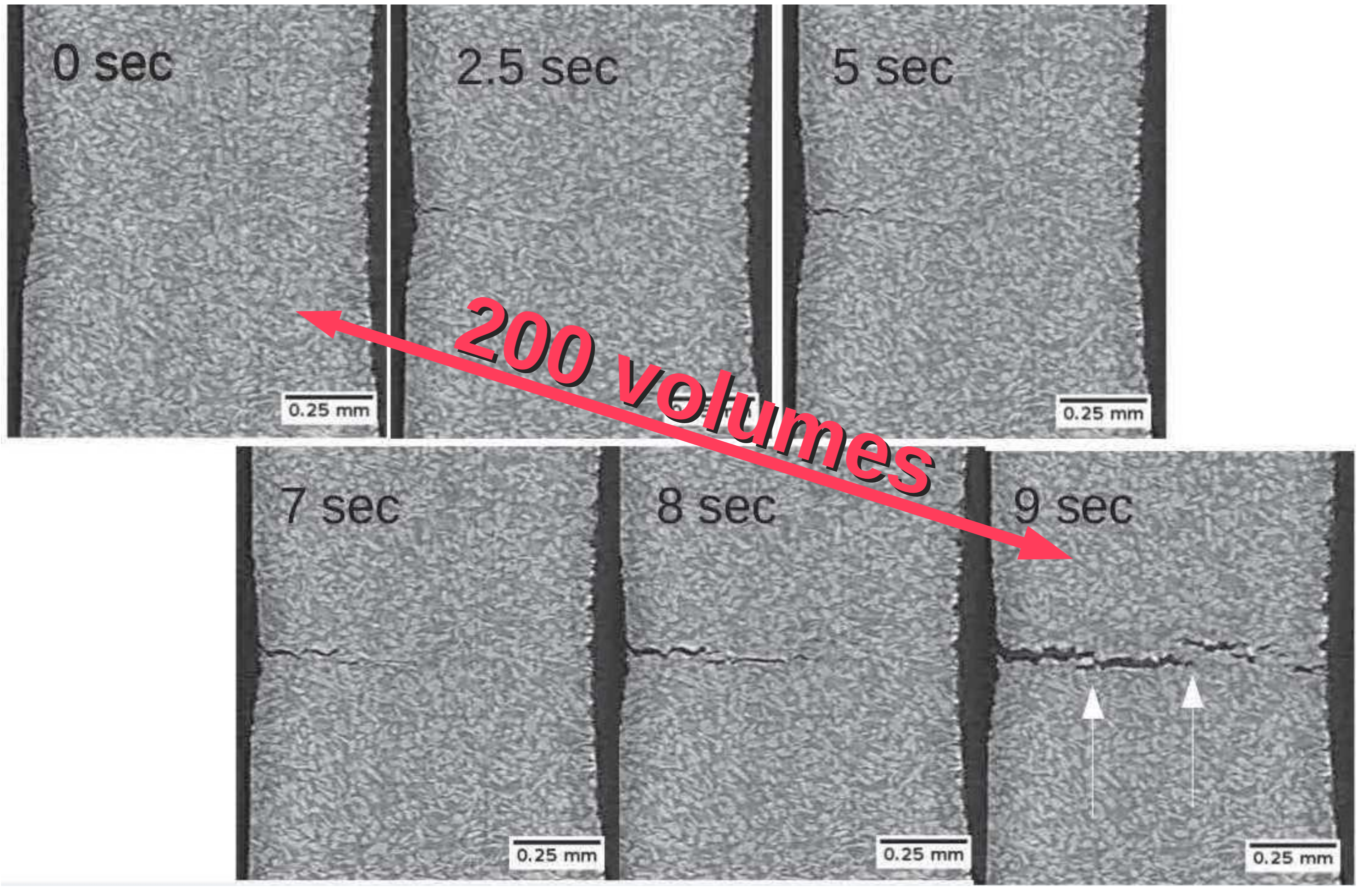
> real time 11s



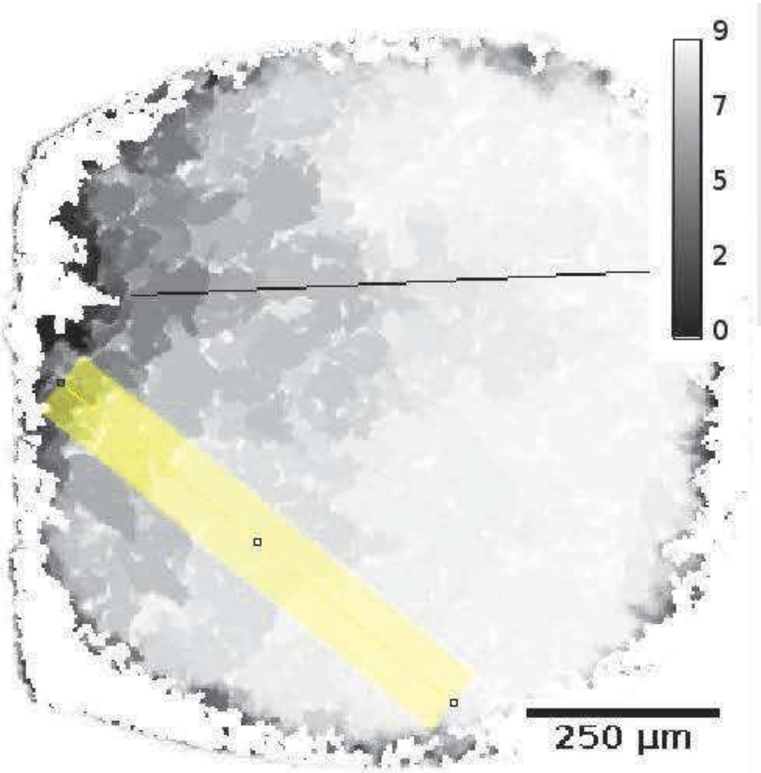
> with R Mokso

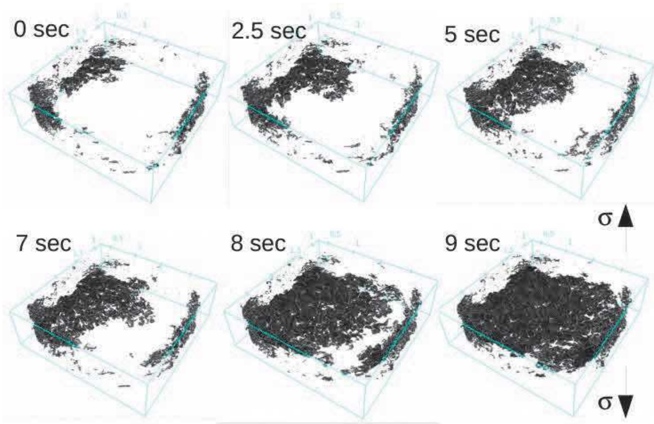
> SLS Tomcat

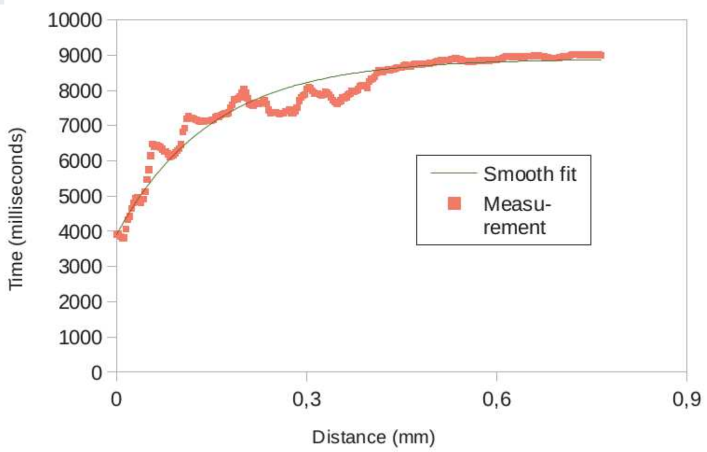
Faillure – Fast tomo



Faillure – Fast tomo



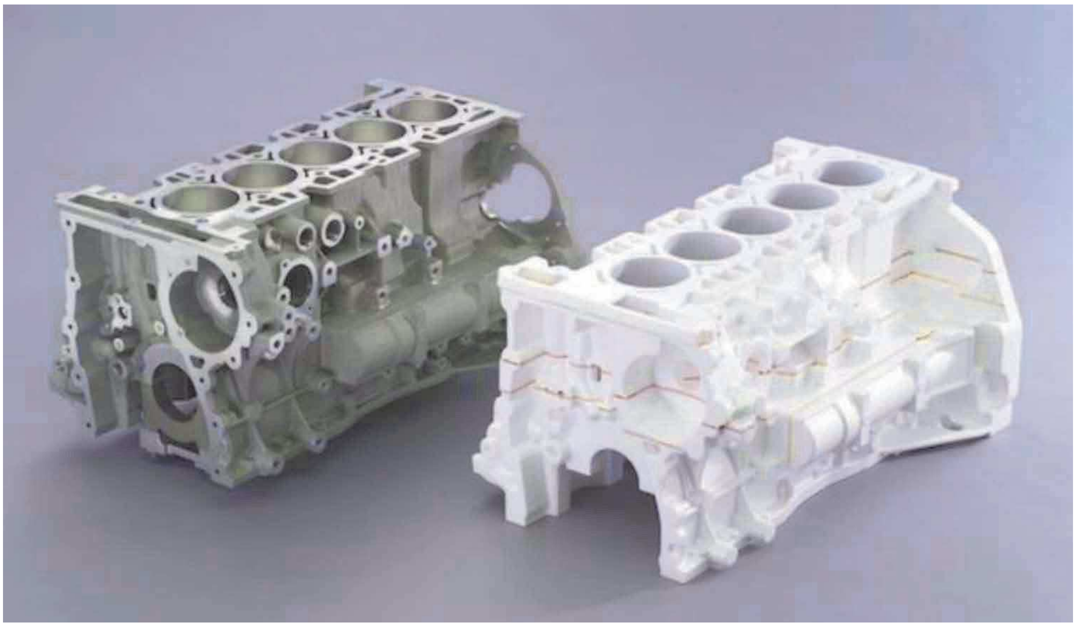




> 20 Hz X-ray tomography during an in situ tensile test, E. Maire · C. Le Bourlot · J. Adrien · A. Mortensen · R. Mokso, IJF (2016)

LCF at (moderatly) high temperature

