Point de vue et images de la métallurgie:

Novel characterization techniques in Metallurgy

E Maire

Mateis, INSA de Lyon

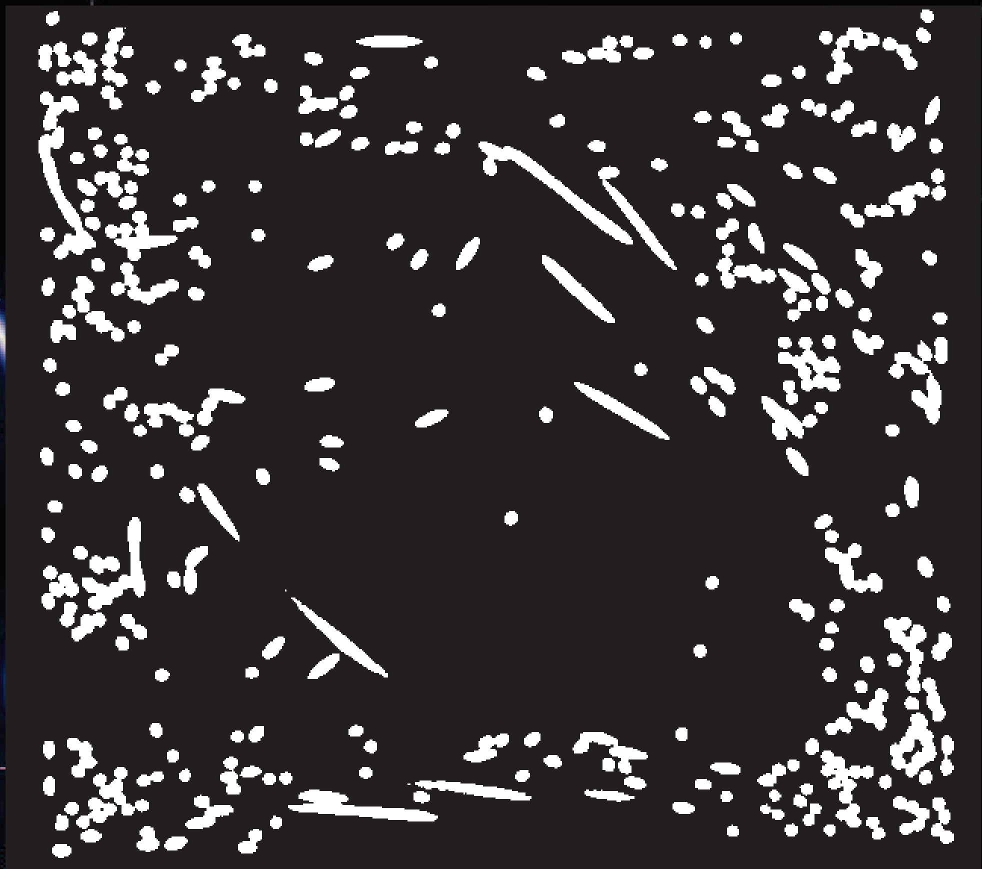
Introduction

- Characterization in metallurgy :
  - Mechanical
  - Microstructural
  - Physical, chemical...
- What can be regarded as “novel” in the world of characterization techniques ?
- Mechanical :
  - Indentation, local measurements, simulation
- Microstructural
  - Progress in microscopy, fields measurement
  - **3D imaging: novel, potential development**

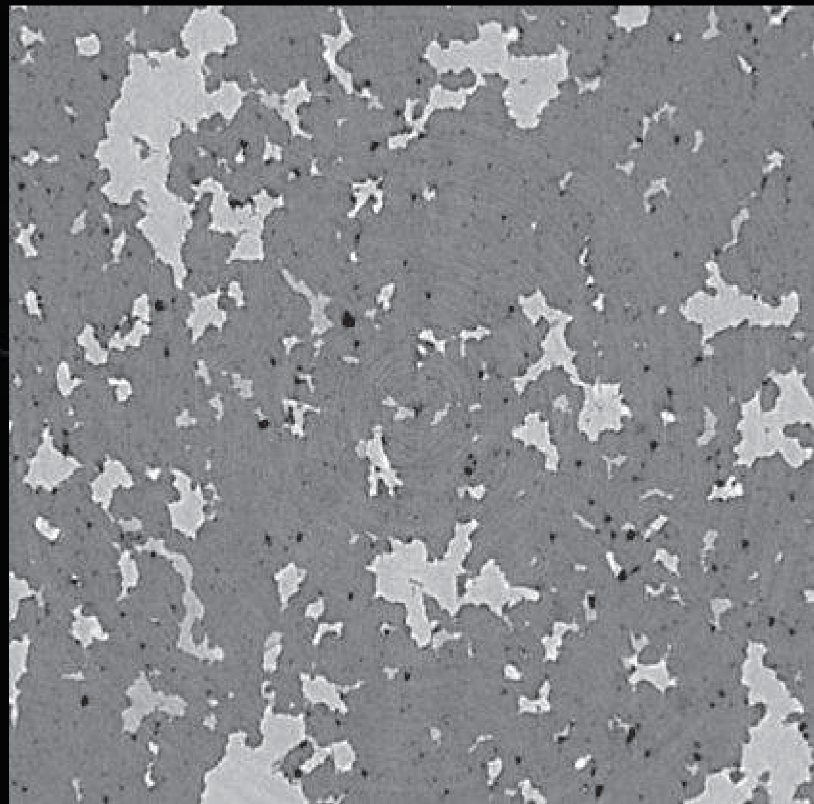
Why is 3D imaging useful for metallurgy:

- Microstructure can have a very complex nature
- Standard procedure = 2D + stereology
- Some cases : not enough
- 3D images to
  - Qualify (to make nice renderings)
  - Quantify
    - Fraction, Size, Shape
    - Connectivity
    - Percolation, coalescence
- Some are non destructive: **evolution during loading (in situ)**
  - A lot of new infos for mechanical, physical and chemical metallurgy

Little quizz to convince you further



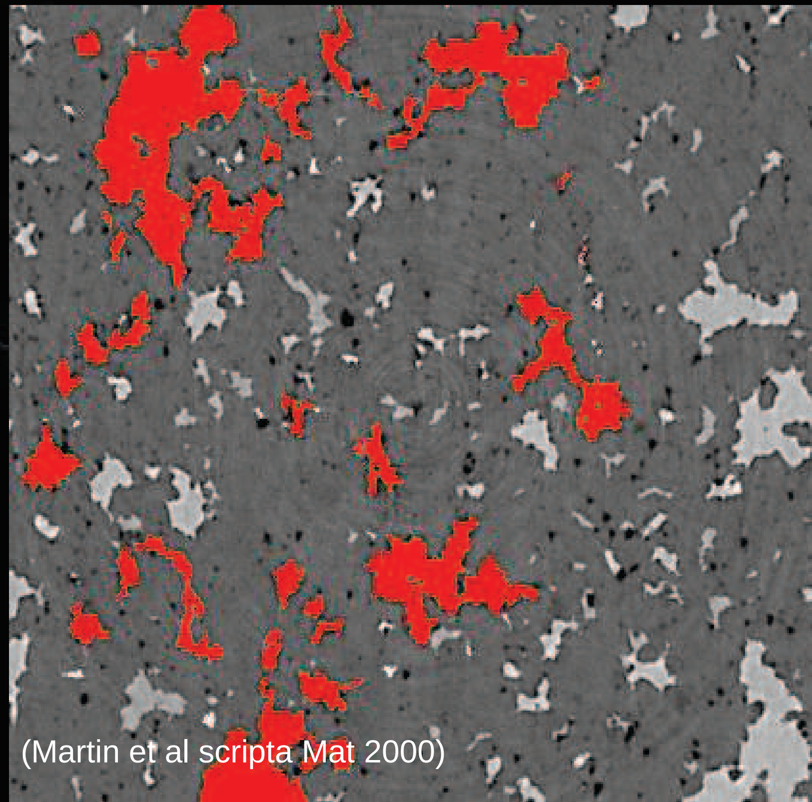
How many wires ?



How many pores ?

The answer can only be given by the 3D image :

Only one wire



Fully connected porosity

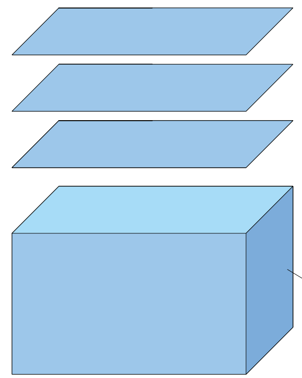
Outline

1) A general panorama of 3D imaging methods
   1) Destructive (serial sectioning, FIB, APT)
   2) Non destructive (electron, Neutron, X ray Computed tomography)
2) X Ray CT
   1) In situ experiments
   2) XRCT in metallurgy, Modeling based on the microstructure
   3) Digital Volume Correlation
   4) Future of XRCT

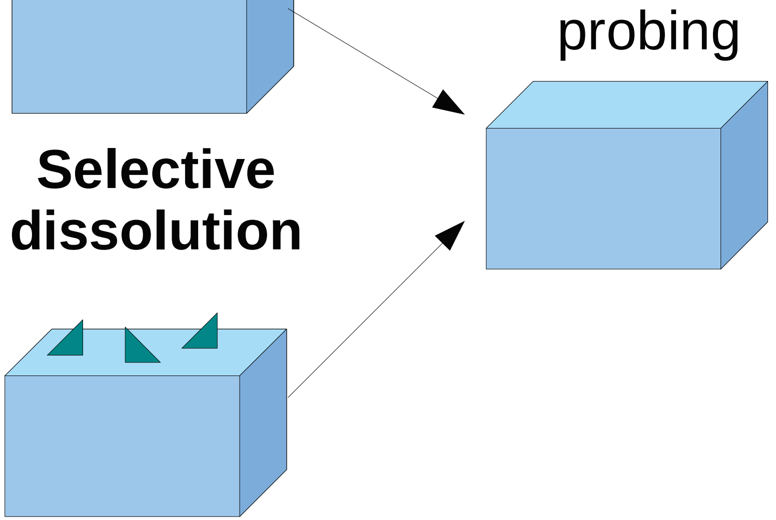
3D imaging methods

Destructive methods

Successive abrasion of layers
- mechanical polishing
- ion milling



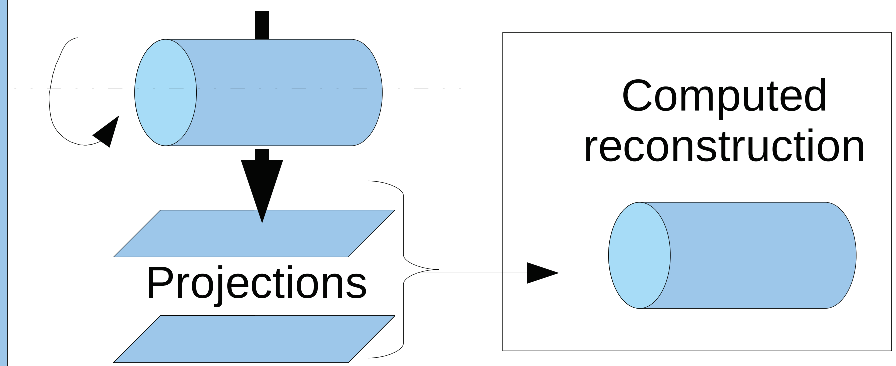
+ Imaging
ex situ : OM, SEM
or in situ : SEM, ion, atome probing



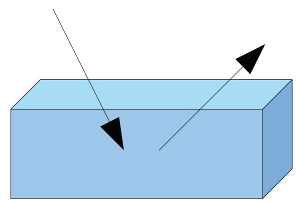
Selective dissolution

Non destructive methods

Transmission + rotation :
electrons or X rays



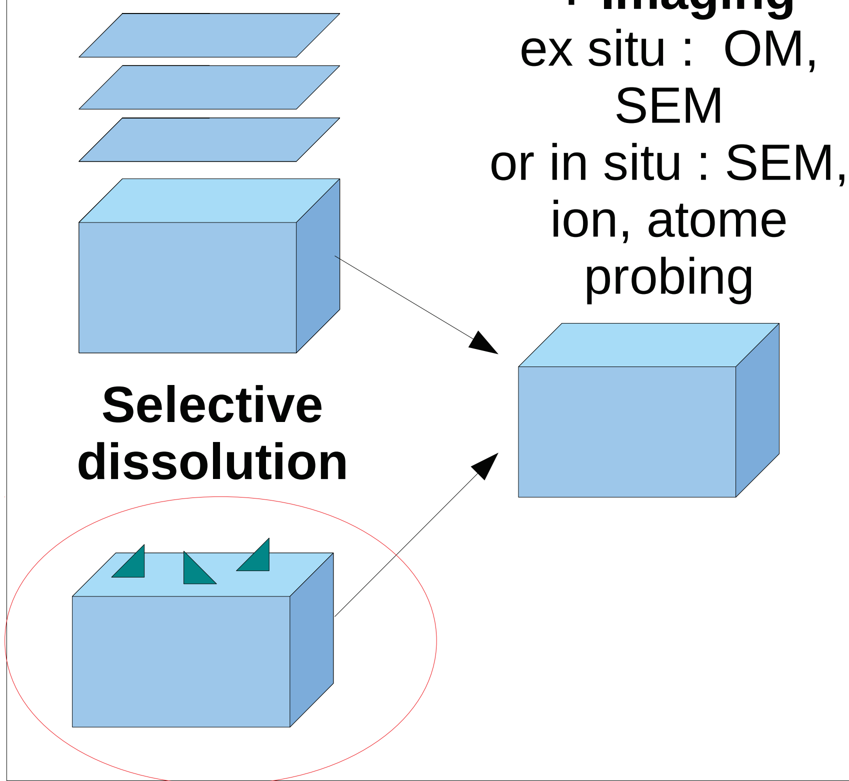
Reflexion, emission or scattering
Acoustic waves, magnetic field, X rays