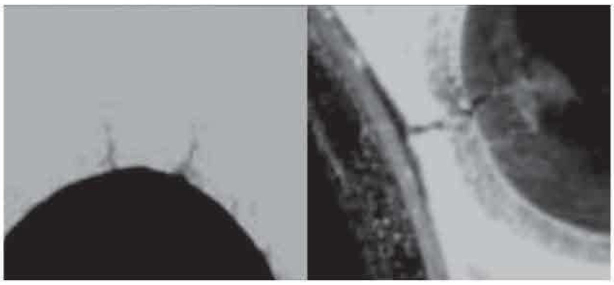
- Al Si alloys AlSi7Cu3 (A319)
- **Lost foam casting process**
- Geometry optimization and cost reduction



LCF at (moderatly) high temperature

Dissipated inelastic energy density
[Szmytka and Oudin, 2013]

- Cylinder heads are highly loaded during start/stop operations
- Inter-valve bridges are critical regarding thermomechanical fatigue



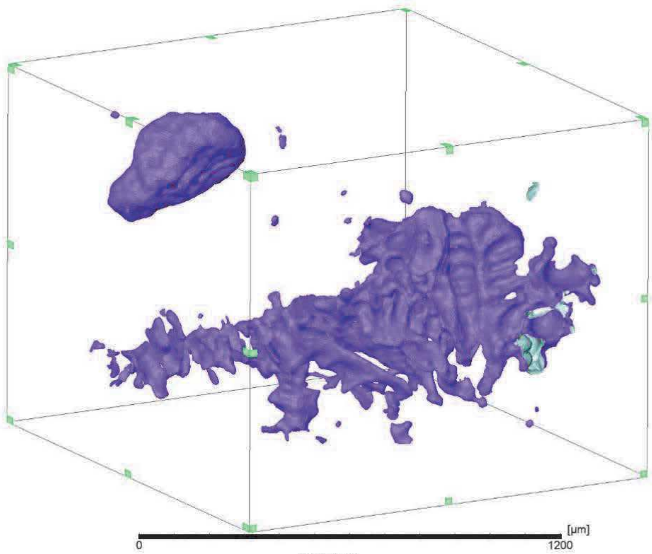
Cracked cylinder heads
[Thomas and *al.*, 2003]

Material microstructure

• **Pores**

Pores

- Vol. fraction ≈ 1%
- Max. Feret diam. ≤ 1.5mm

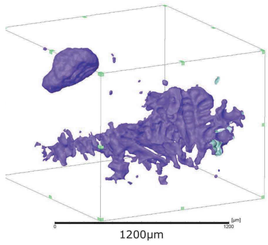


> Coll. N. Limodin E.C. Lille

Mateis

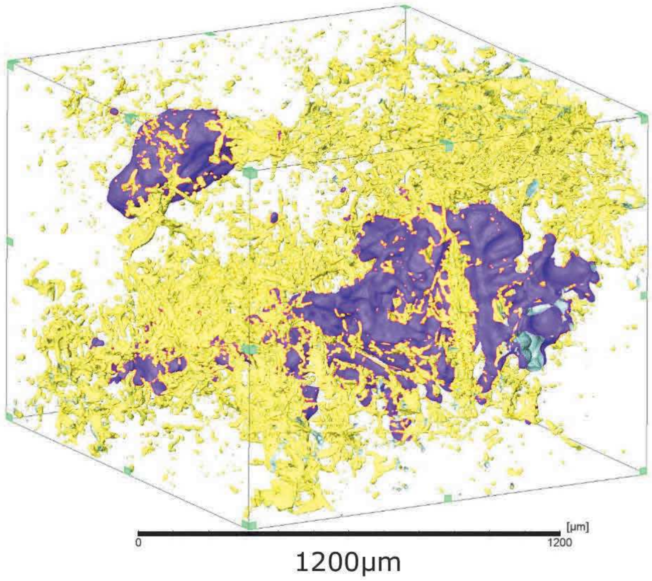
Material microstructure

- Pores
- Cupper phase - Al_2Cu



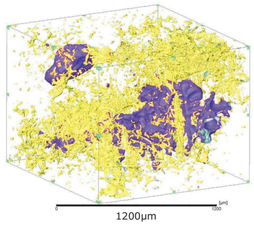
Copper containing phases Al_2Cu

- Vol. fraction ≈ 1.6%
- Max. Feret diam. >1.76mm
- Average thickness ≈ 12.2µm



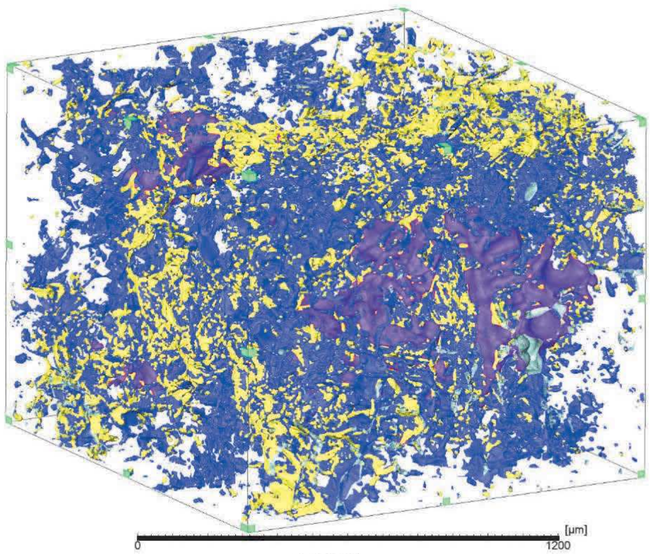
Material microstructure

- Pores
- Cupper phase - Al_2Cu
- Iron intermetallics



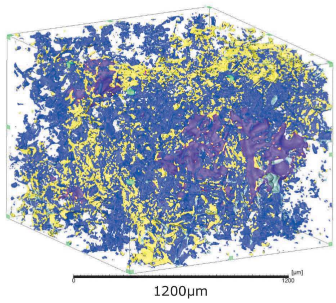
Iron intermetallics Alx(Fe,Mn)ySi

- Vol. fraction ≈ 3.2%
- Max. Feret diam. >2.7mm
- Average thickness ≈ 8.3µm



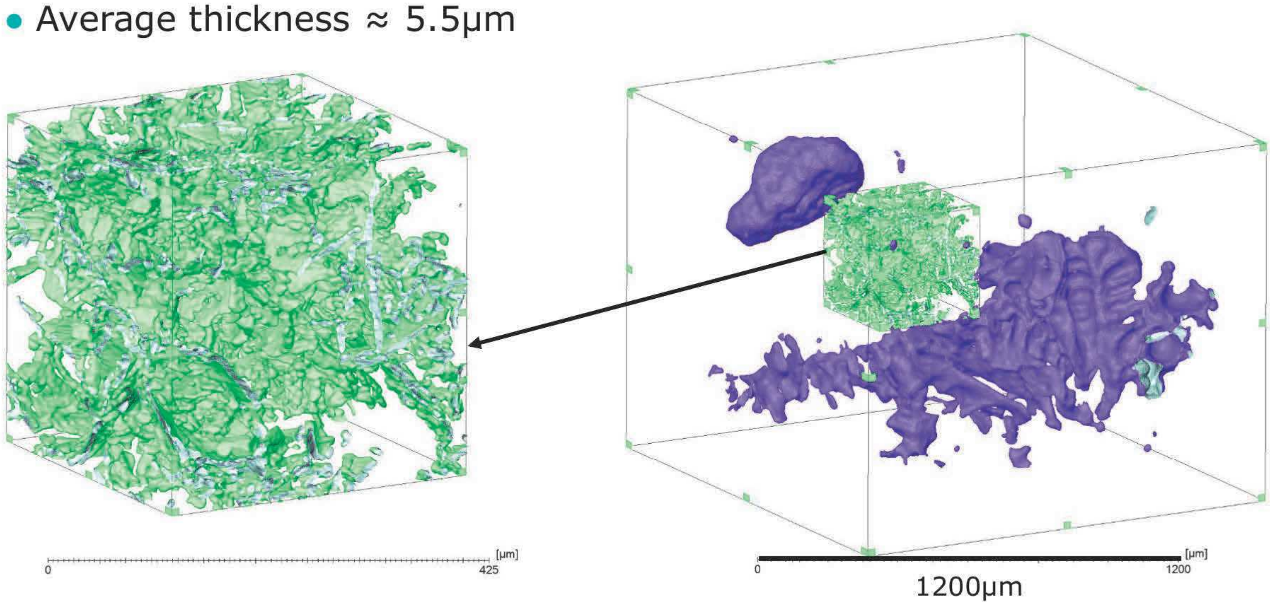
Material microstructure

- Pores
- Cupper phase - Al_2Cu
- Iron intermetallics
- Eutectic Si



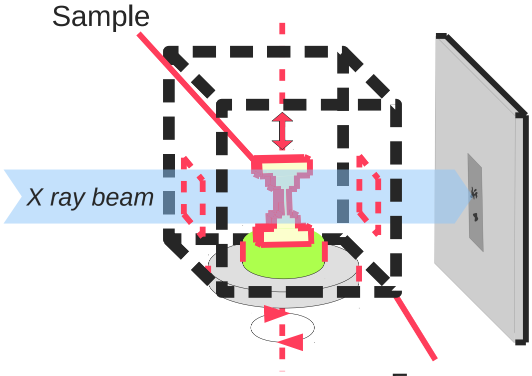
Eutectic Si

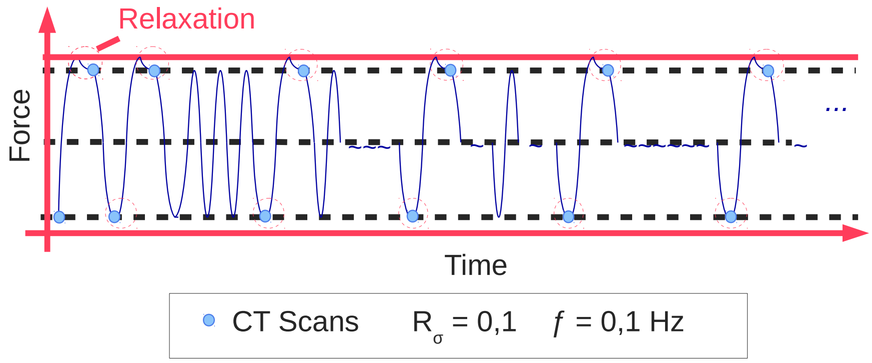
- Vol. fraction > 5.4%
- Max. Feret diam. >3mm
- Average thickness ≈ 5.5µm



- Three dimensional microstructure!

Experimental



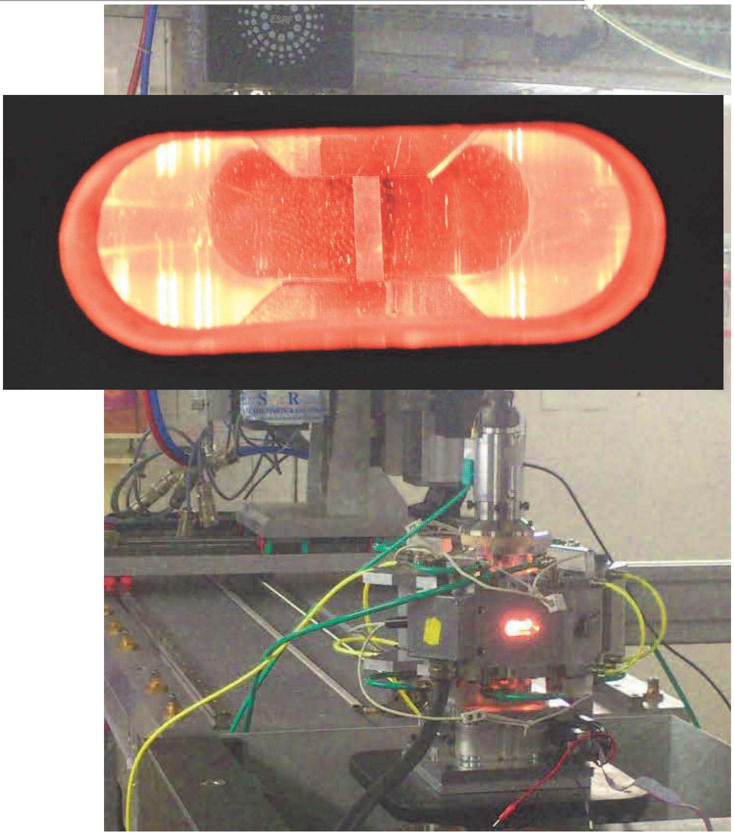


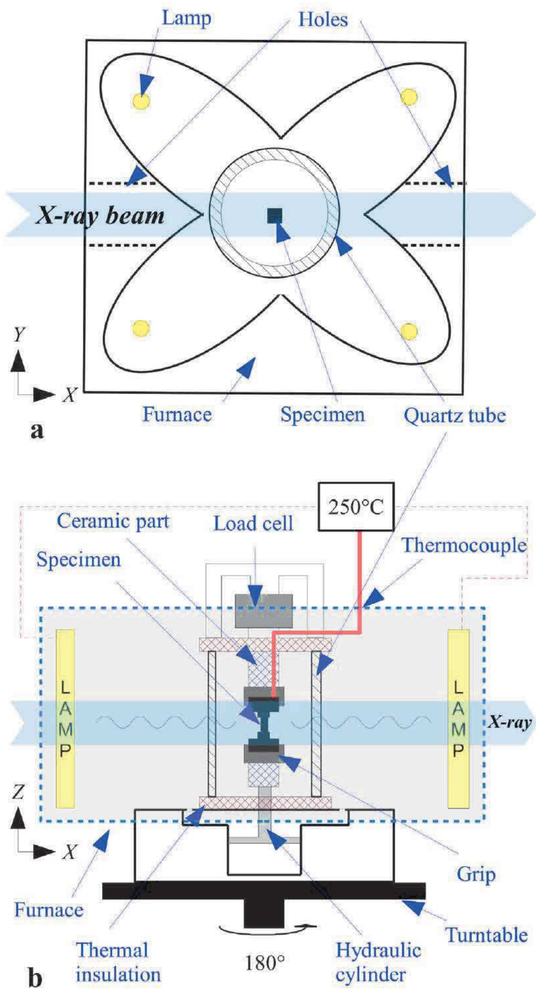
- ESRF ID19
- 35 KeV
- Voxel sizel = 2,75 µm
- Scan duration = 45 s.
- PCO camera