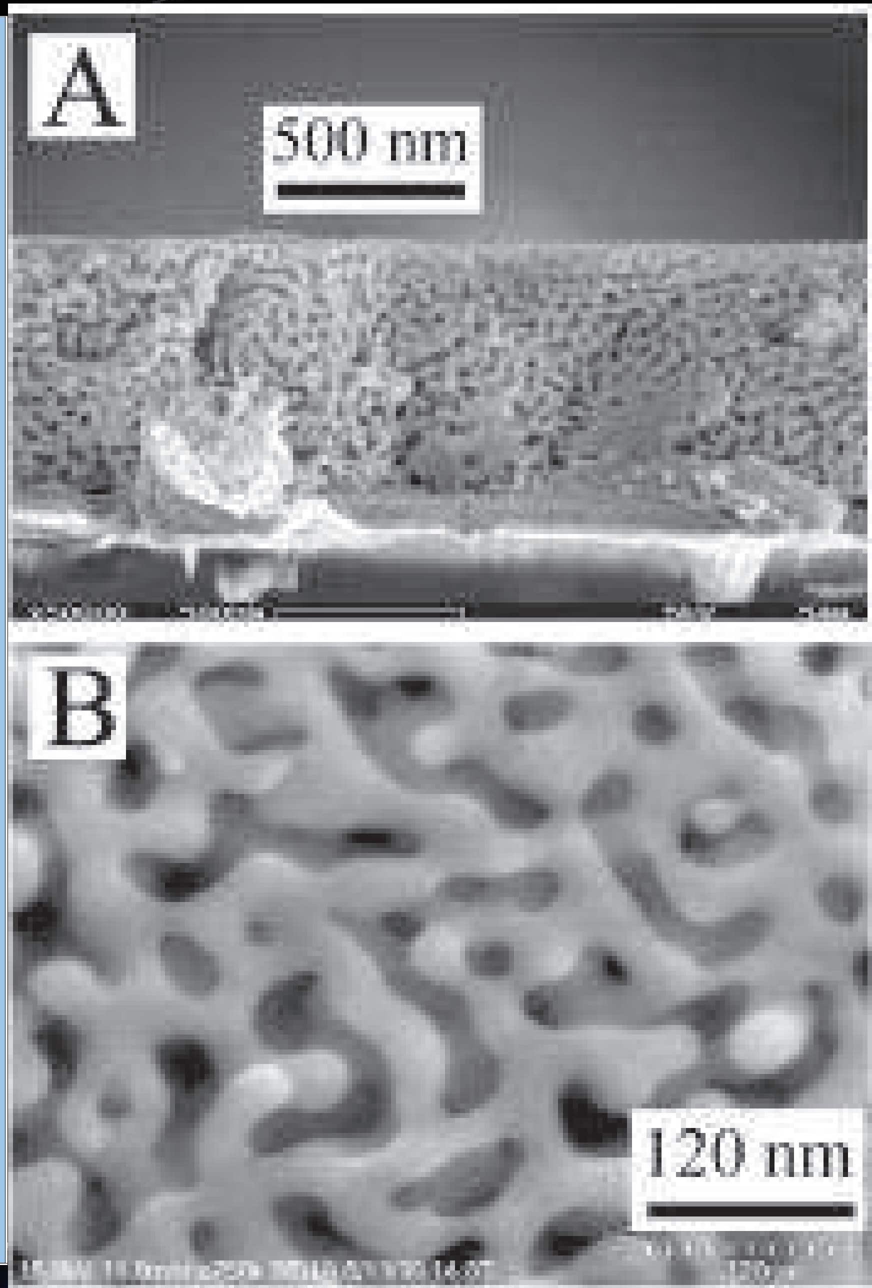


1. Selective dissolution, deep ecthing

Destructive methods

Successive abrasion of layers
- mechanical polishing
- ion milling





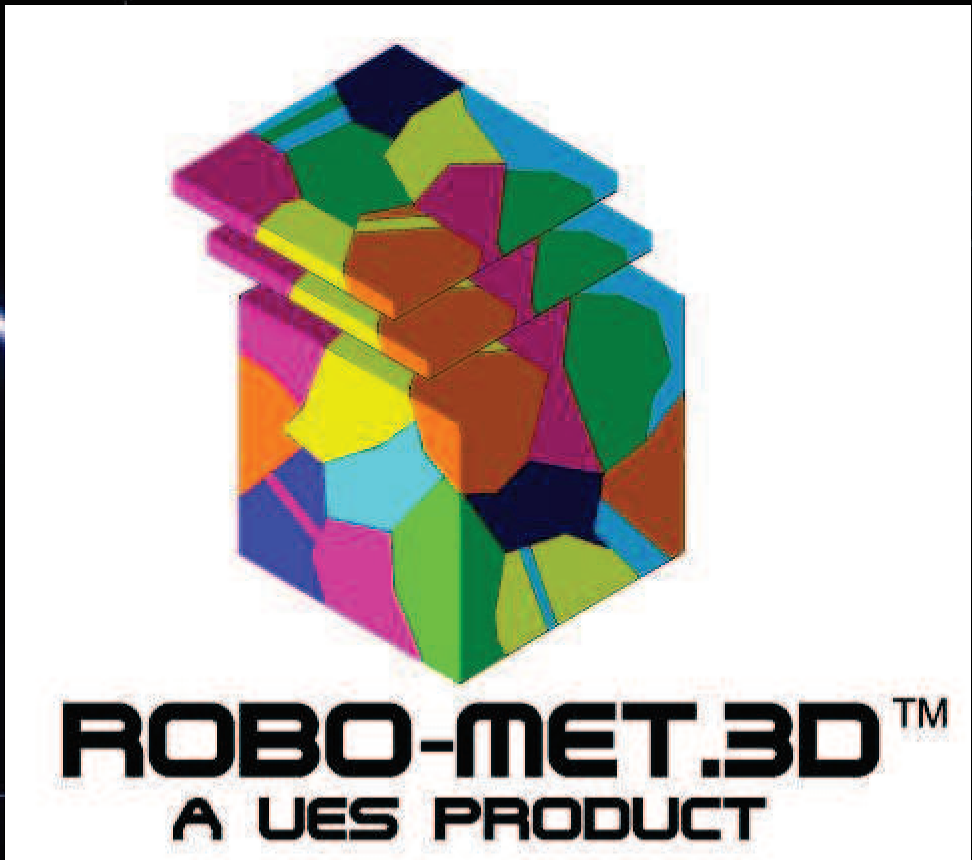
2. Serial sectioning

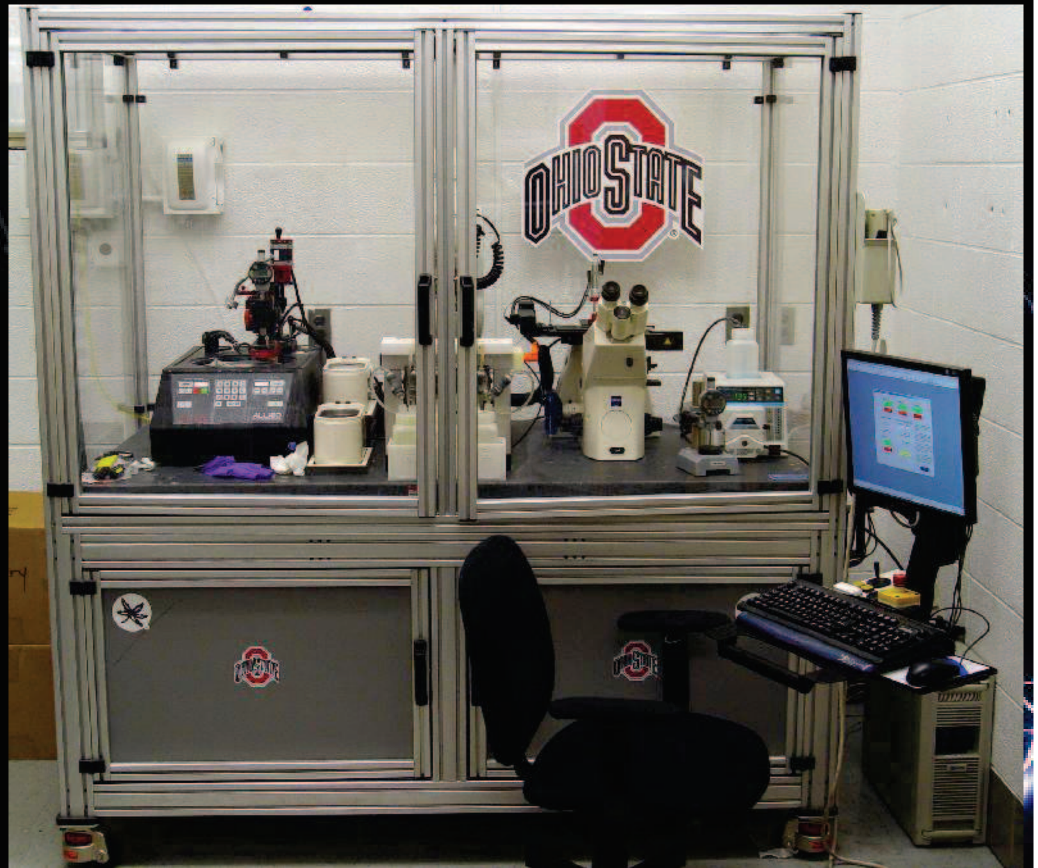
2.1 Mechanical polishing

2.2 Ion milling

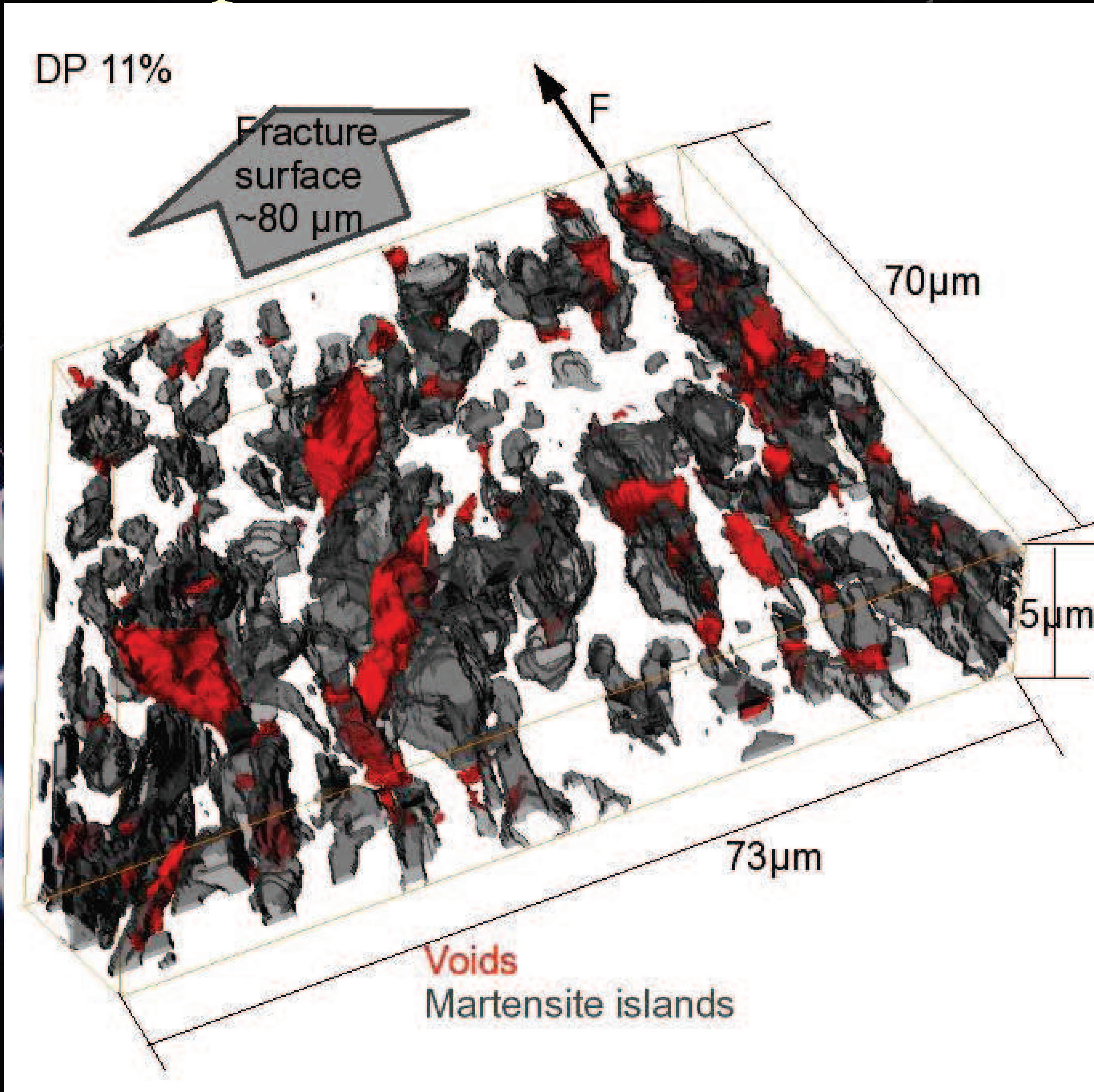
Existing devices to do mechanical polishing for serial sectionning automatically

- Serial sectioning + optical microscopy





Example : ferrite martensite + cavities



PhD C Landron
Collaboration
TU Vienna

Ion milling

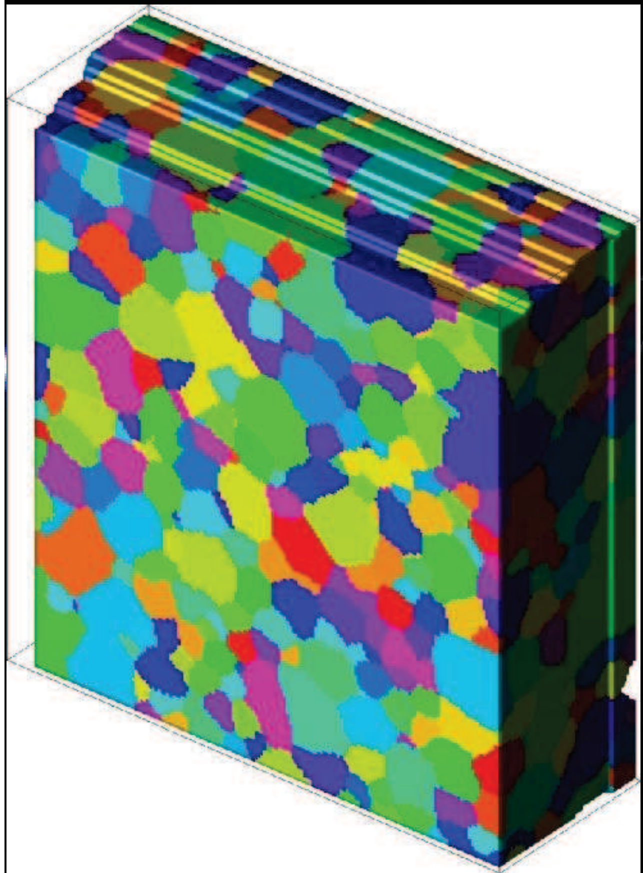
- Can be done like serial sectioning
- New devices: dual beam
  - Ion milling
  - SEM imaging
    - EDX
    - SEI, BEI
    - EBSD

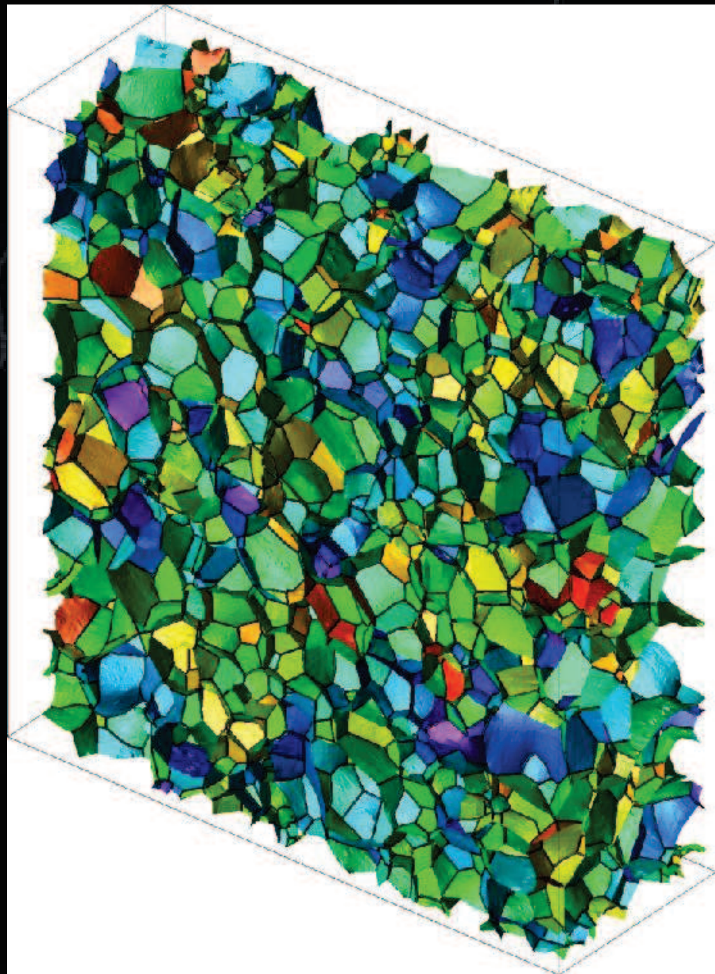
Duplex stainless steel

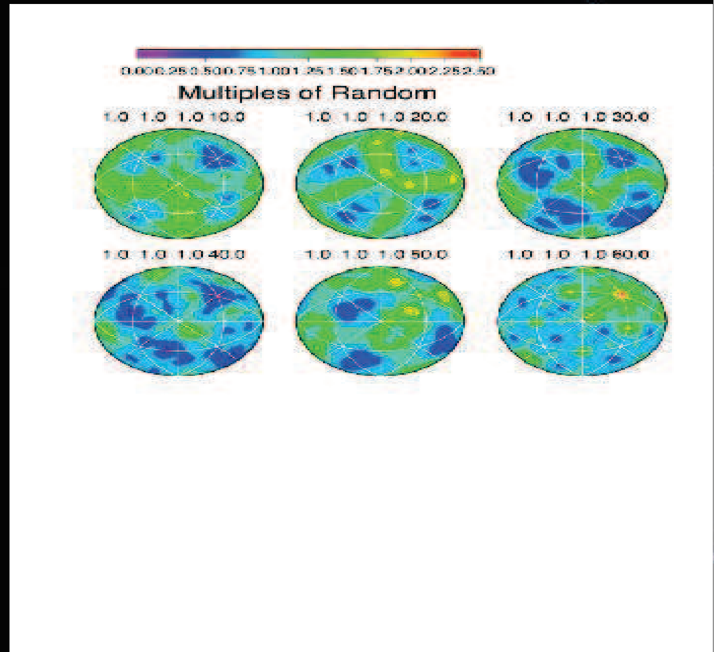
JY Maetz, S Cazottes, C Verdu, X Kléber

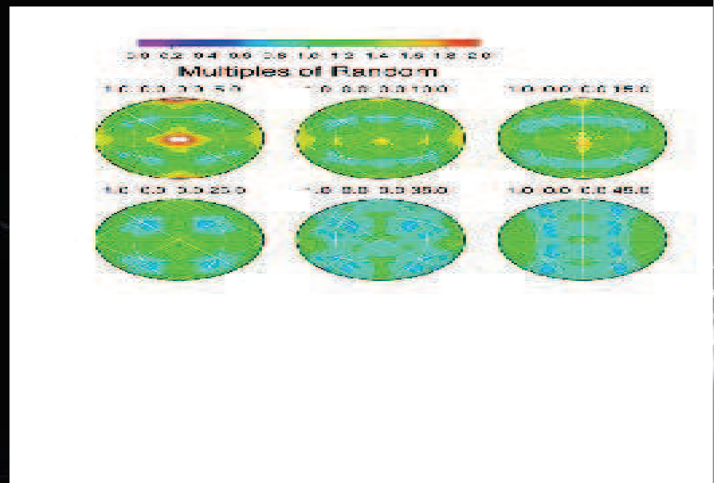
Images : FIB ZEISS du CLYM

Yttria stabilized zirconia results



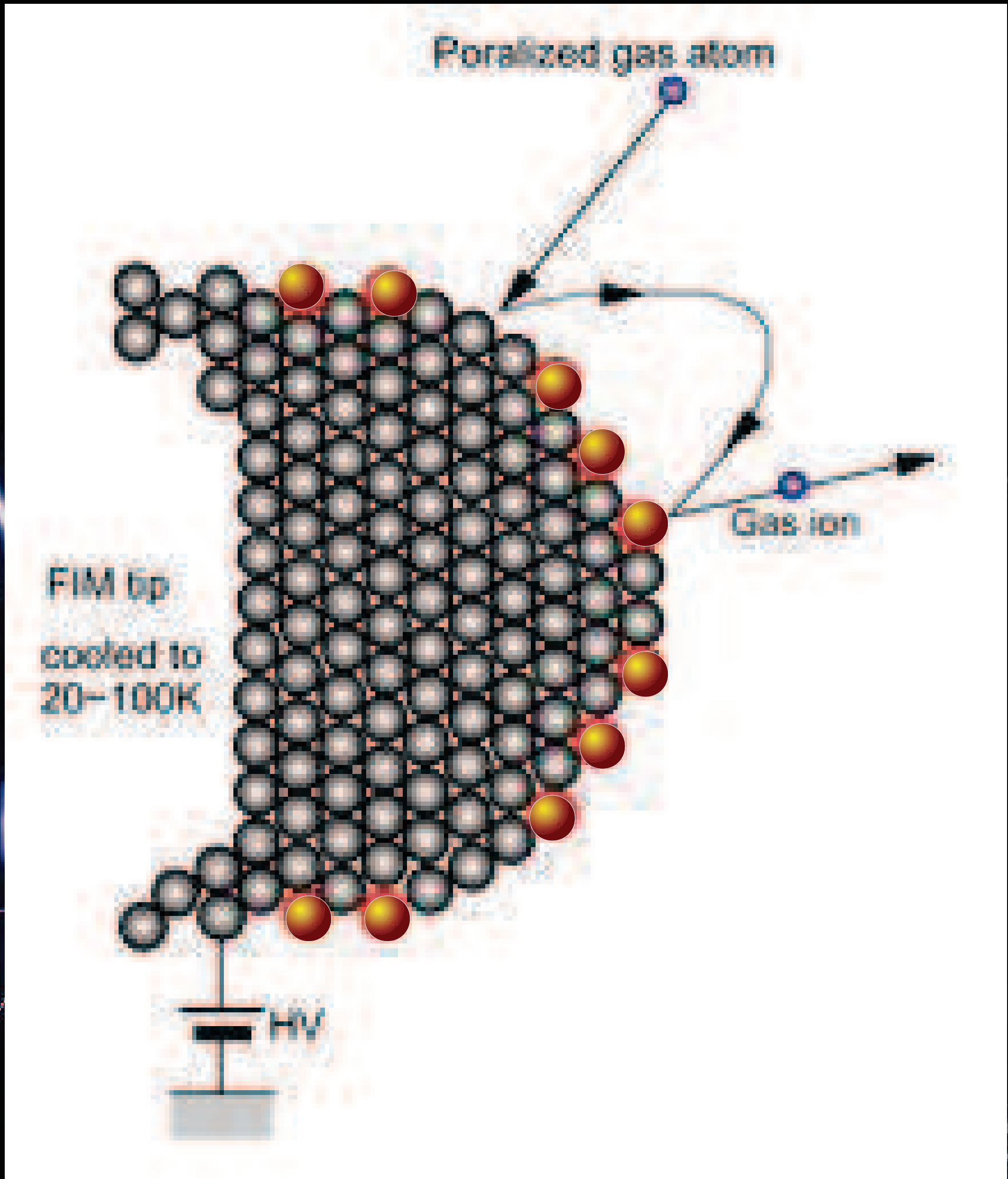


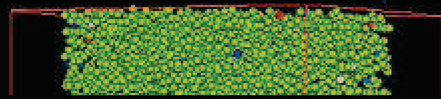




Reconstruction and smoothing by Sukbin Lee
Lee, Dillon, Rollett, Rohrer, *Microscopy and Microanalysis* 2007

Ion milling : Tomographic Atom Probe





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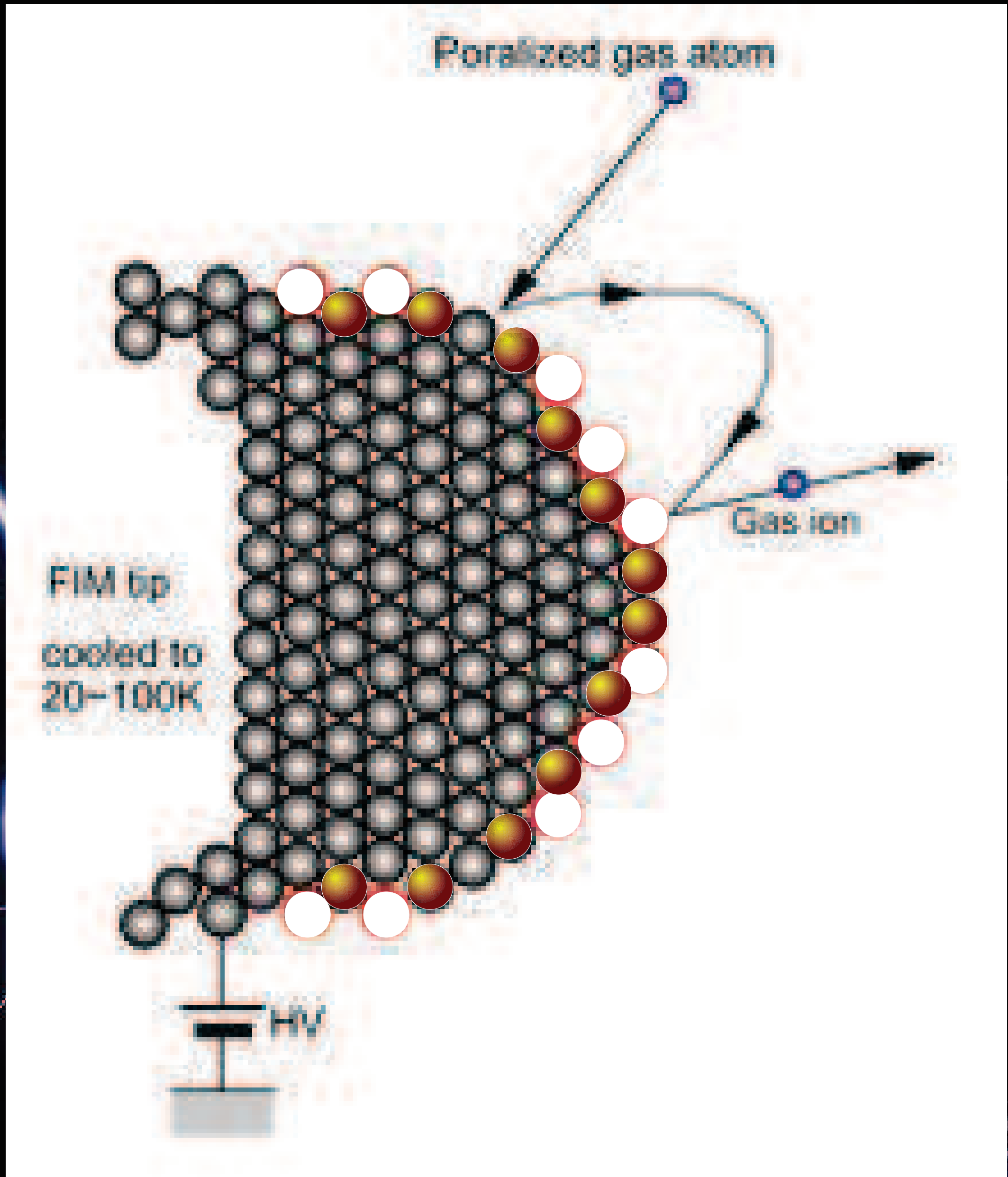