- Sp./Detector distance = 200 mm
- Temperature range up to 250°C

> S. Dezecot et al. Scripta Mater. 2016

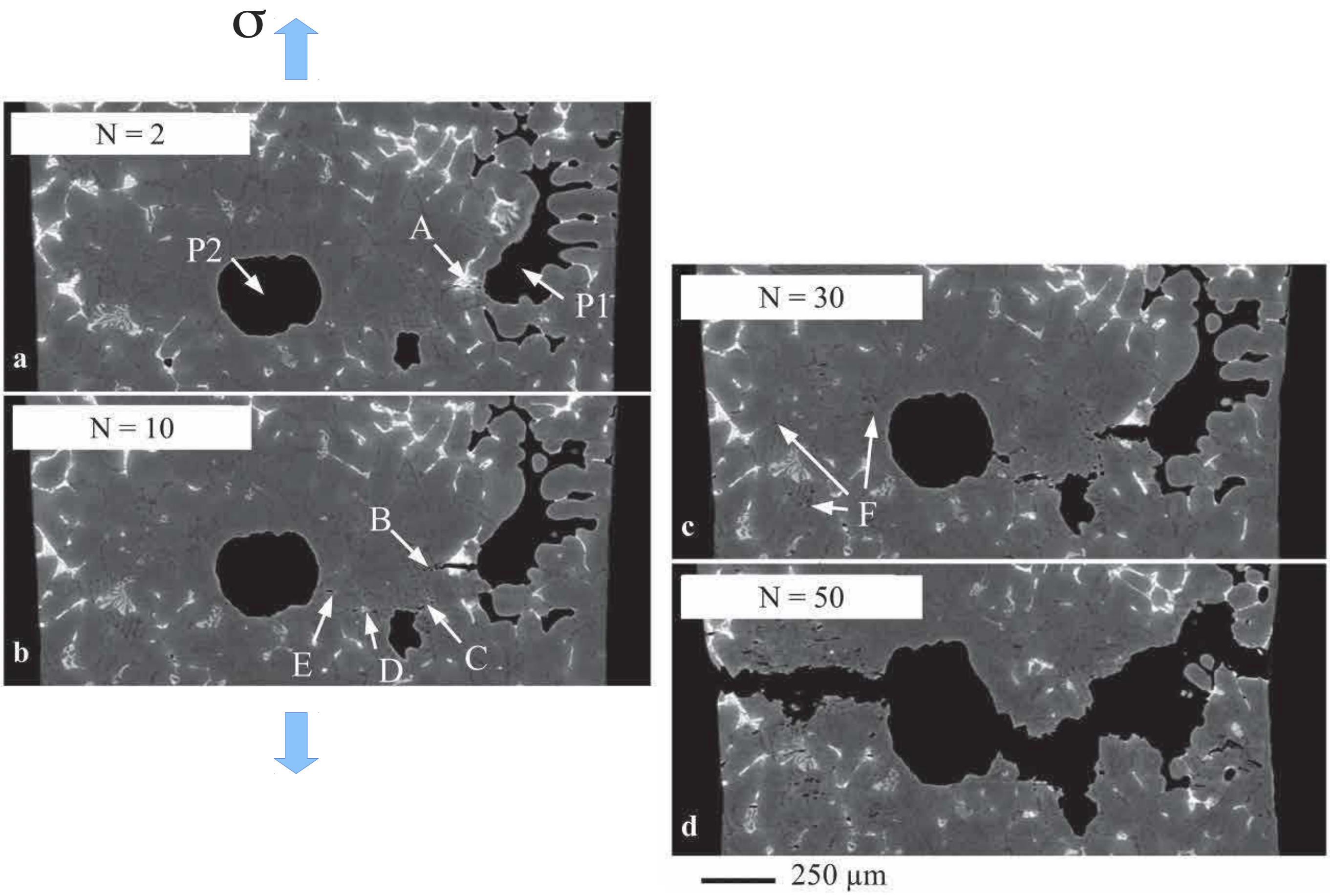
Experimental



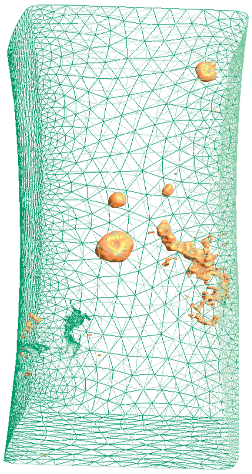


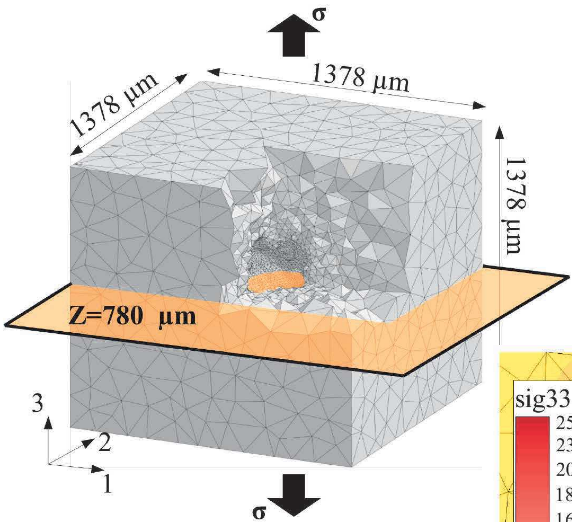
> Coll. A. Koster (ENMP)

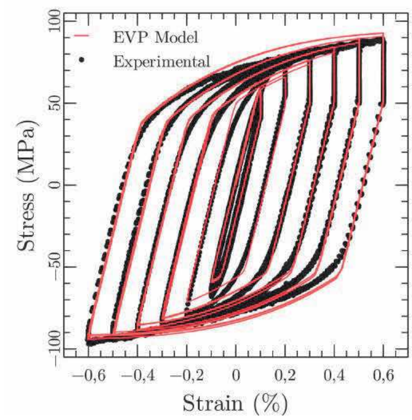
Damage mechanisms



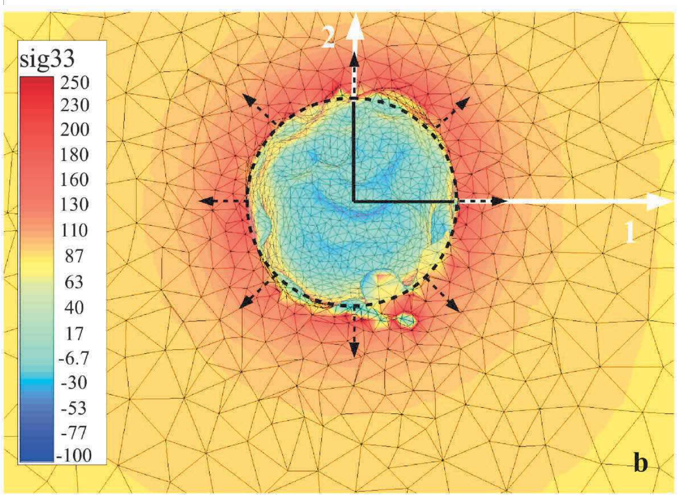
Modelling



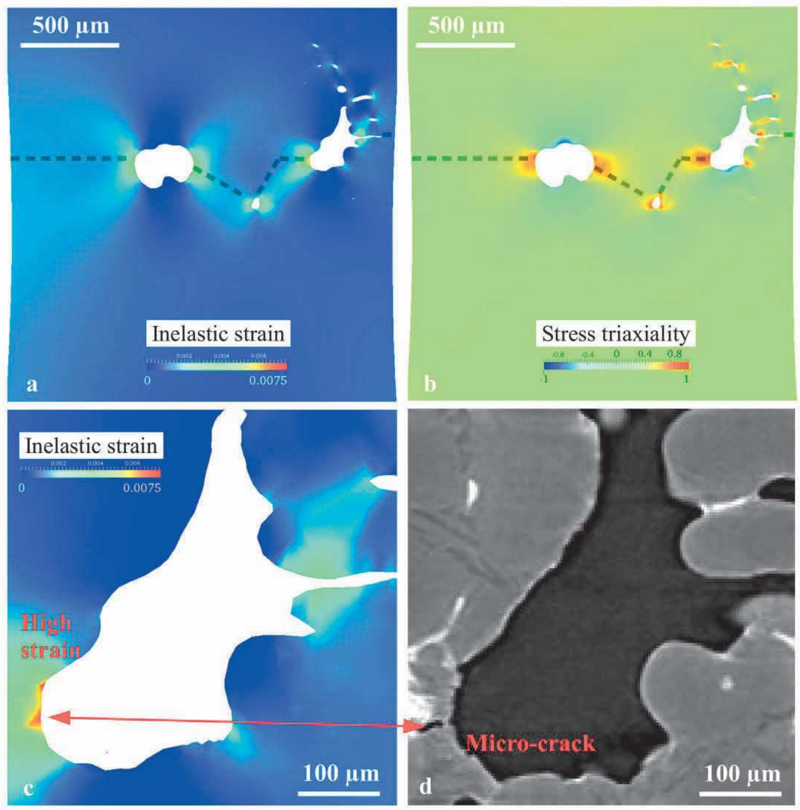




- Temperature : 250°C
- Chaboche law
- Linear elasticity
- Non linear kinematic hardening
- Norton flow



Modelling

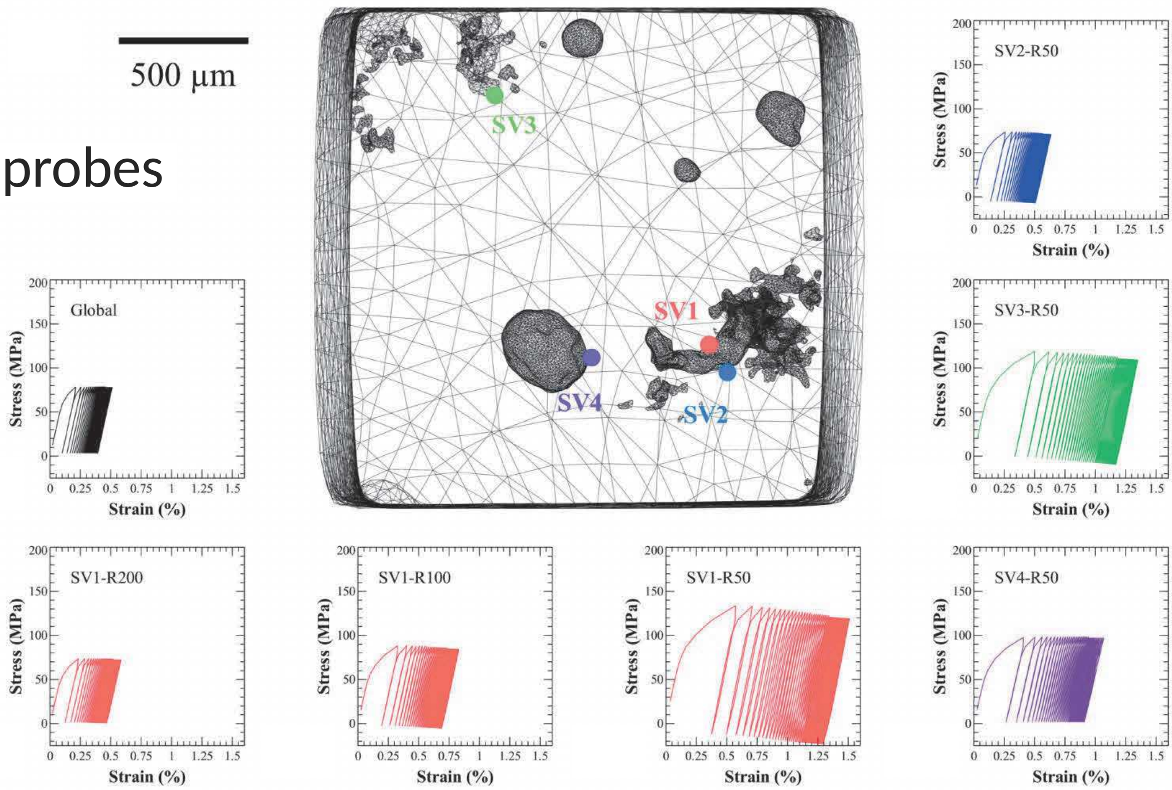


Inelastic strain and stress triaxiality

S.Dezecot et al. Acta Mater. 2017

Modelling

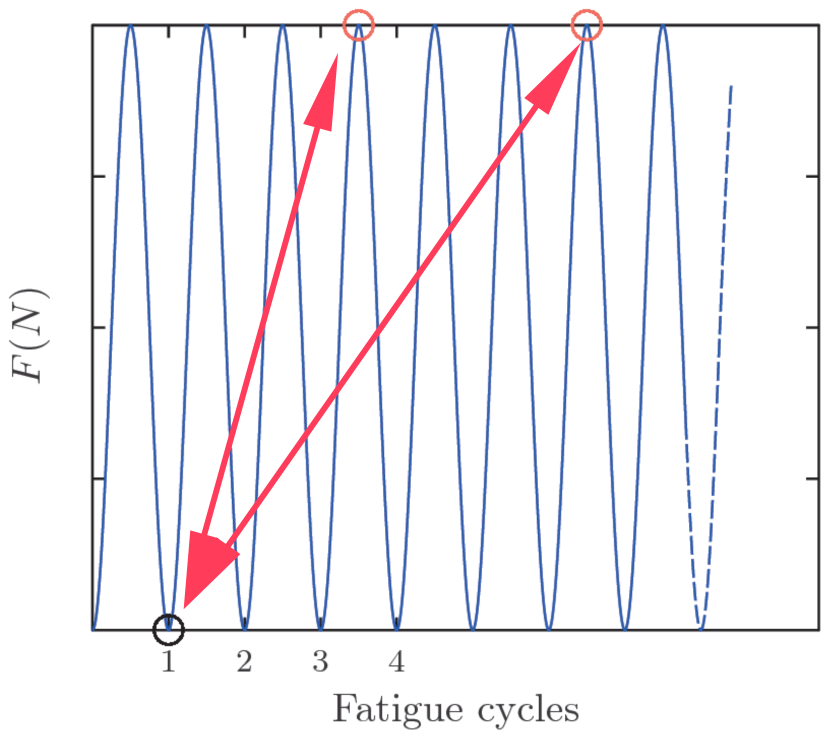
Local probes



- Cracks initiate at large strain heterogeneities → **experimental values**?