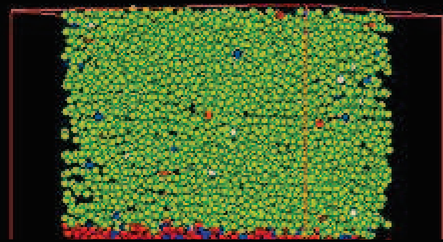

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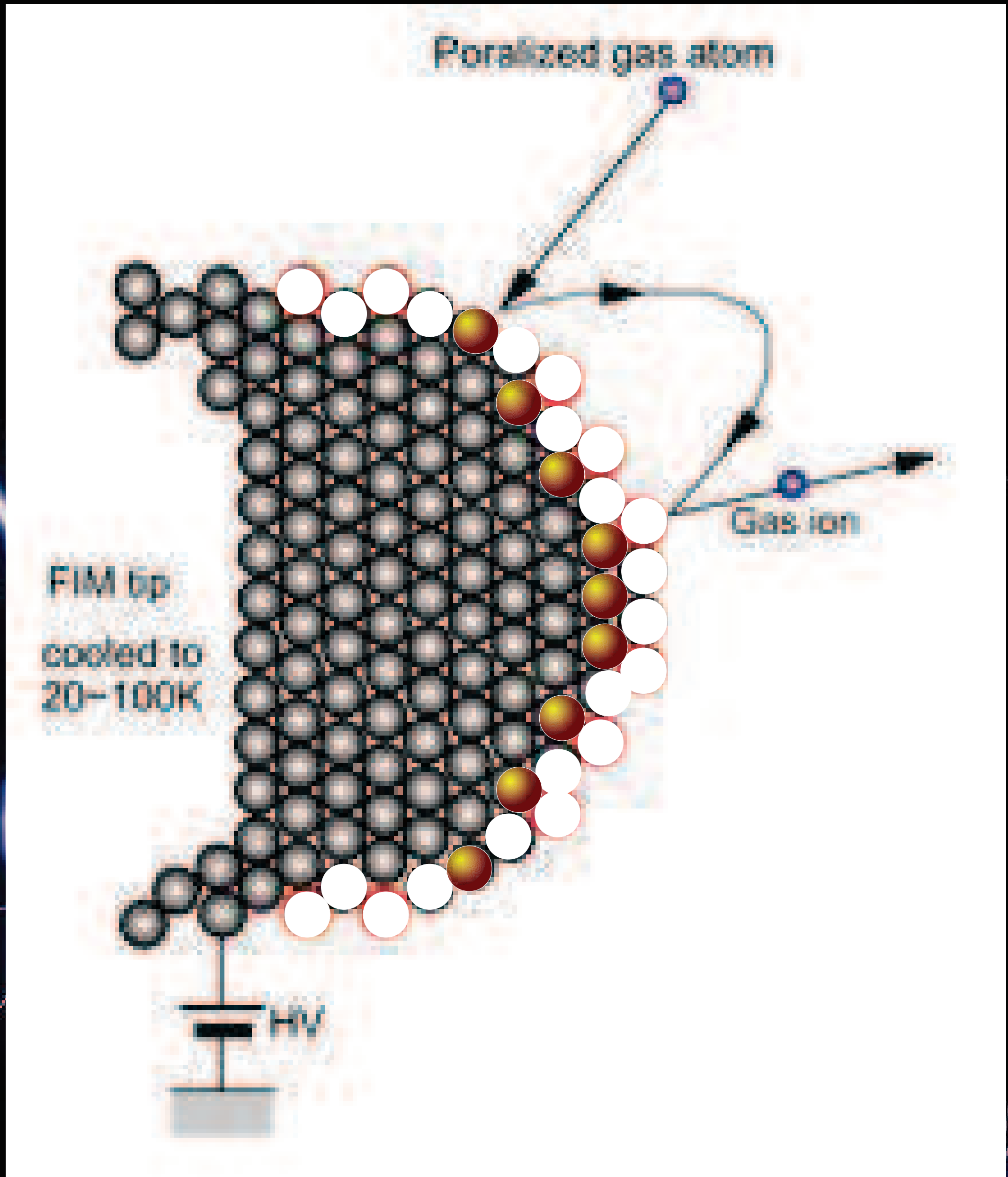
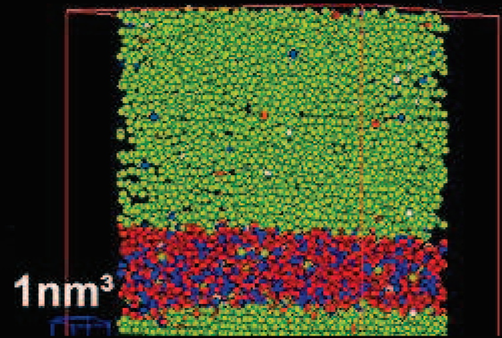
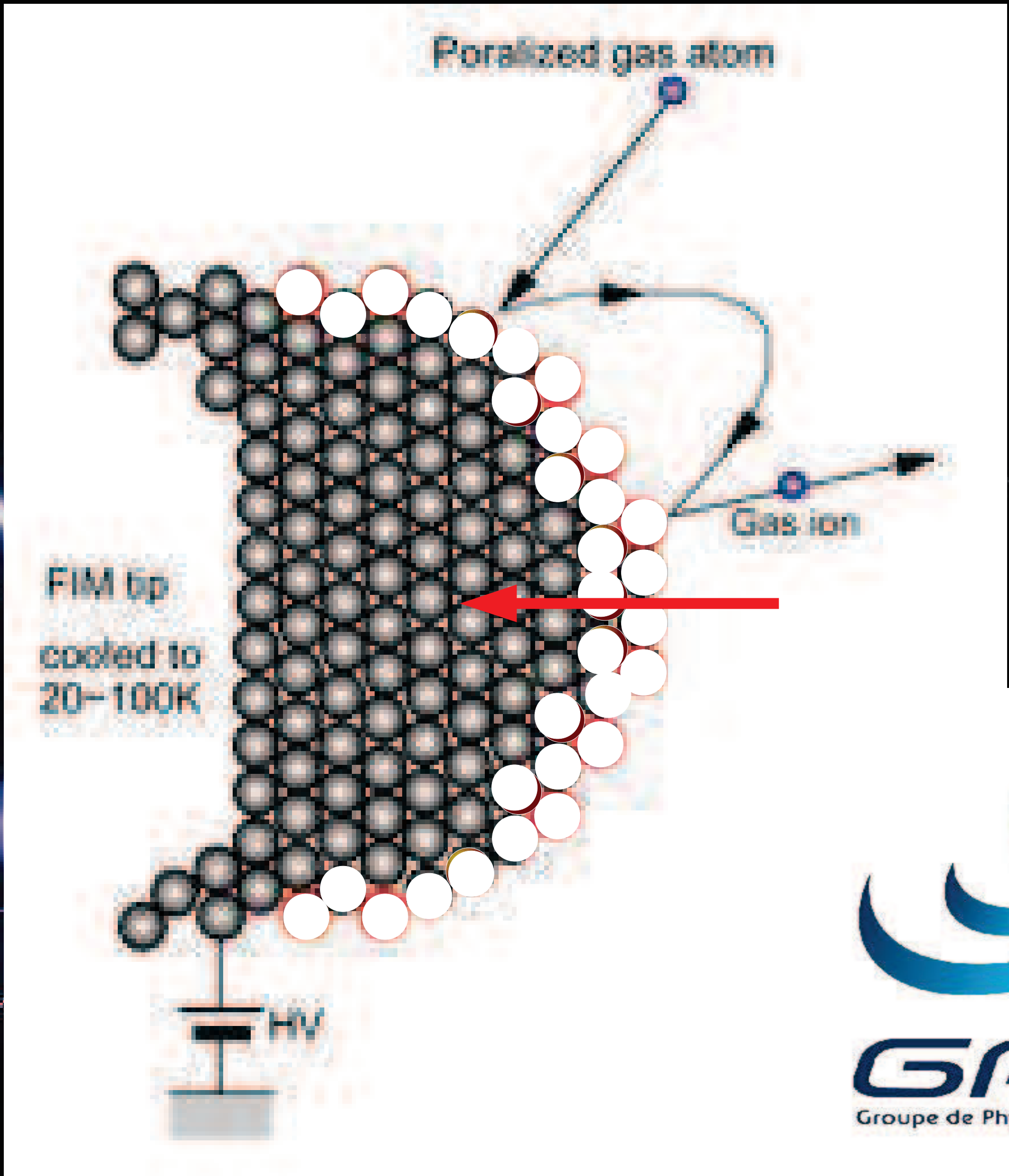
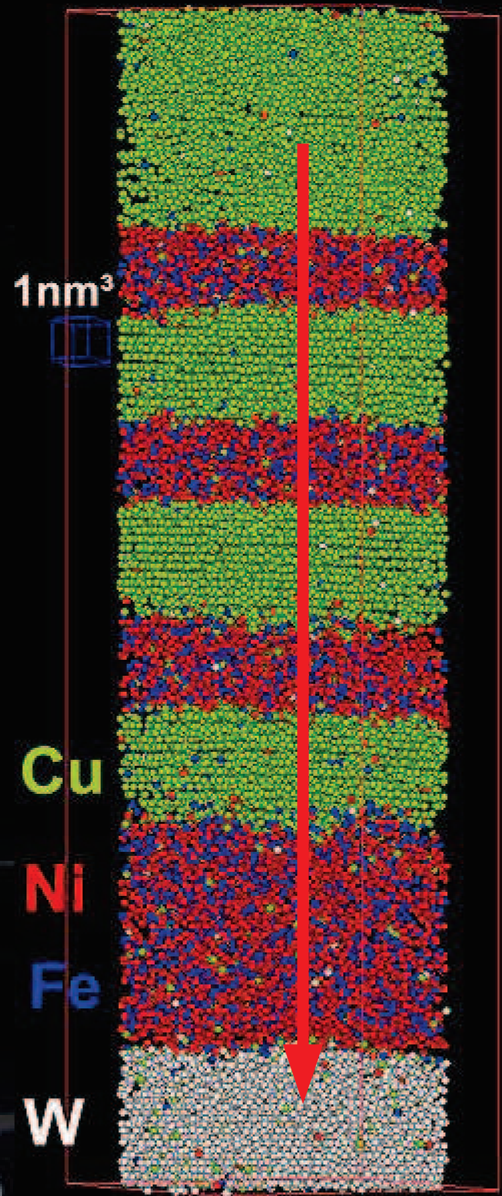