Digital Volume Correlation

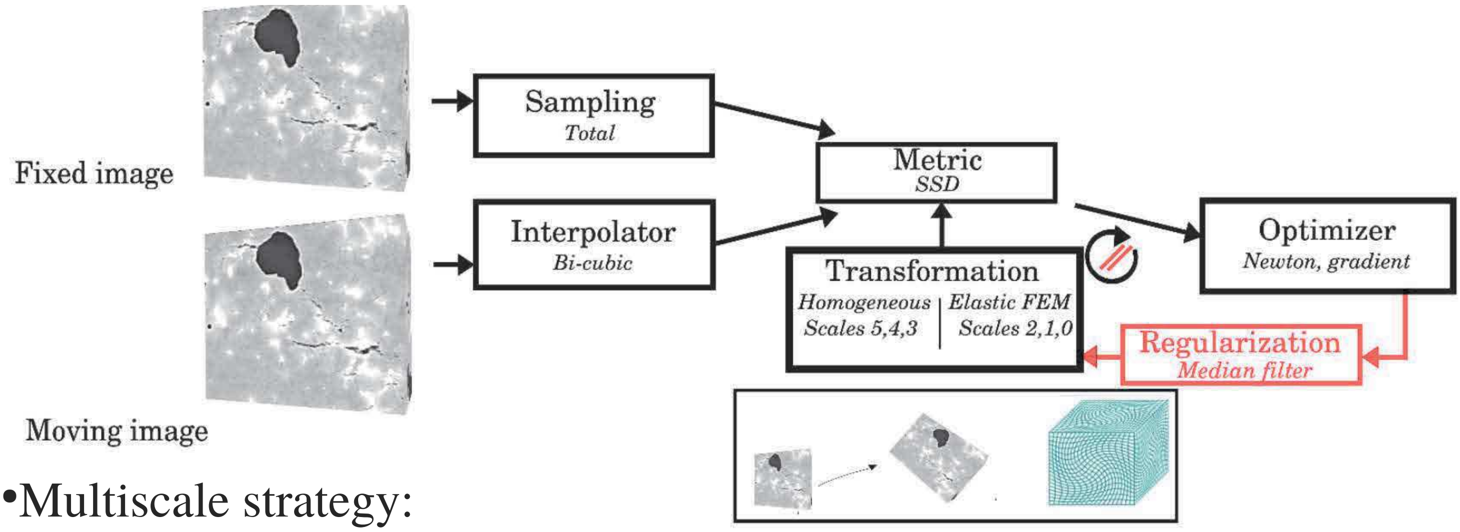
- DVC between min of the 1st cycle and max. of a given cycle → Cumulated strain.



Digital Volume Correlation

Coll. J.F. Witz E.C. Lille

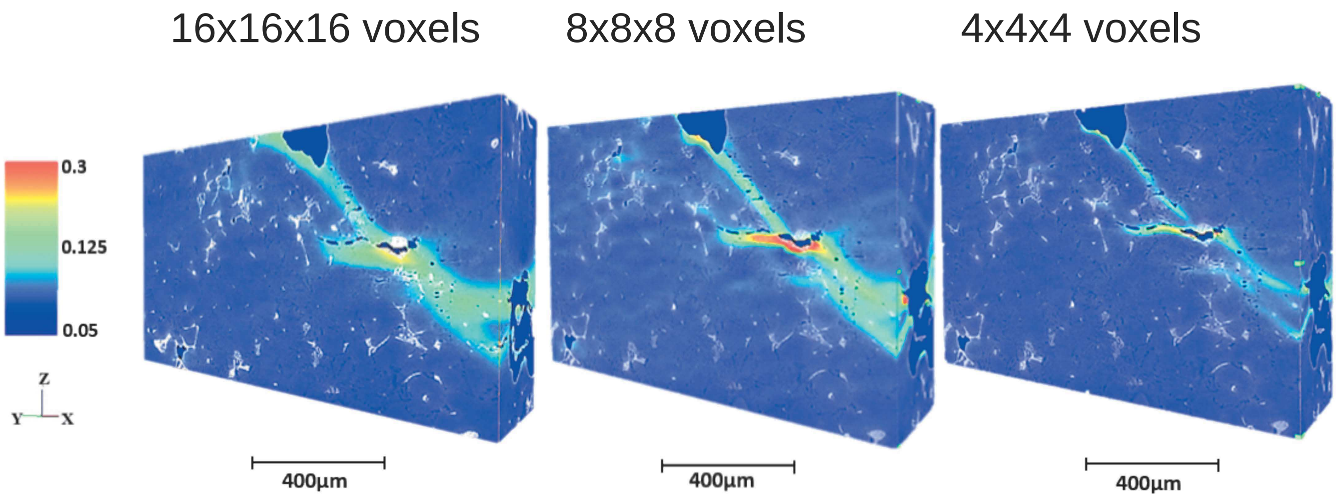
- YADICS software



- Multiscale strategy:
  - Scale “n”: a macrovoxel = average of $2^n x 2^n x 2^n$ voxels
  - Scales 4 to 3: homogeneous transformations
  - Scales 2 to 0: local transformation
  - Regularization: median filter

YADICS (http://yadics.univ-lille1.fr/wordpress/) Dahdah et al. Strain (2016)

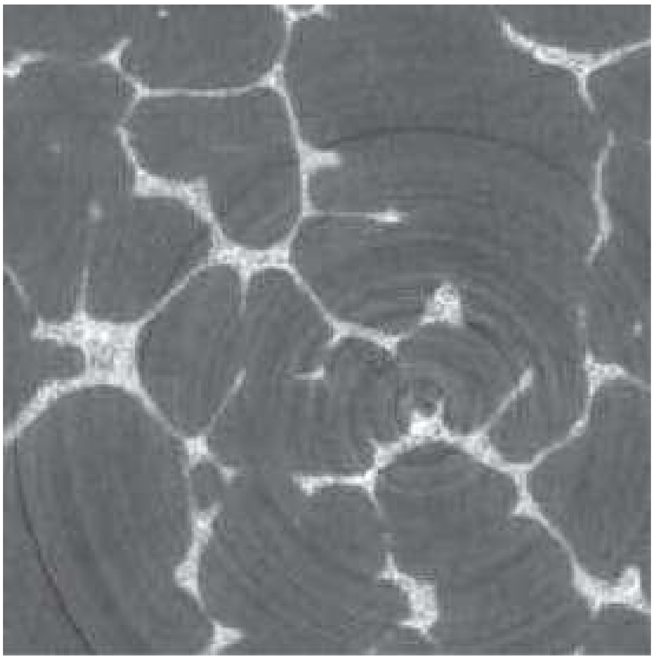
Digital Volume Correlation



- Image size ~ 1560x1450x2160 voxels (2.5x2.4x3.5 mm^3)
- ROI: ~ 1400x1300x800 voxels (2.3x2.1x3 mm^3)
- Small element size required → 4x4x4 voxels

PhD Nora Dahdah Coll. N. Limodin, A. El Bartali, J.F. Witz, E. CharkalukJ.F. Witz E.C. Lille