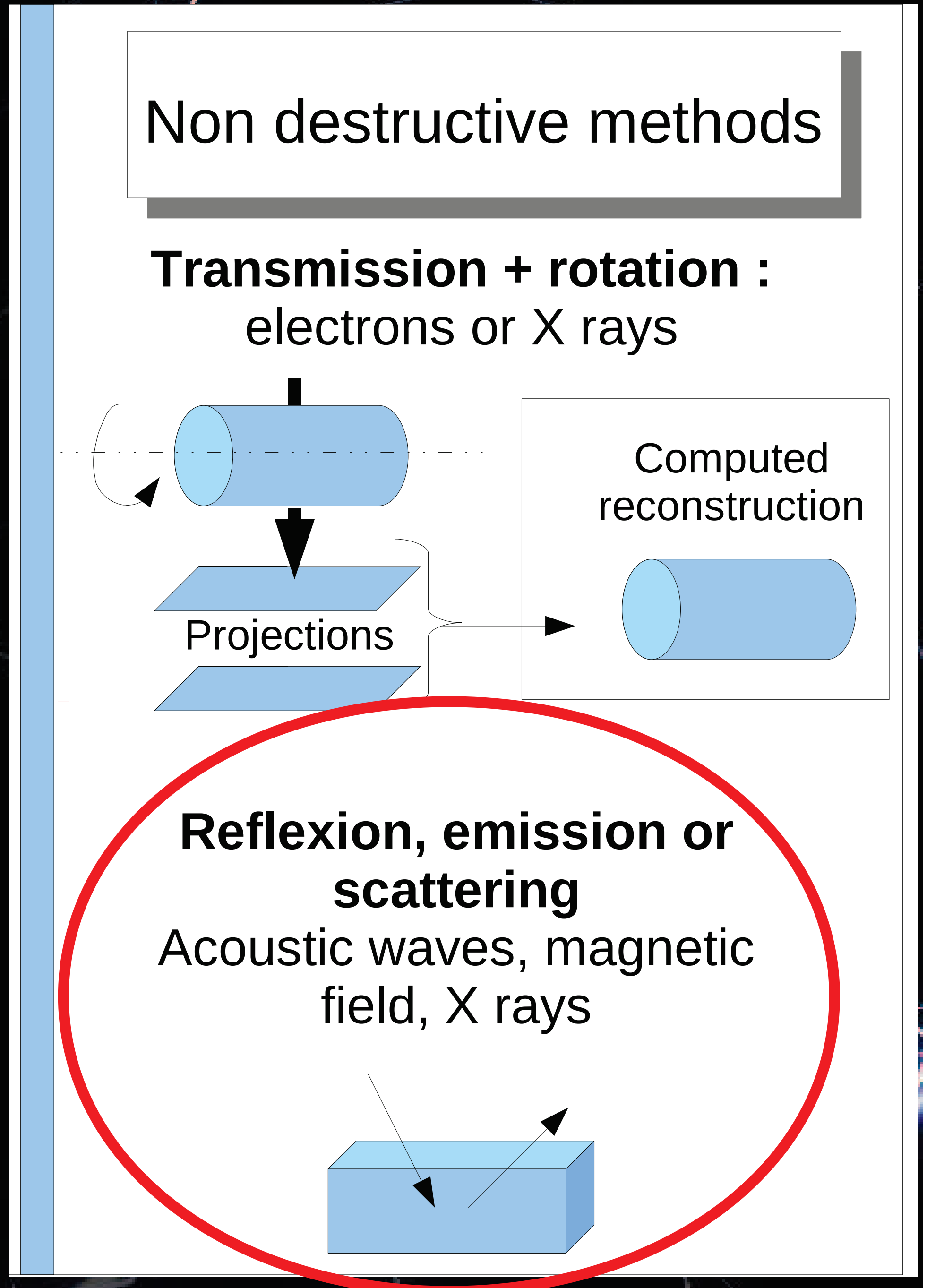


Ion milling : Tomographic Atom Probe

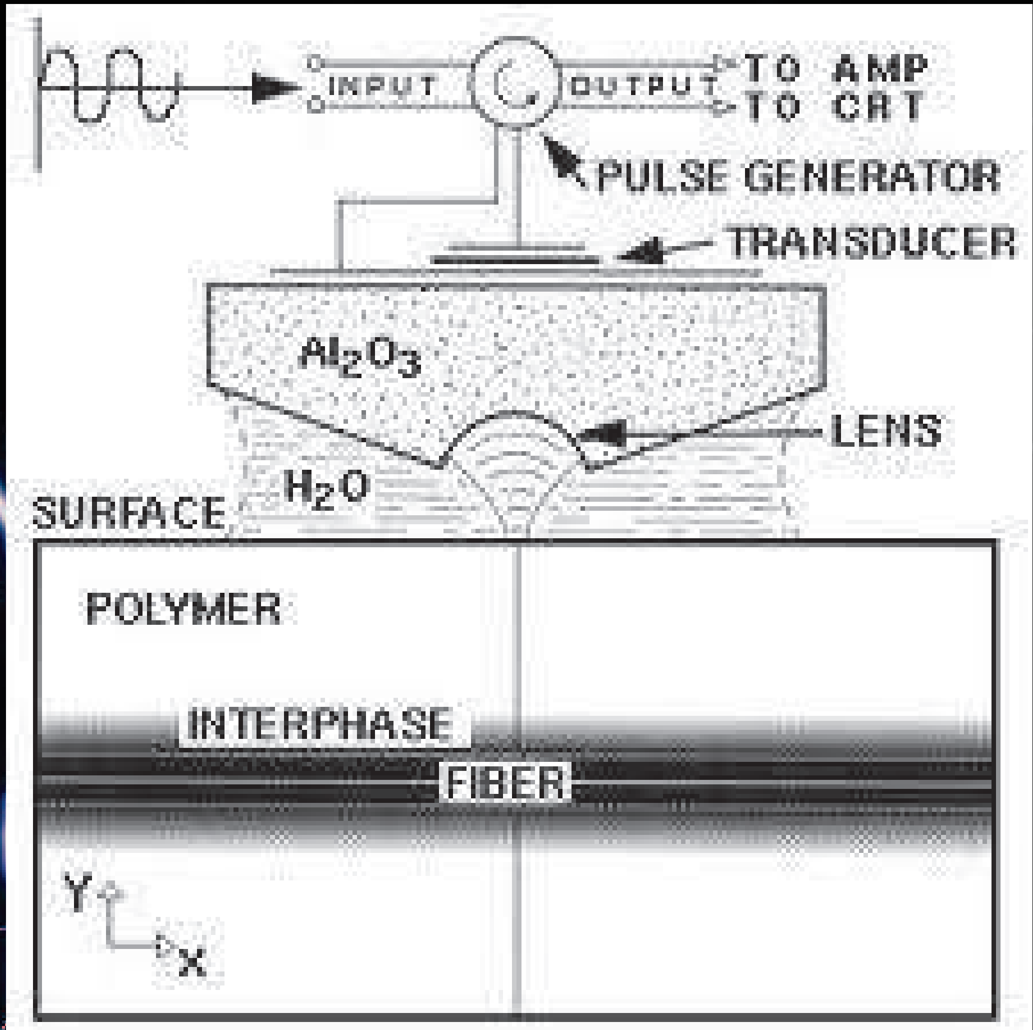


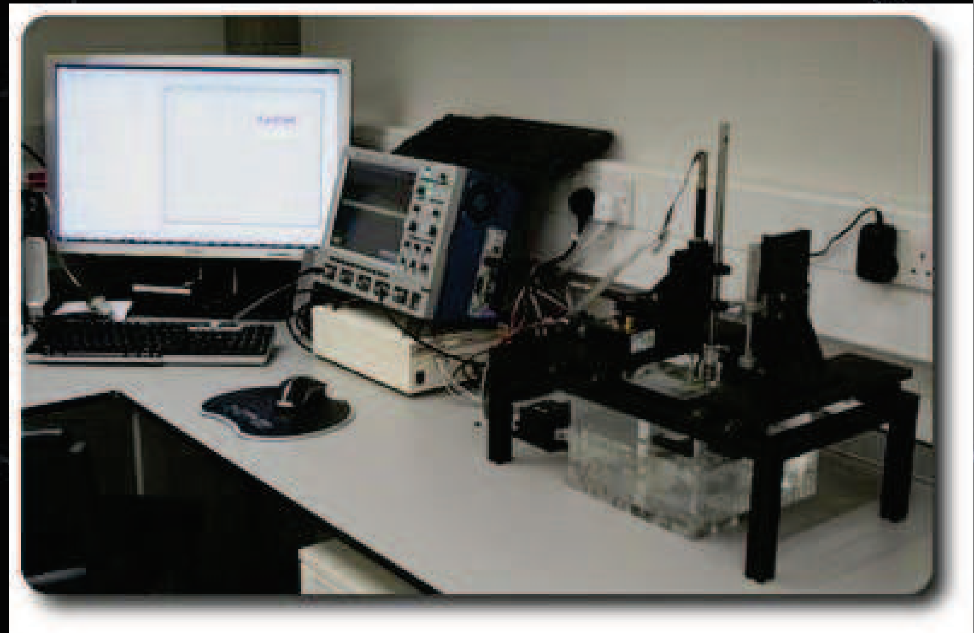


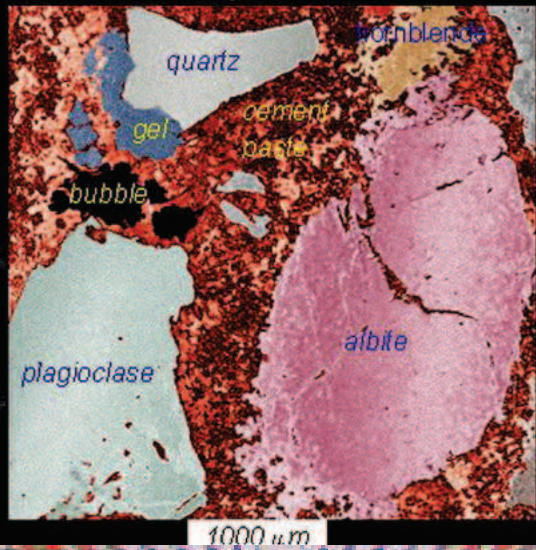
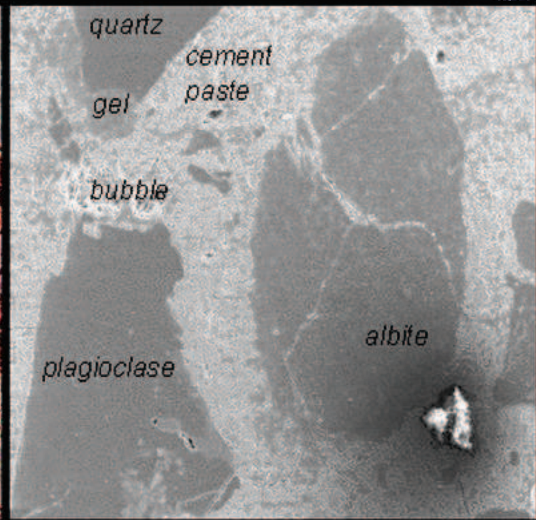
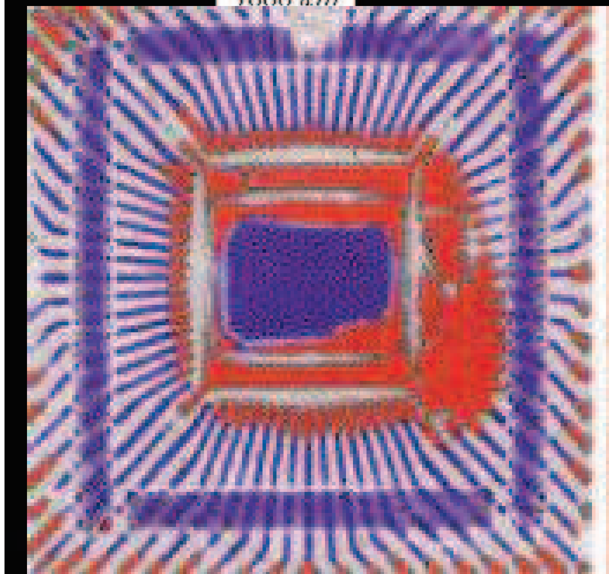
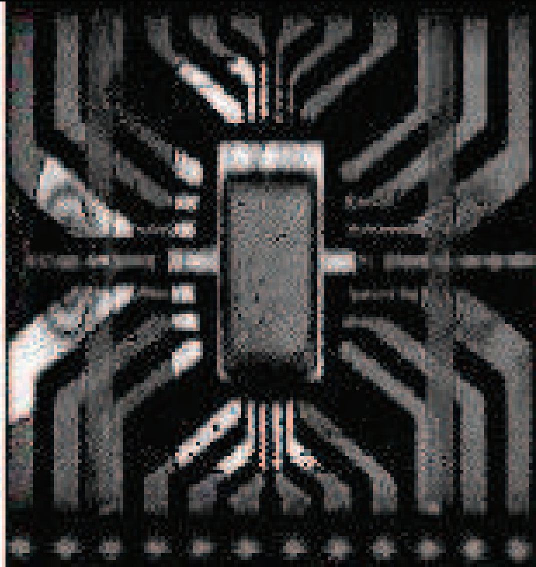
Non destructive methods for 3D imaging



Scanning acoustic microscope





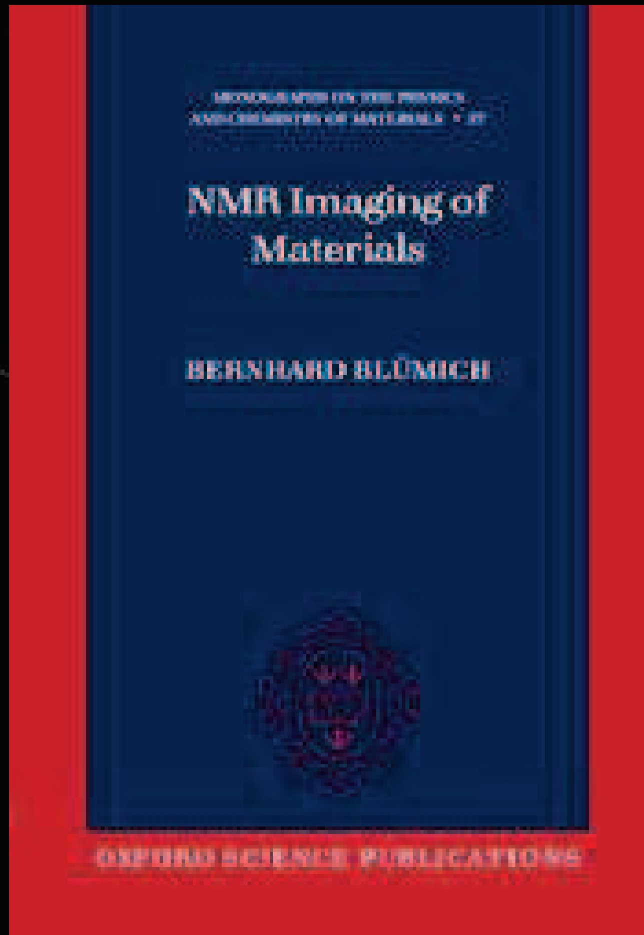

Acoustic
Microscopy

quartz
hornblende
gel
cement
paste
bubble
albite
plagioclase
1000 μm

quartz
cement
paste
gel
bubble
albite
plagioclase



Confocal microscopy of transparent objects

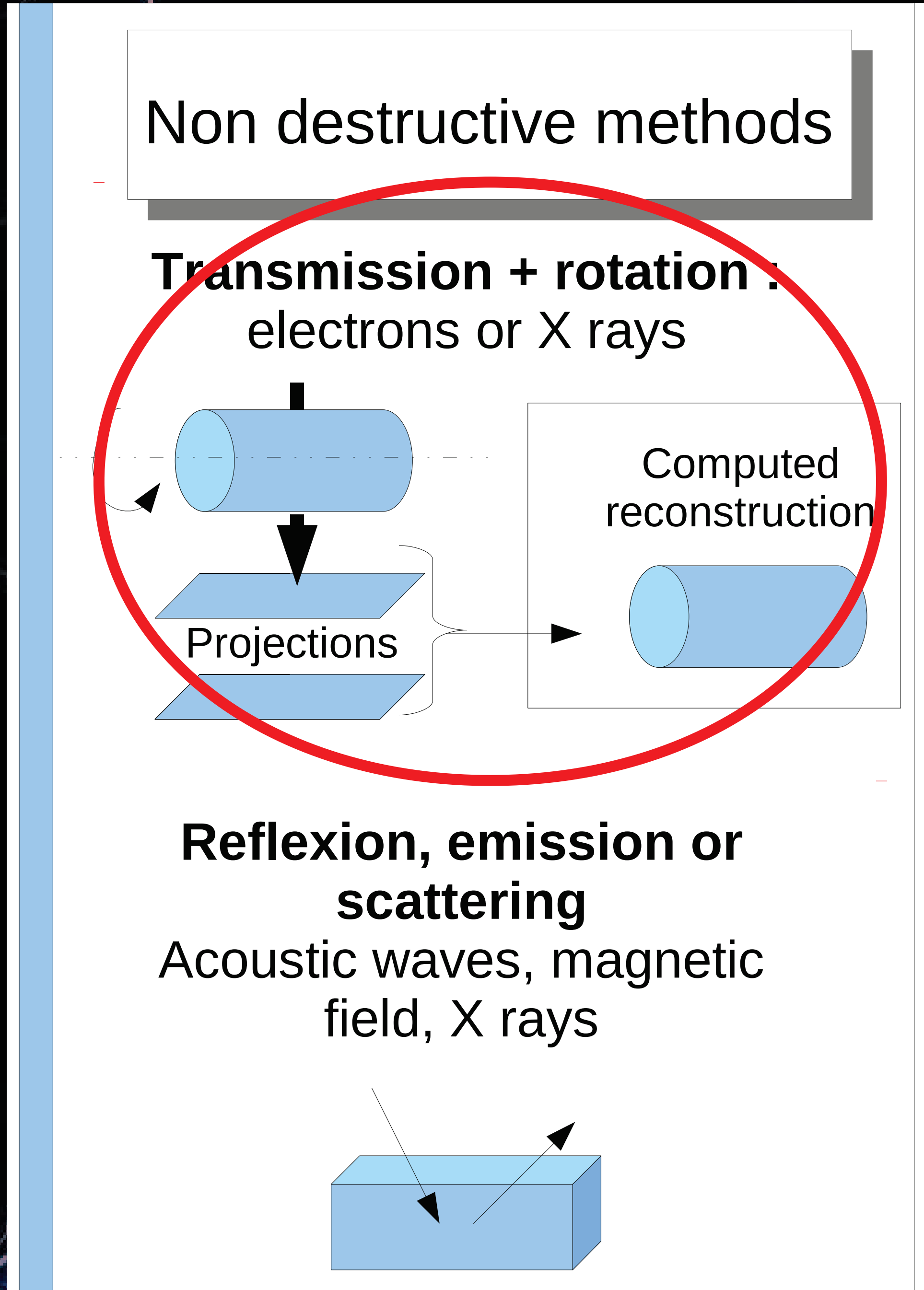
- Send optical light
- If the object is transparent images of different focal planes can be sequentially obtained (contrast = colour)
- Used a lot for colloids
- Food industry (ice cream)
- Biology samples

Nuclear Magnetic Resonance applied to materials

Not many published studies

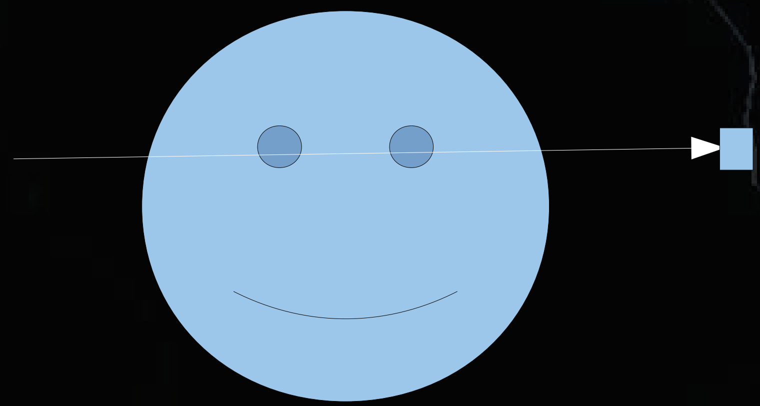


Transmission / Rotation Techniques



Tomography by projection/rotation (+reconstruction)

View from the top :

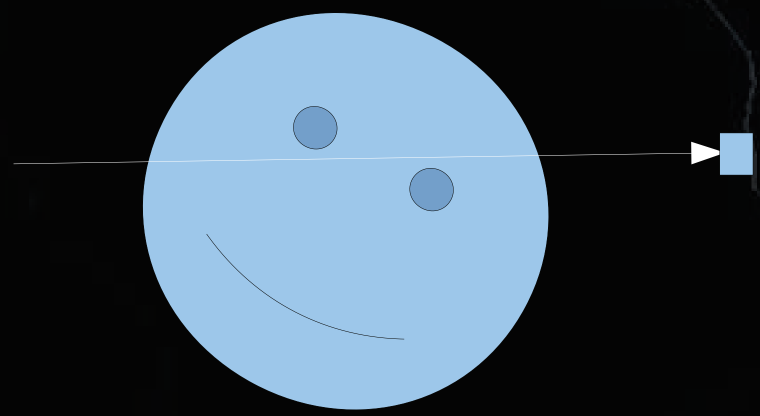


In radiography you record the sum of μ
Ideally one would like to decompose μ along the path

Tomography

- Combines the information of MANY radiographs to reconstruct the 3D map of μ

View from the top :



Tomography

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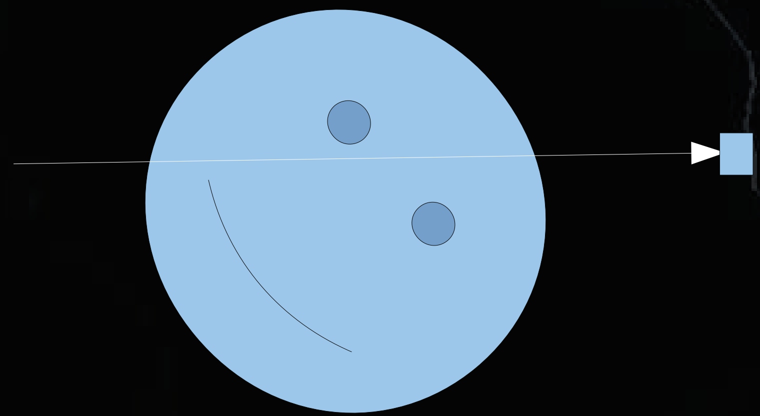
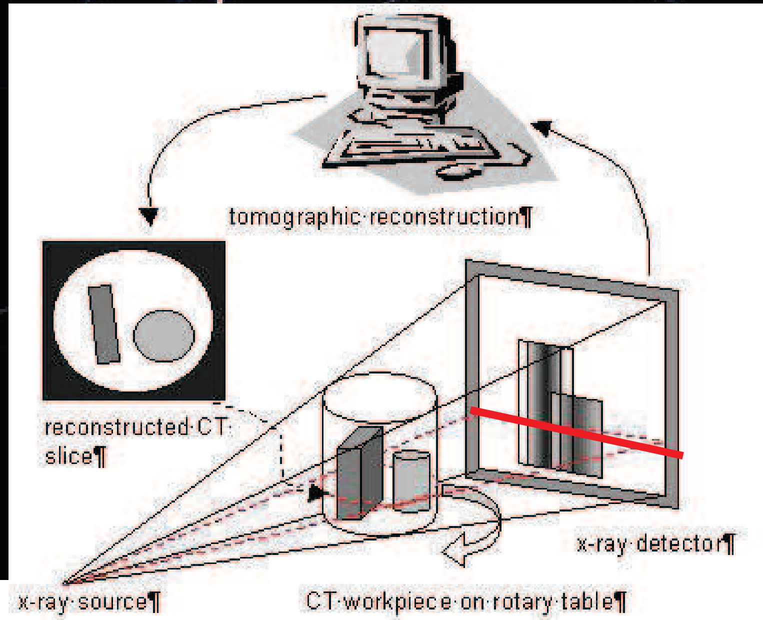
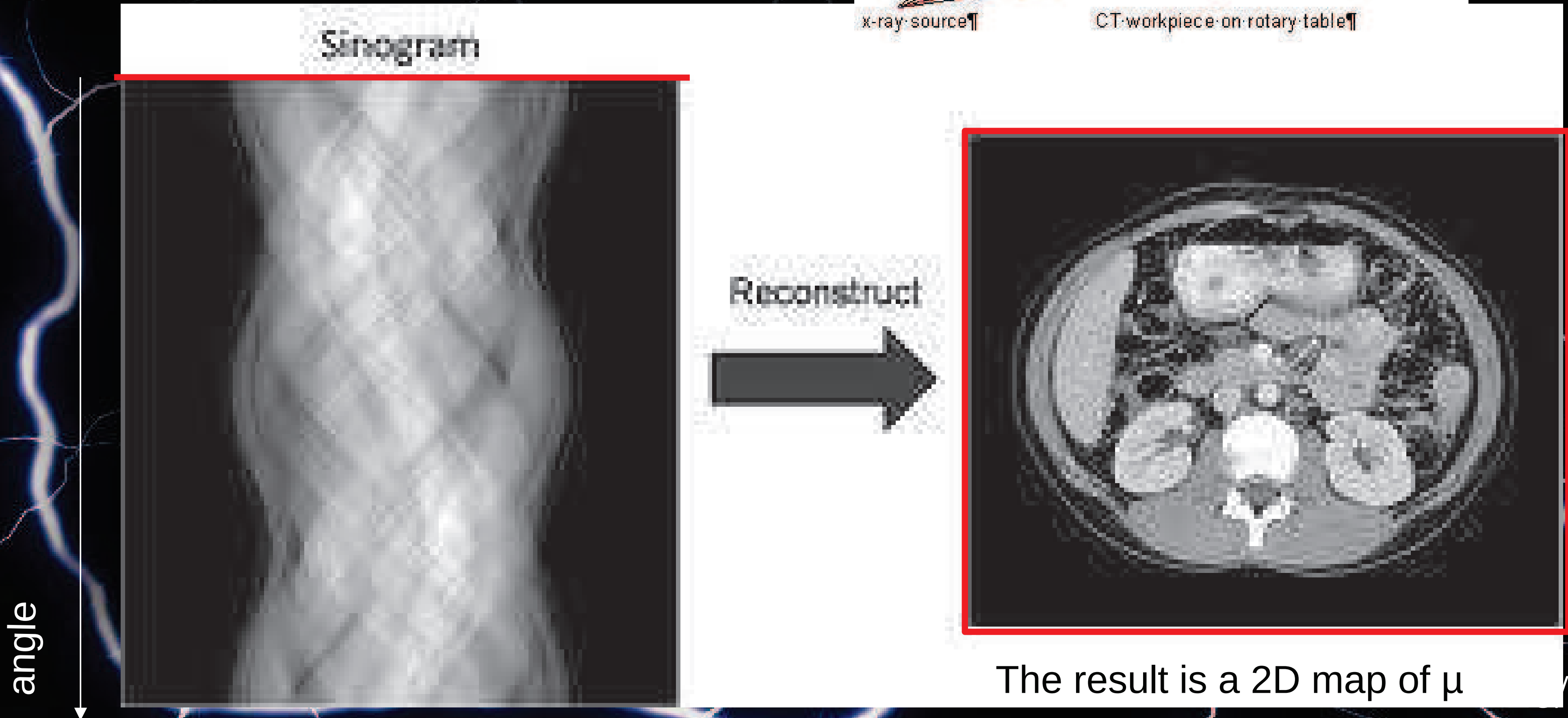
View from the top :



There is a COMPUTED step (reconstruction)