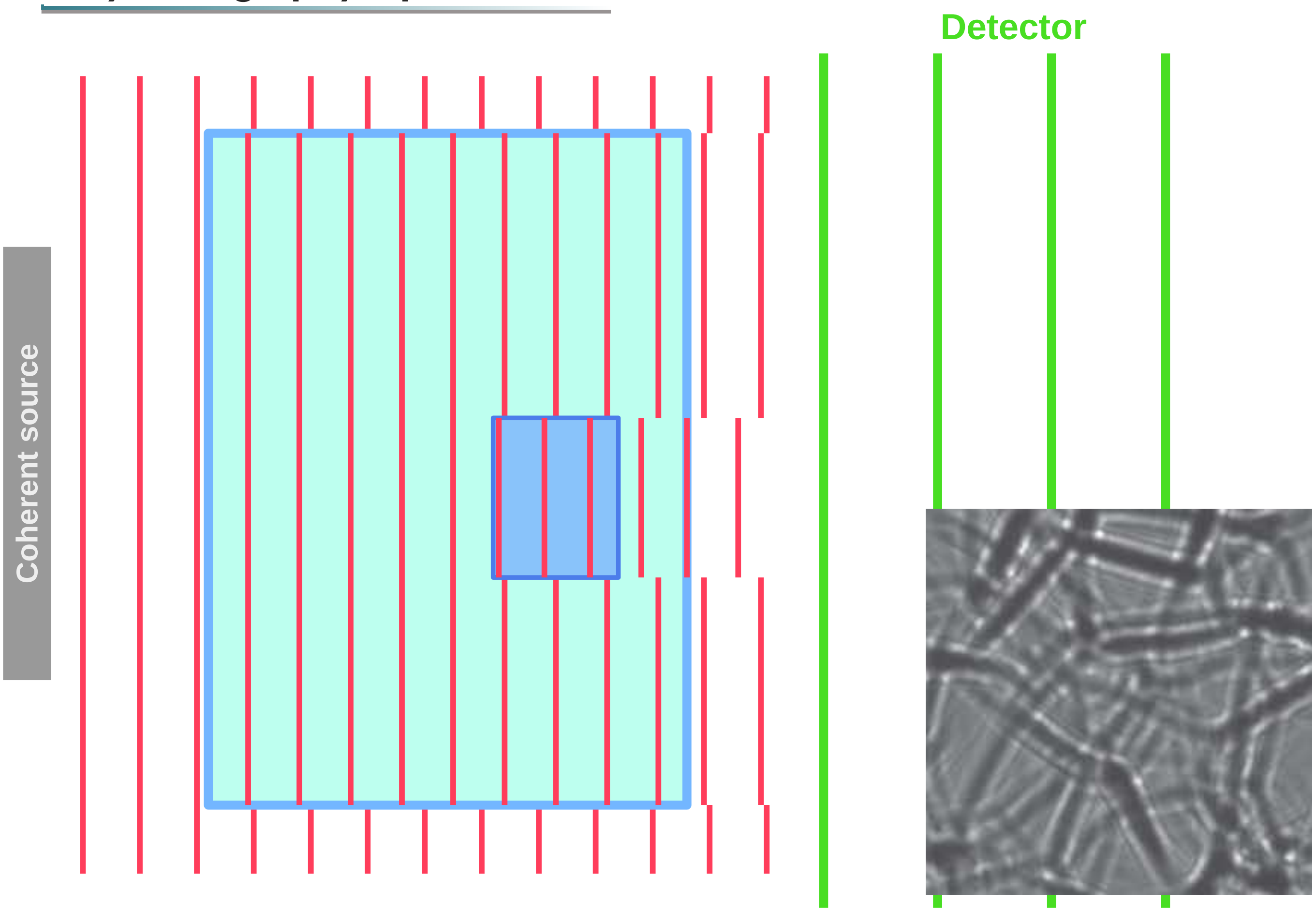
Xray tomography - limitations



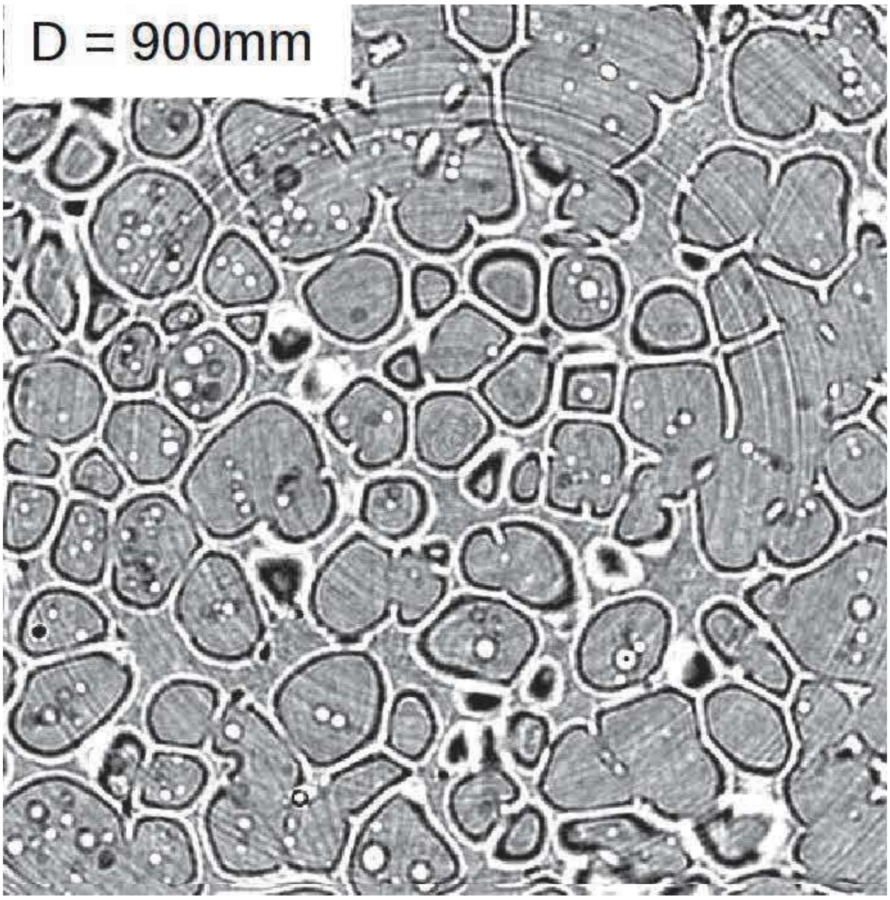
Al-Cu

Al-Si

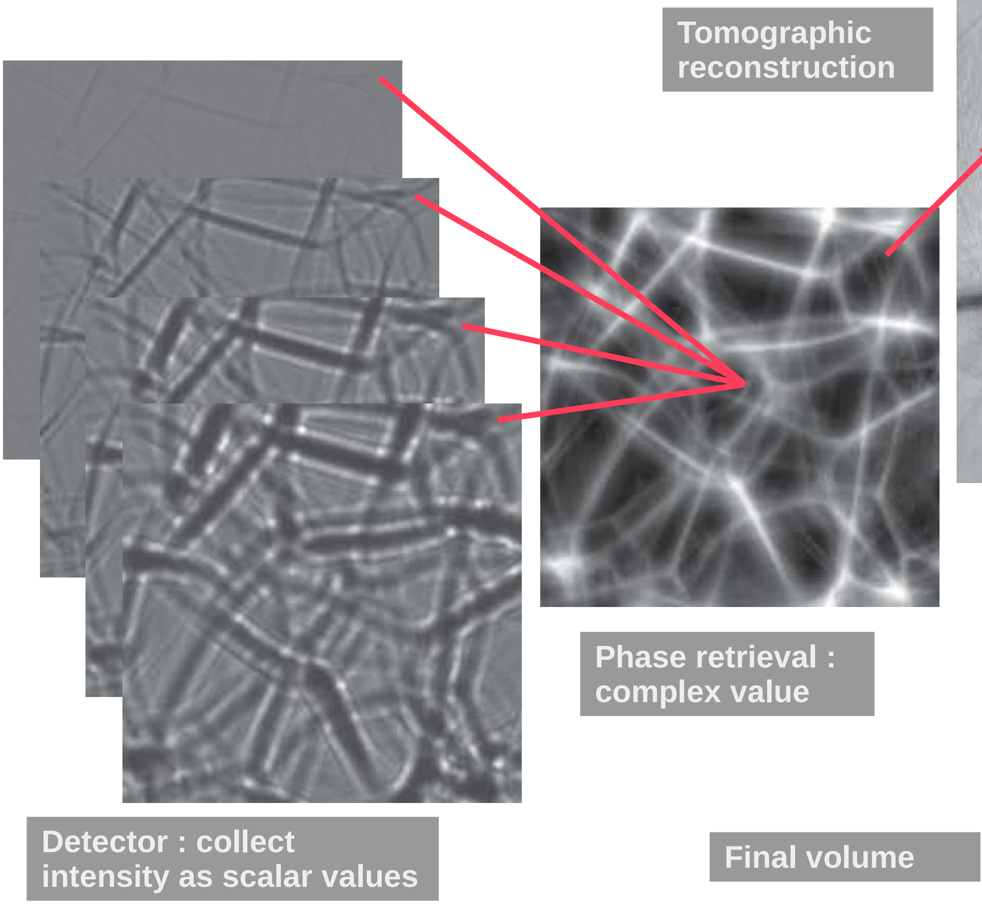
Xray tomography – phase contrast

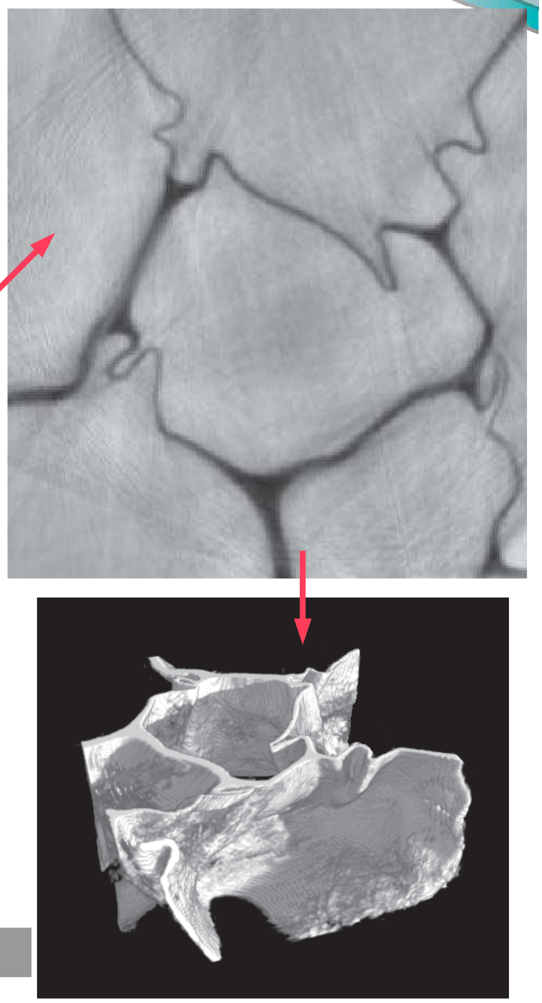


Xray tomography – phase contrast



Xray tomography – holotomography



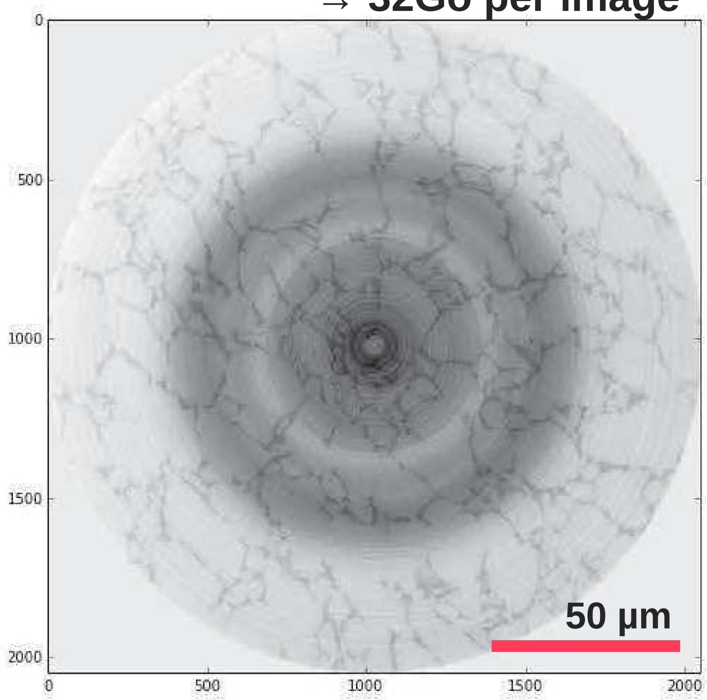


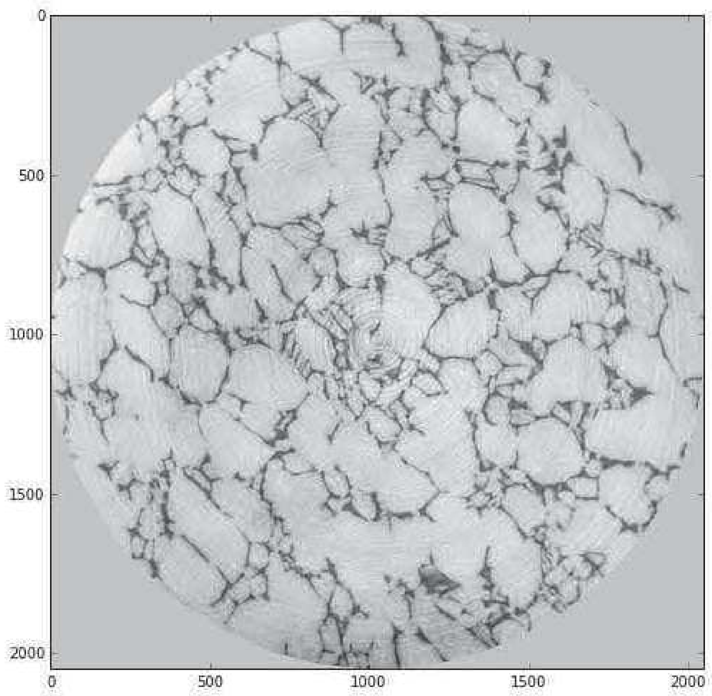
1a : voids nucleation – high resolution - Ti

ESRF, ID16B :
- 1 voxel ~100x100x100 nm^3
- 2048*2048*2048 voxels
- Dynamic of 32bit

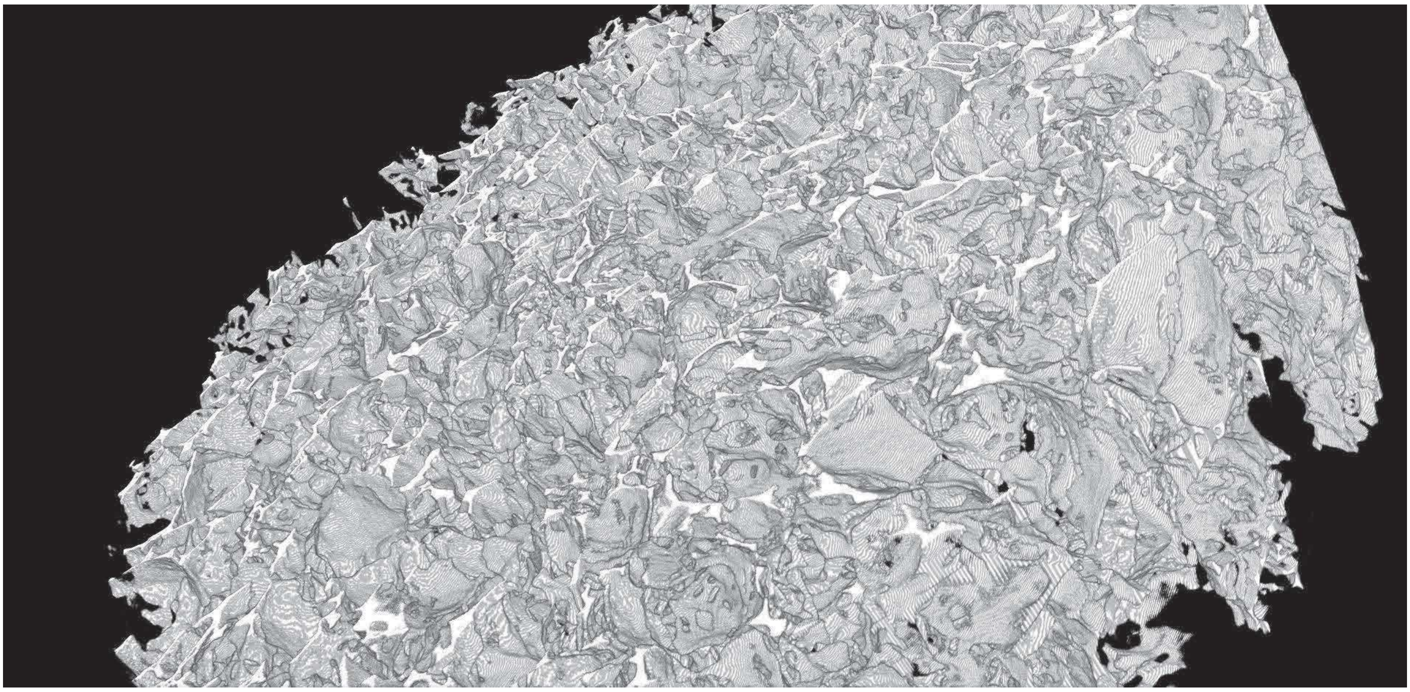
→ **32Go per image**

Dedicated tools ! *(python)*





1a : voids nucleation – high resolution - Ti



> ESRF, ID16B

Conclusion on tomography : full field scalar caracterisation

1) Need contrast for visualisation (Z, phases, voids...)
2) Resolution : from nano- to mili-
3) Volume of ~1000^3 voxels
4) In situ acquisition (meca, Th, ...)

Tracks:

5) Morphology
6) Evolutions