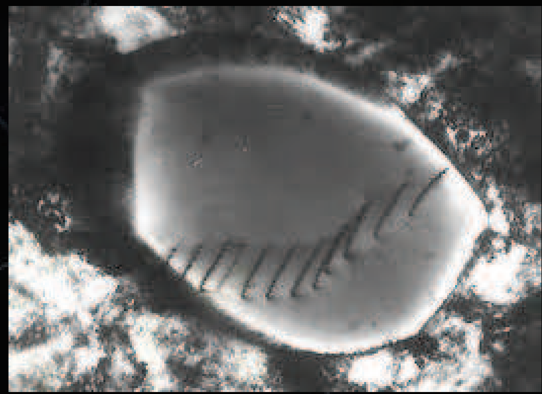


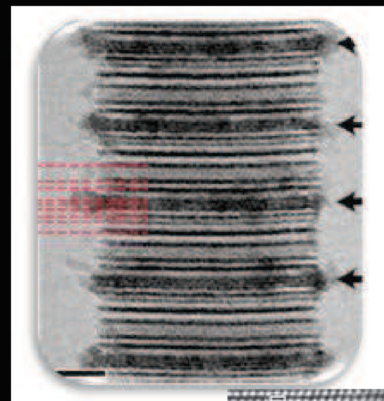


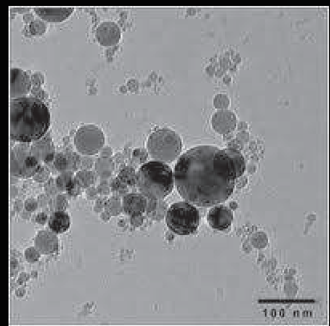
The result is a 2D map of μ

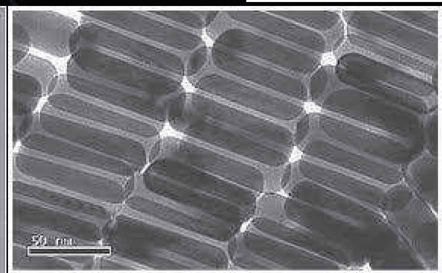
Electrons

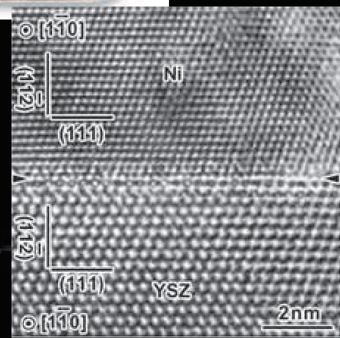
- Transmission electron Microscopy
- Some problems :
  - Absorption
    - Thin samples
  - Diffraction
    - HAADF
    - Energy loss imaging
  - Missing angles
- Cf P. Midgley, T. Epicier









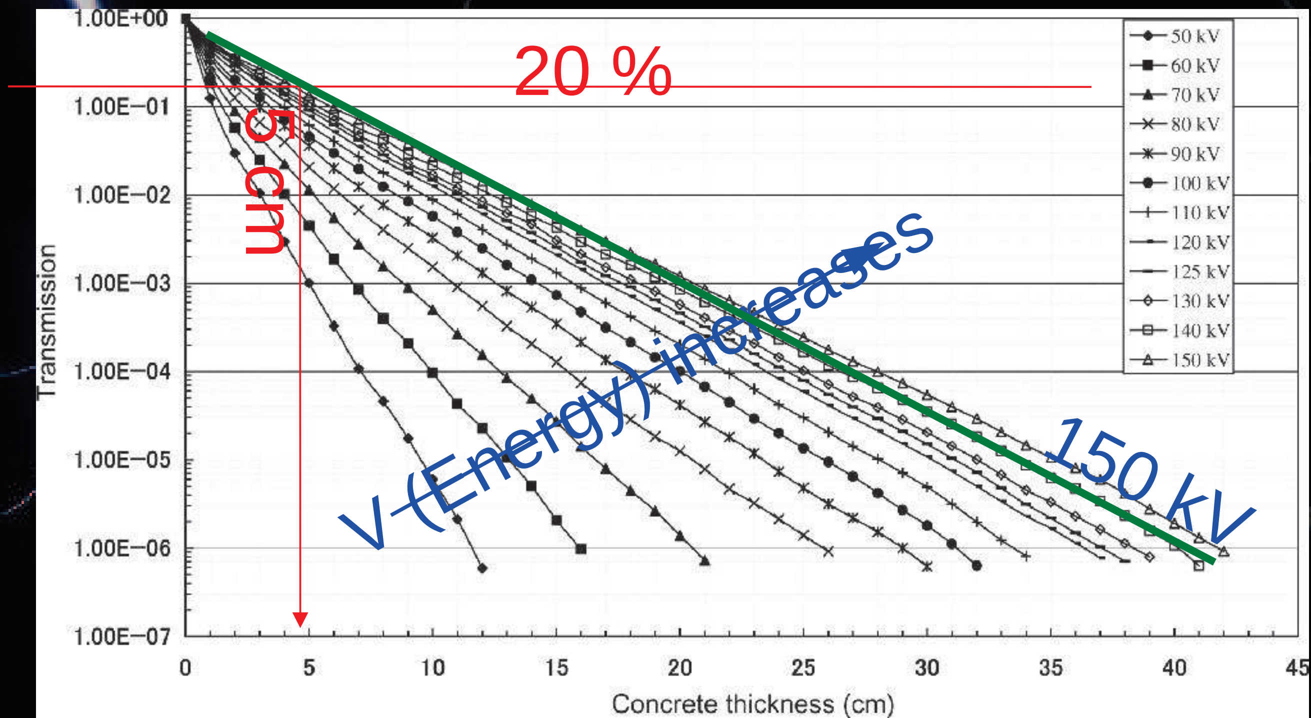


Neutrons

- Penetrate interact and are not absorbed
- Imaging : radio/tomo
- Low flux of the sources
- Low resolution, detectors not so much developed
- **Very useful for highly absorbent** low resolution

X ray imaging

- Limited in terms of resolution (less than SEM although 20 nm is the upcoming target)
- But interesting because non destructive bulk information
  - Transmission of X-rays THROUGH opaque materials



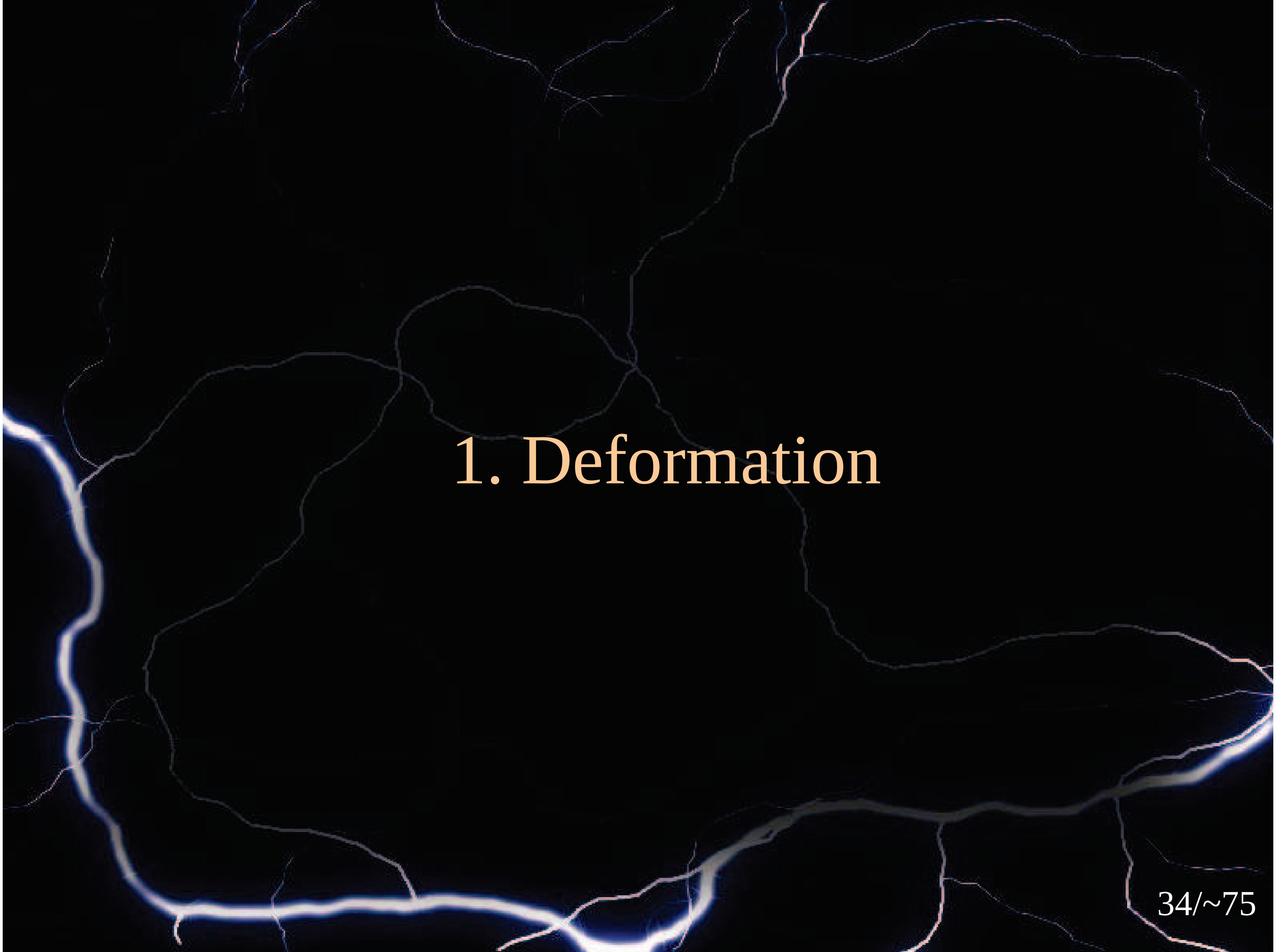
- **Access to information IN THE BULK**
- **Of particular interest for**
  - **Damage studies (because damage is different at the surface)**
  - **Architectured materials materials**

X ray tomography in metallurgy

- Non destructive
- 3D
- “Easy” to access
  - Lab tomographs
  - Synchrotron beam lines
- 16 years of background

In situ experiments in X-Ray tomography

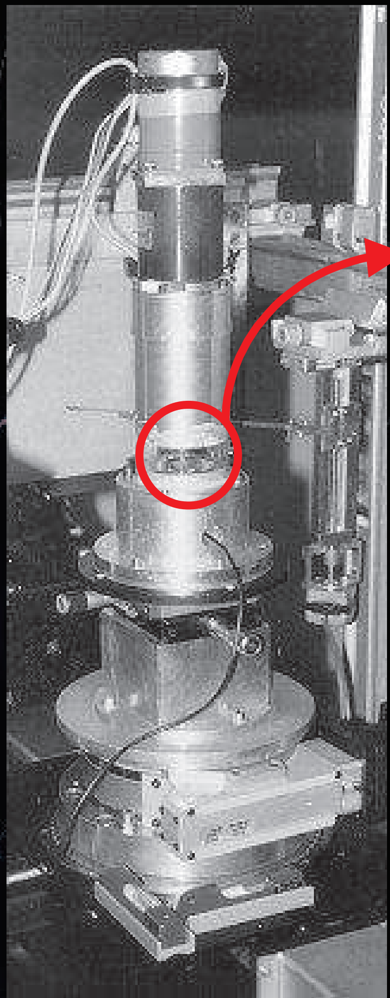
- Restricted to non destructive methods
- Best way to understand the microscopic mechanism involved during your
  - Deformation
  - Transformation
- Apply a stimulus to your material *in situ* (in the tomograph)
  - Mechanical, thermal, electrical…
- + Analyze the change in the microstructure of a similar region of your material

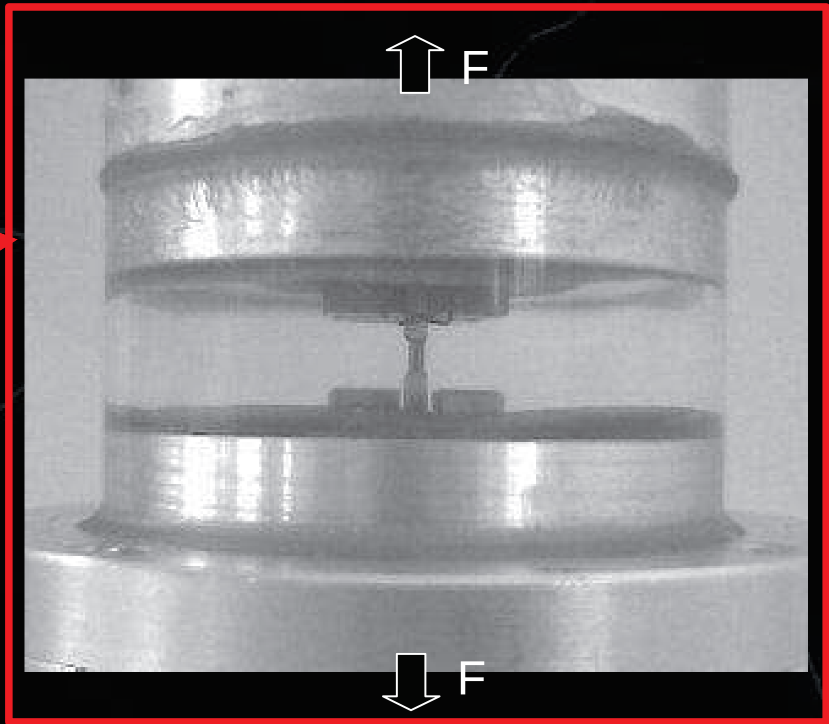


1. Deformation

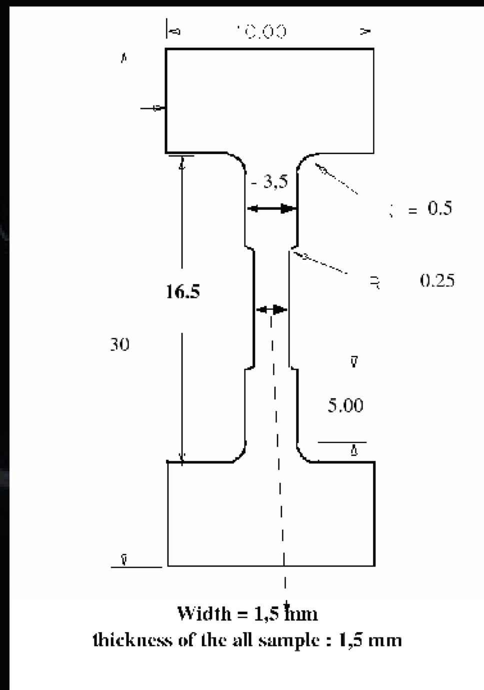
In situ testing

- Tension, compression





•Stepping motor
•Reductor
•F and disp recorded
•10^{-5} – 1 mm/s
•Several Force sensors :
50 – 5000 N
•Grips adapted for different geometries

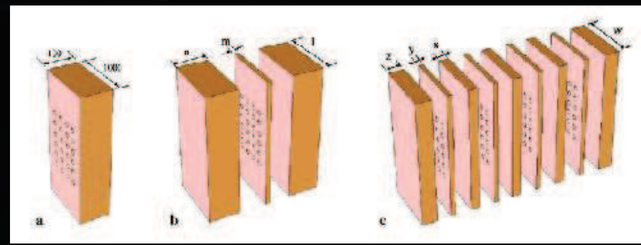


Buffière et al. Acta Mater 1998
Buffiere et al Exp Mech 2009

A lot achieved so far in the interrupted mode (15 years at the ESRF)

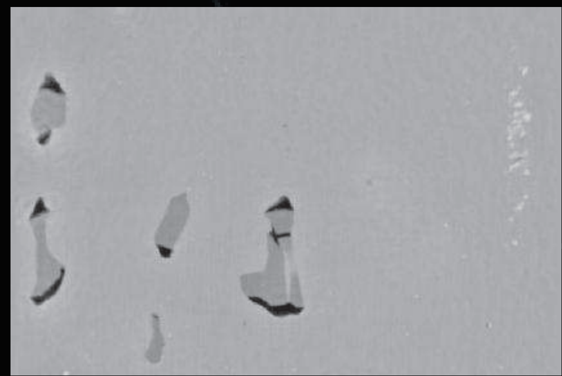
- Al/SiC
- TiSiC
- Al alloys
- Polymers, Composites
- Steels
  - DP
  - Trip
  - TWIP
- Co, Cu, Ti

- Model materials



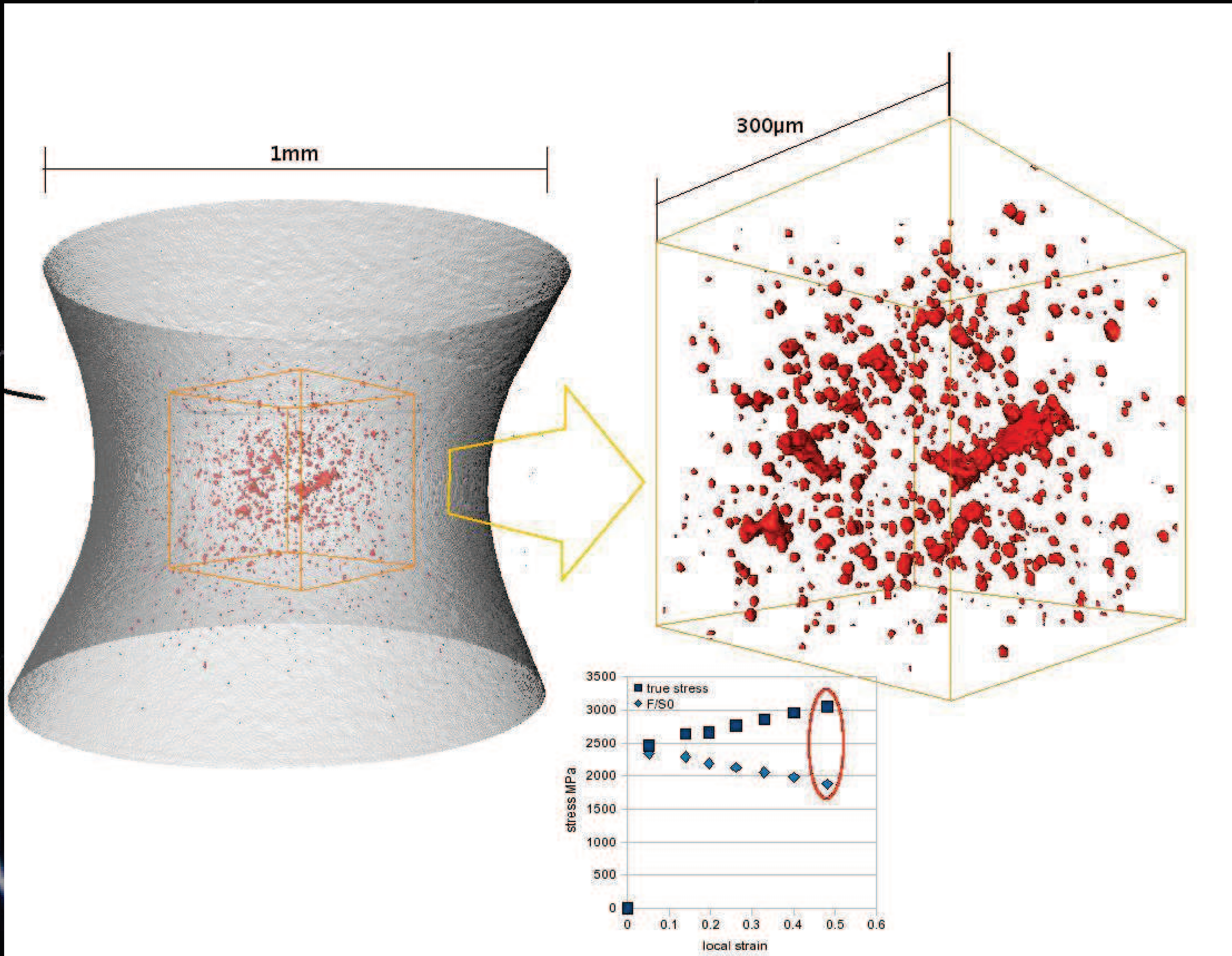
Cu sheets
Mc Master University

- Industrial

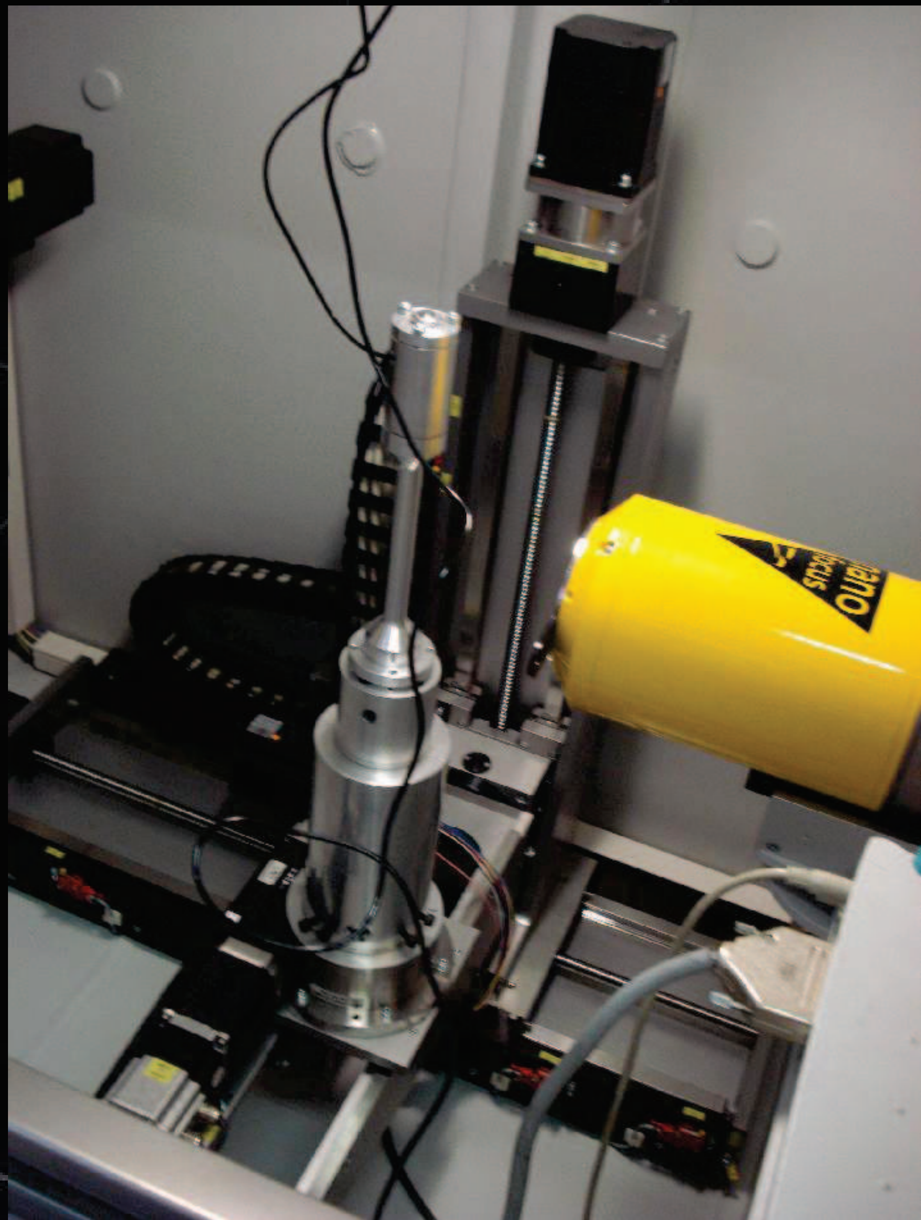


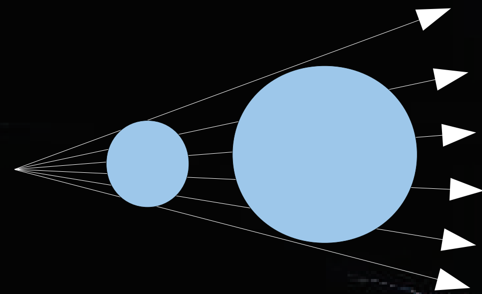
Al alloy
5xxx

CoCr Biomedical material



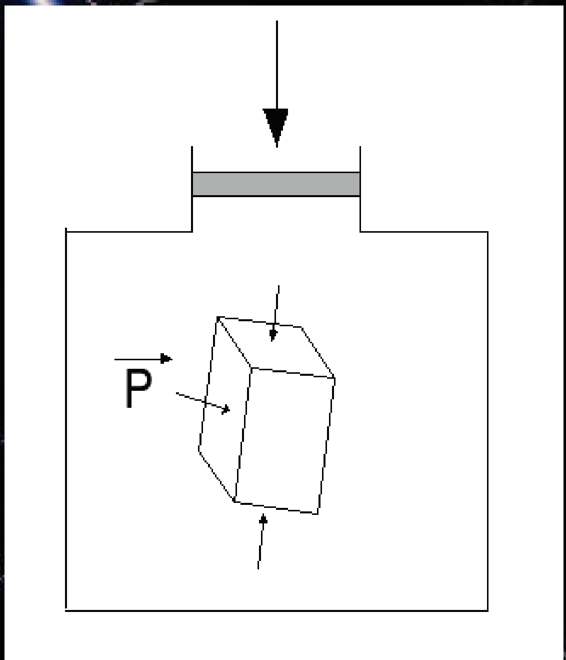
For a lab tomograph :

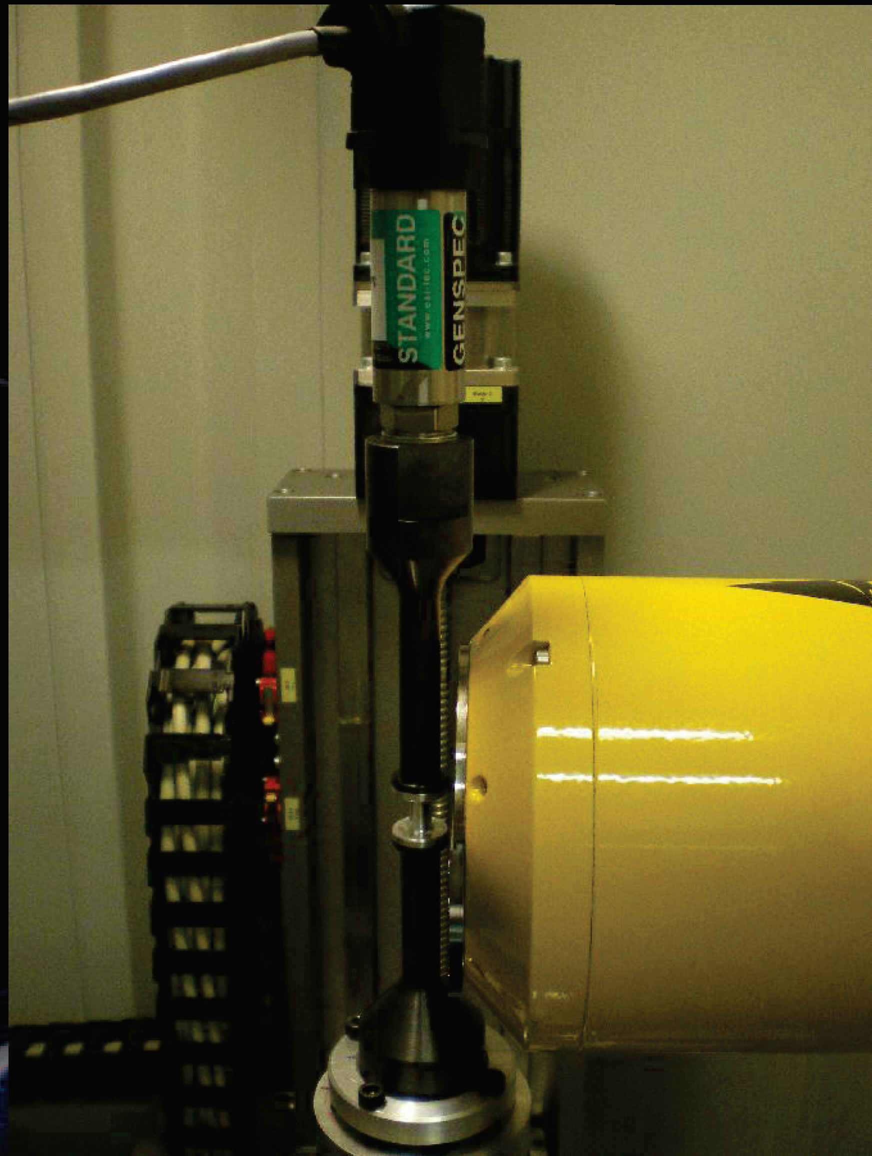




Hydrostatic compression

For the collapse of polymer foams in sea water
J. Lachambre, D. Choqueuse





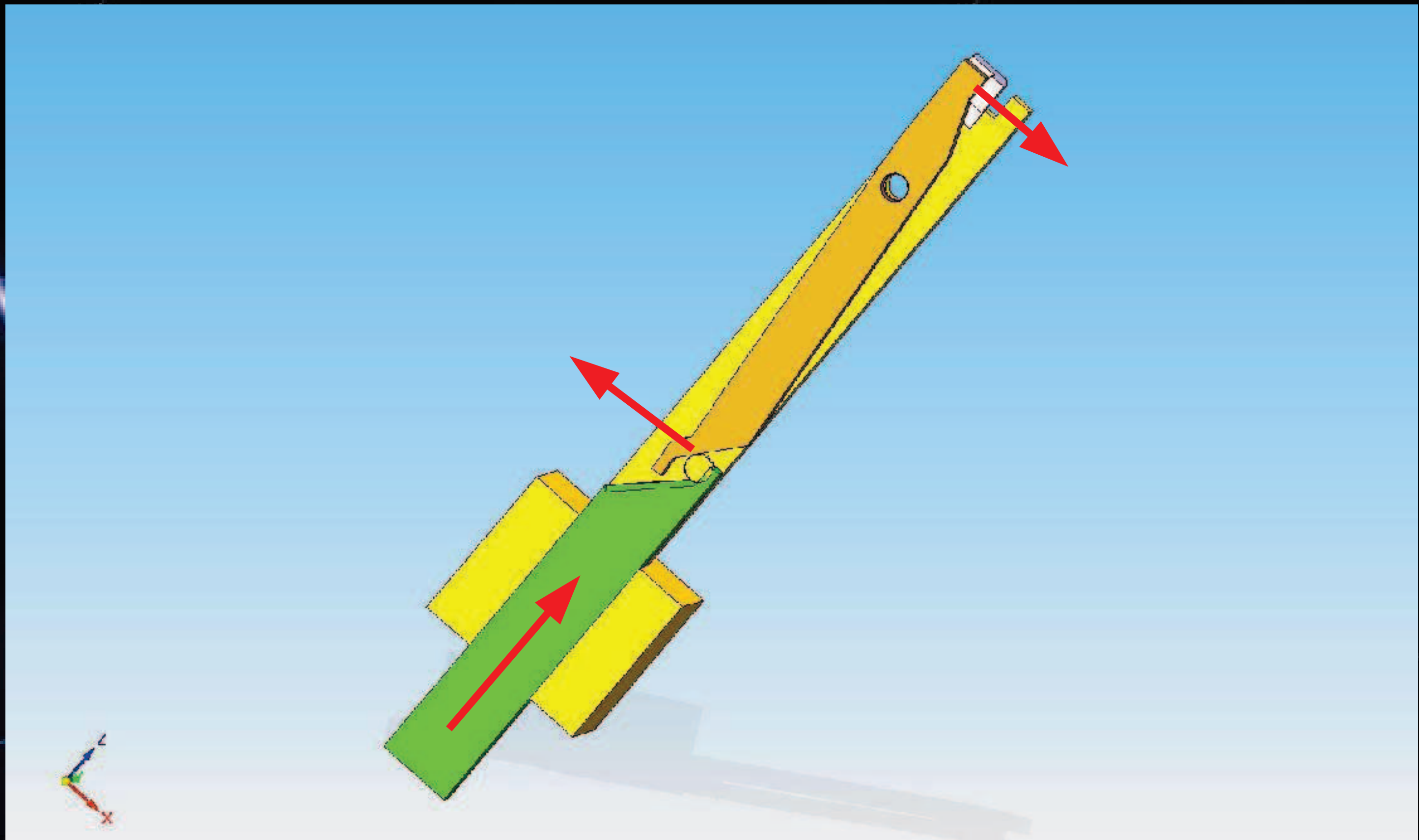
(collaboration IFREMER)

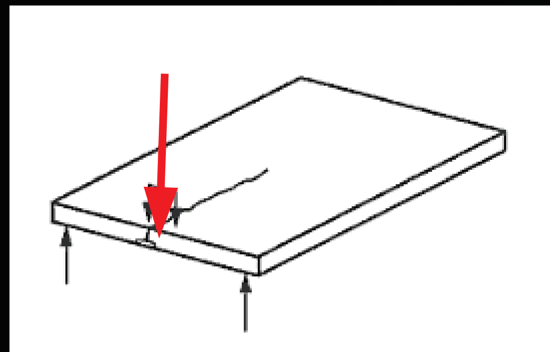
Indentation

- PhD P Clément, S Meille

Double torsion INSA

- « Stable » crack propagation Ceramic materials

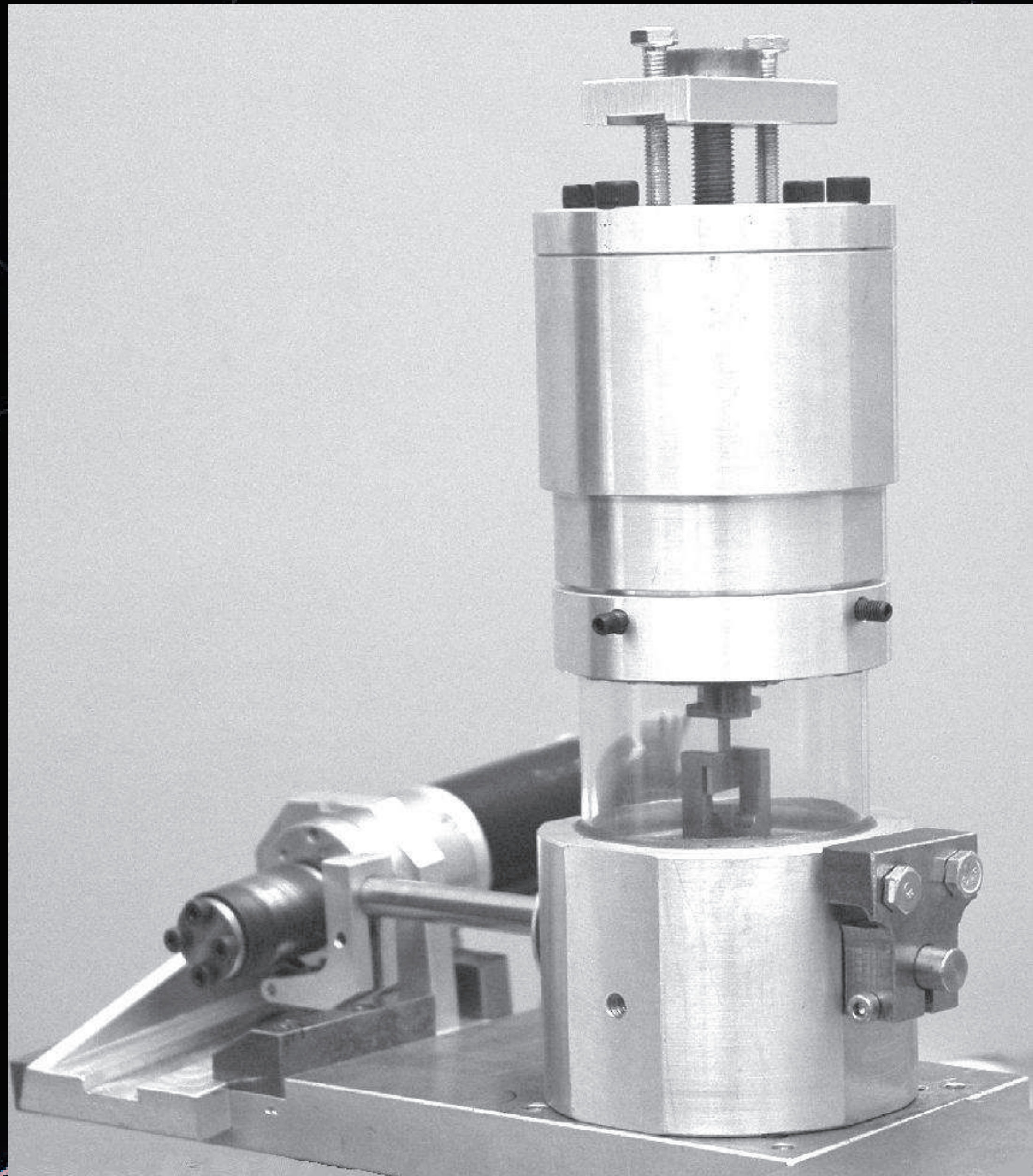


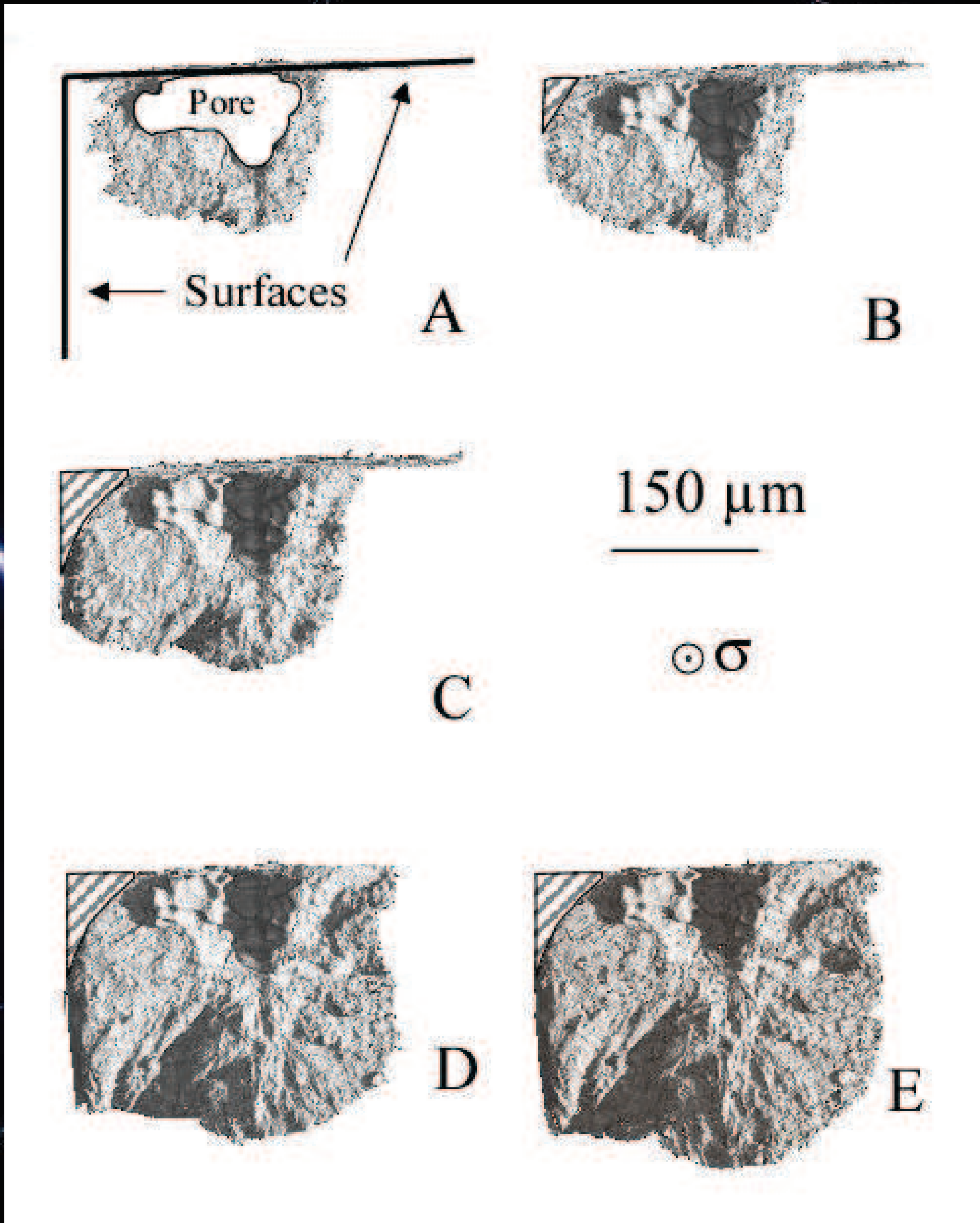


Sylvain Meille
Julien Réthoré
PhD Paul Leplay
for CREE

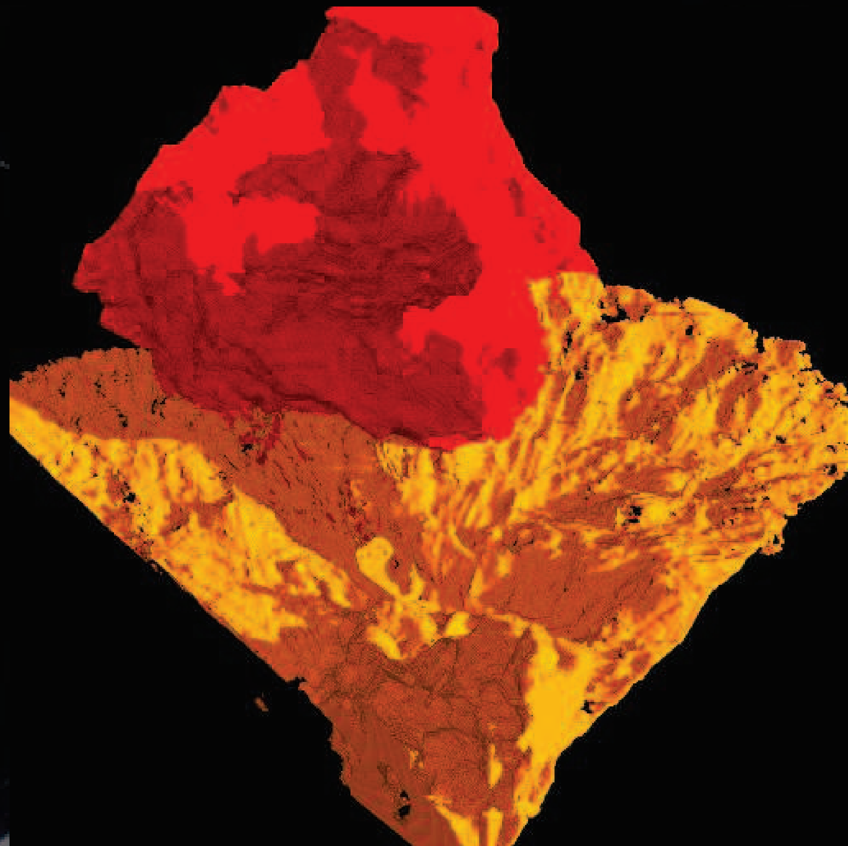
Faster (fatigue device)

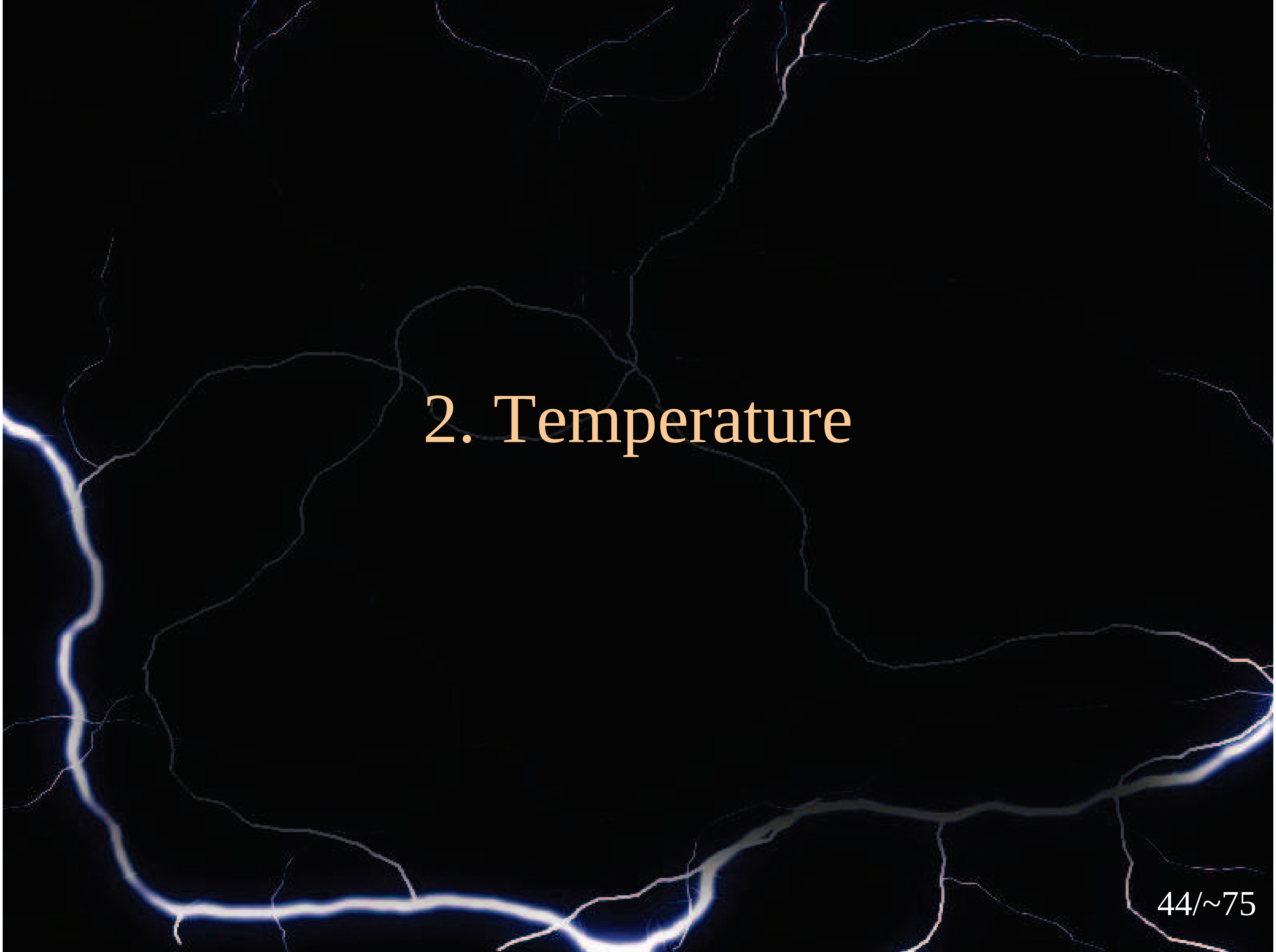
- 50 Hz
- Tension only
- We have done ex situ compression of metal hollow spheres too



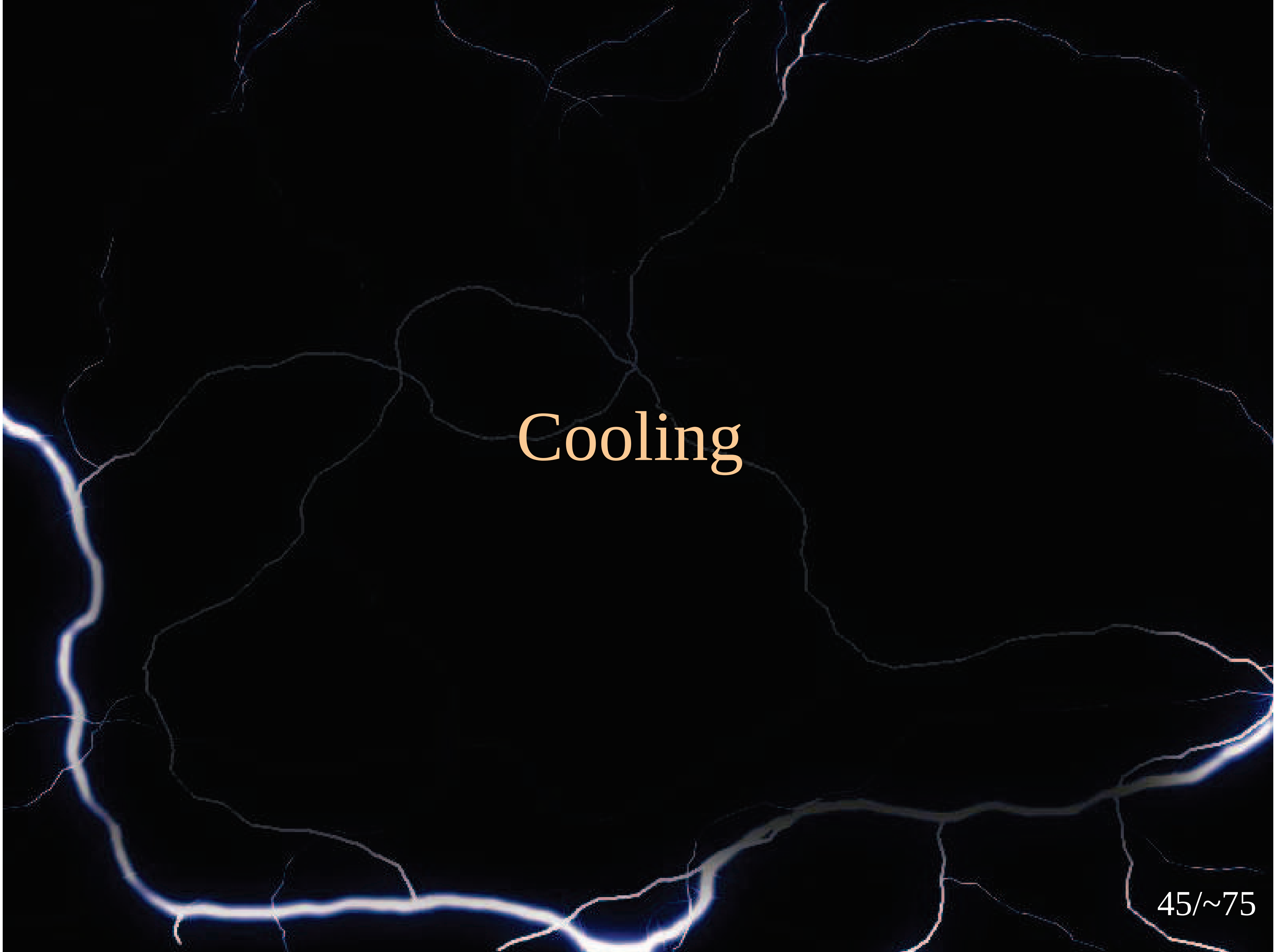


- JY Buffière
- Cracks initiate at the pore/surface intersection

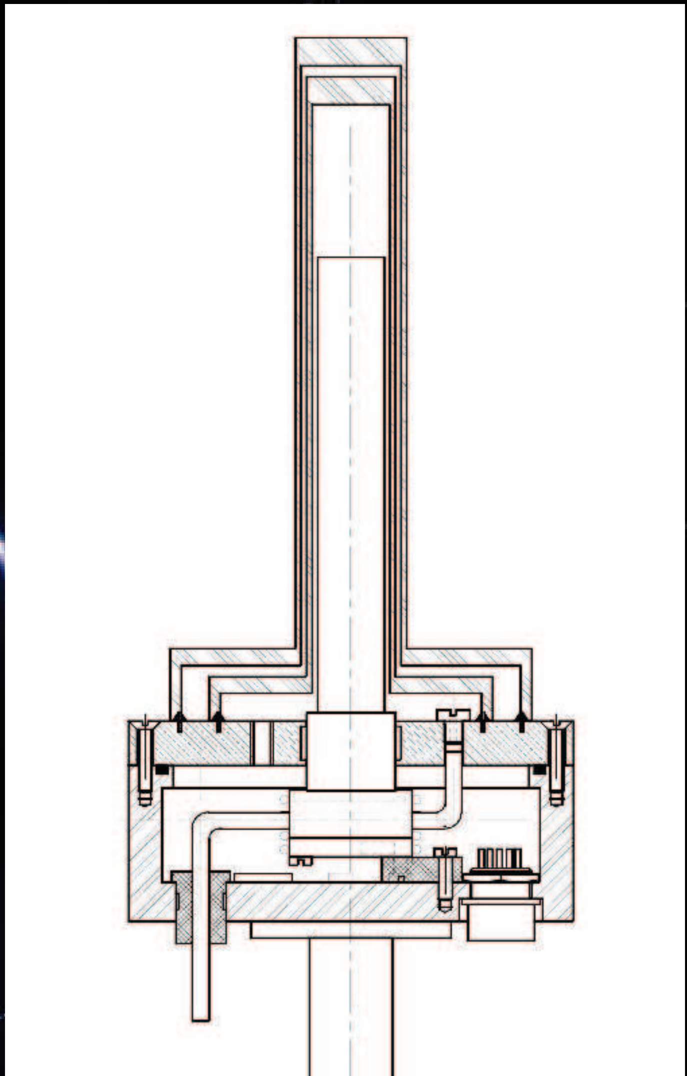




2. Temperature

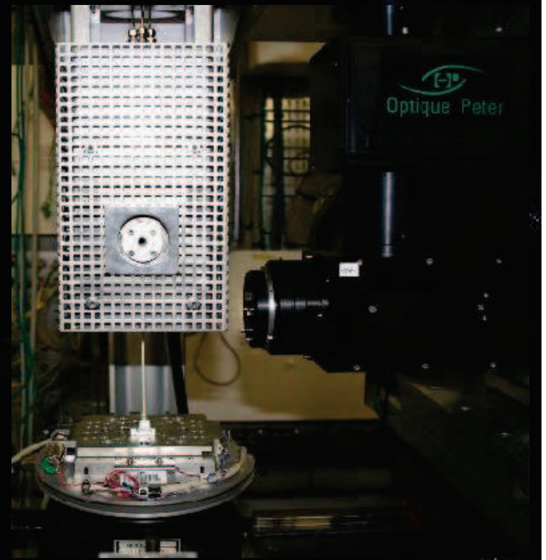

Cooling
45/~75

Cryostat INSA



Reduced diameter of the tube to fit to the lab tomograph
+ computed control of the temperature

Heating

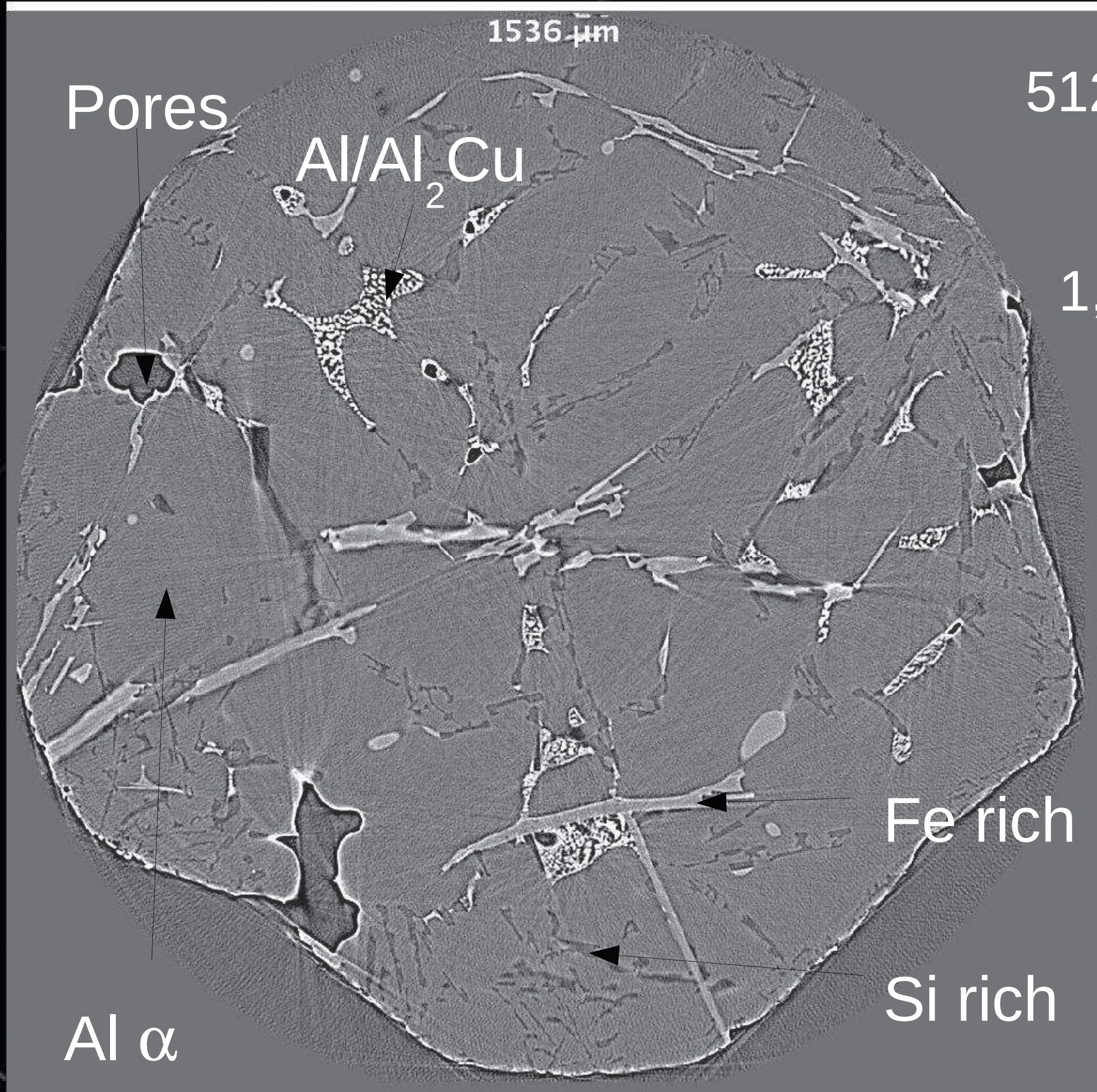


- The problem of missing views vanishes
- Different technologies for heating
  - Lamps
  - Induction
  - Standard resistors
- The sample rotates in the furnance which is fix and equipped with windows for the X rays

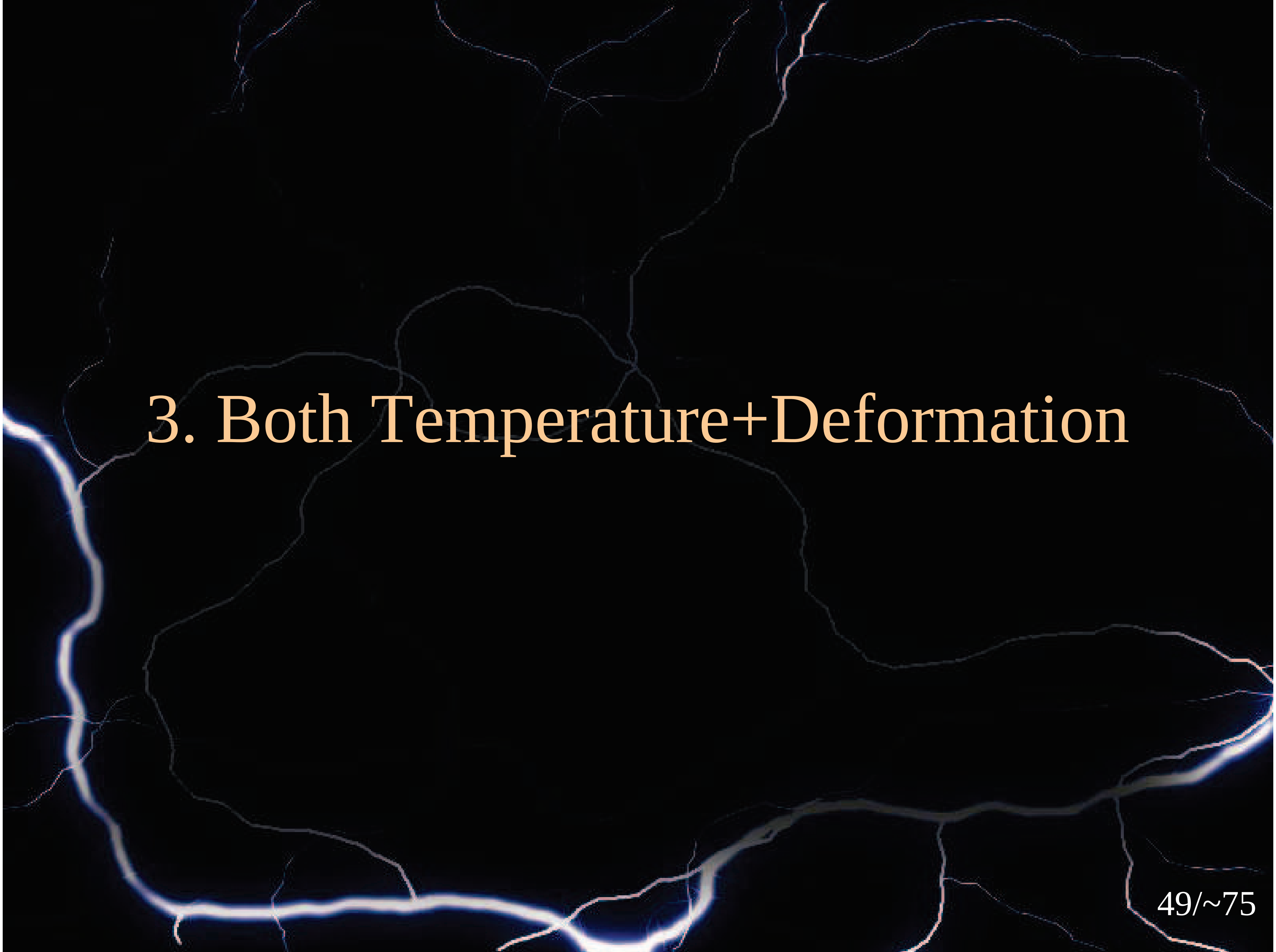
Talk by Michel Suery
In situ solidification of an Al-Cu alloy

ESRF
ID 15
Scan 15 s
res ~ 3 µm

L. Salvo (INPG)
M. Di Michiel (ESRF)

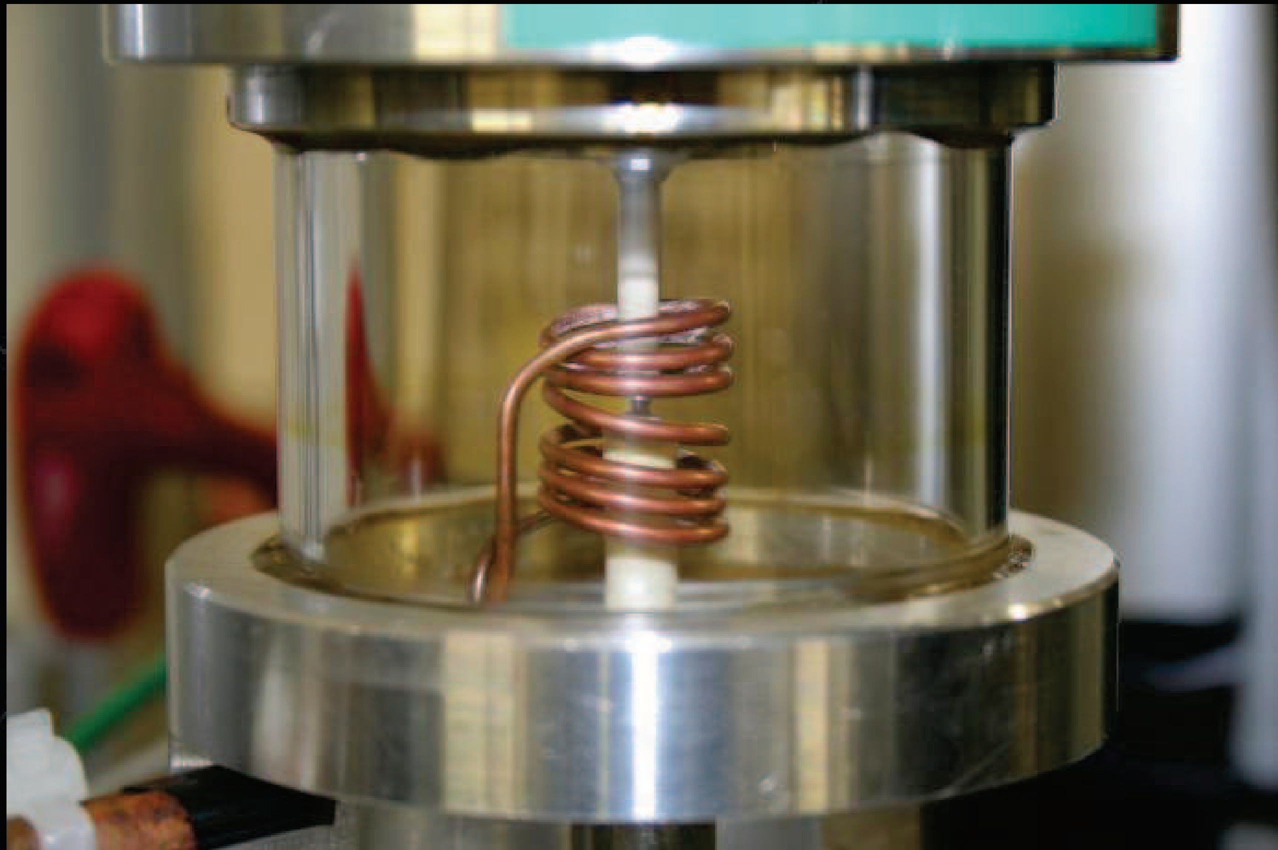


512 pixels
~
1,5 mm



3. Both Temperature+Deformation

Tension test in the semi solid state

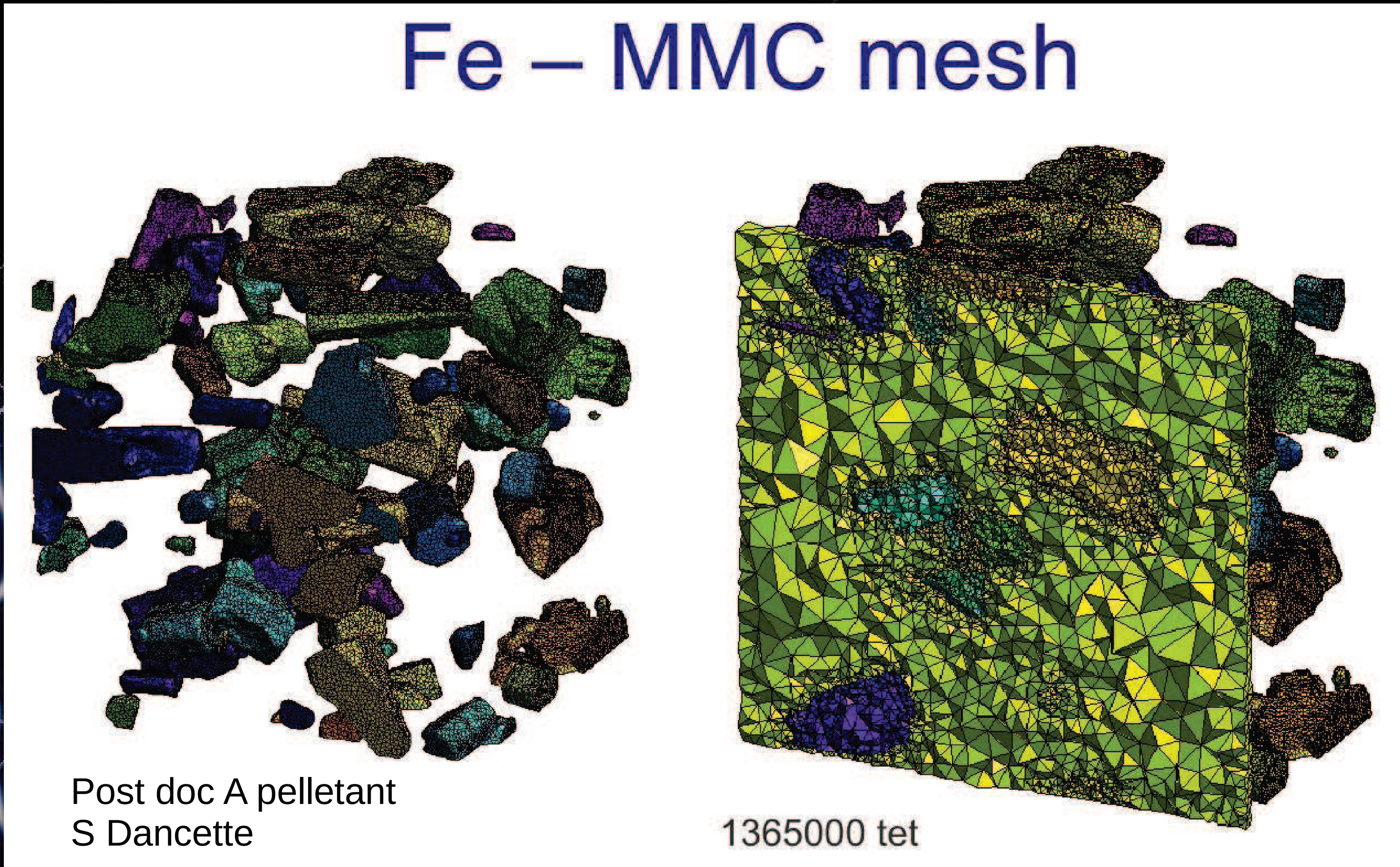


Talk by Michel Suery et al. Cavitation in liquid films

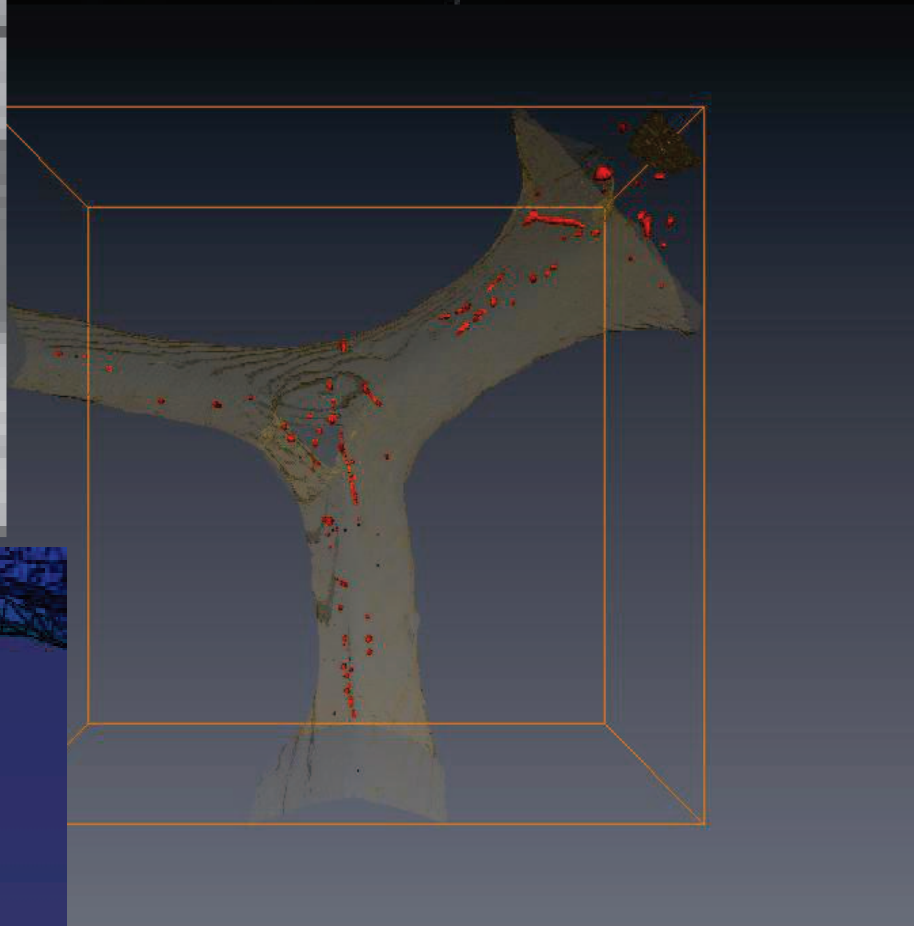
Add ons to the technique

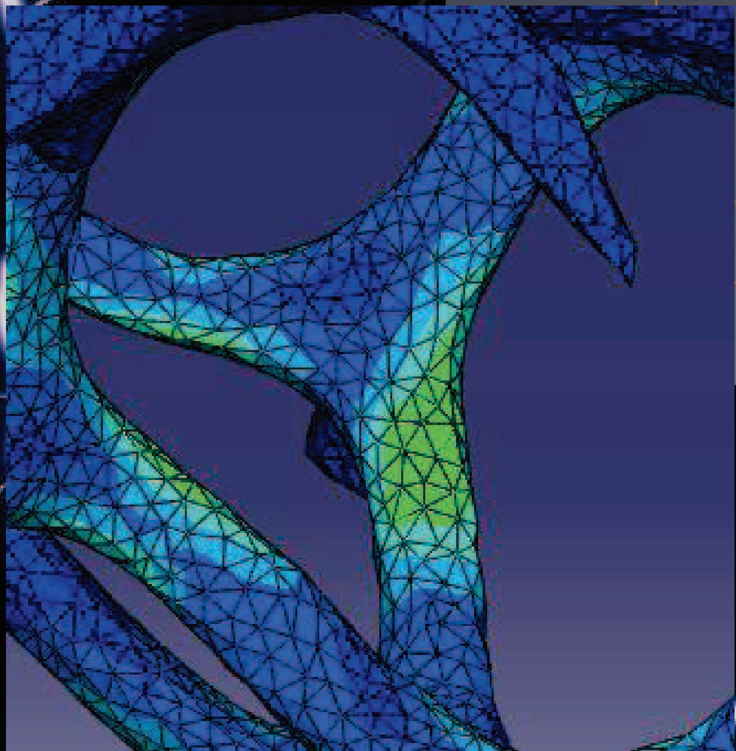
Microstructure calculation
Strain fields measurements

Fe/TiB2 composite (arcelormittal)



Local tomography for multiscale analysis

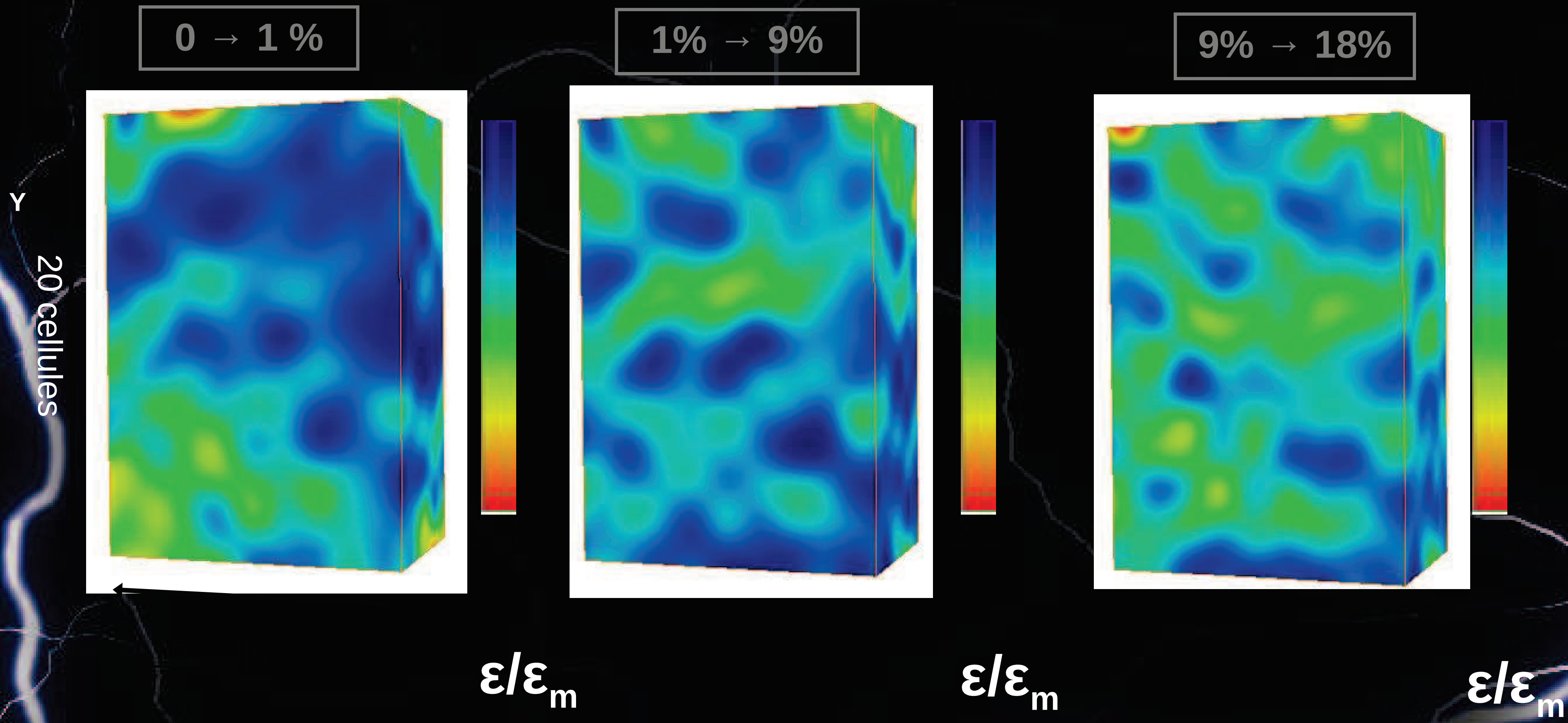




Highly loaded but not broken
No intermets
PhD Tao Zhang / Luc Salvo

A supplementary tool for the analyis : Digital volume correlation

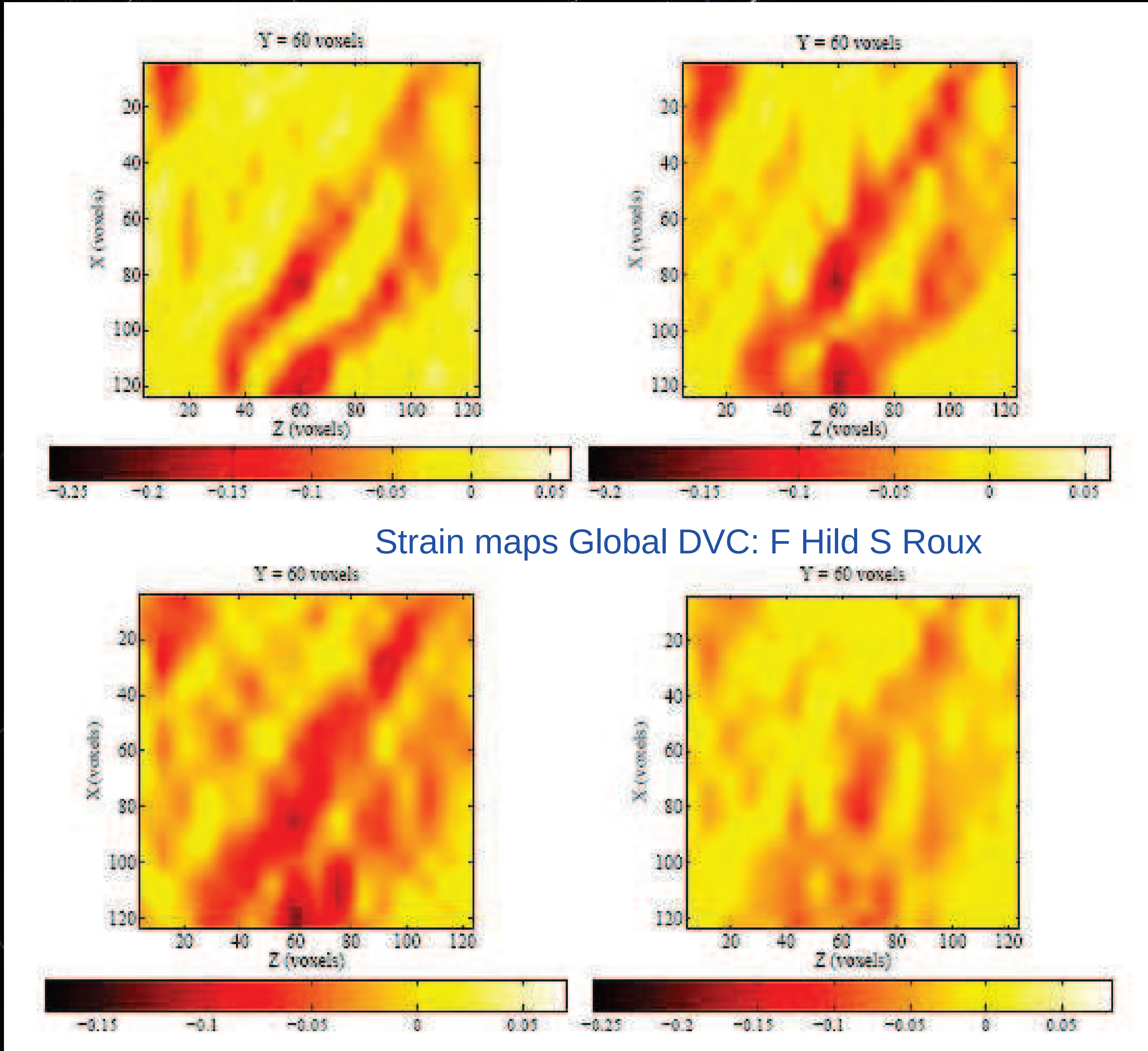
Strain maps local DVC: M Bornert



•No deformation bands for these foams (EPFL)

Incremental strain fields

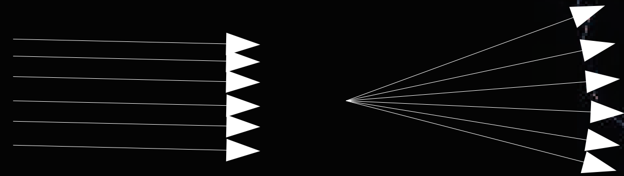
e_{zz}



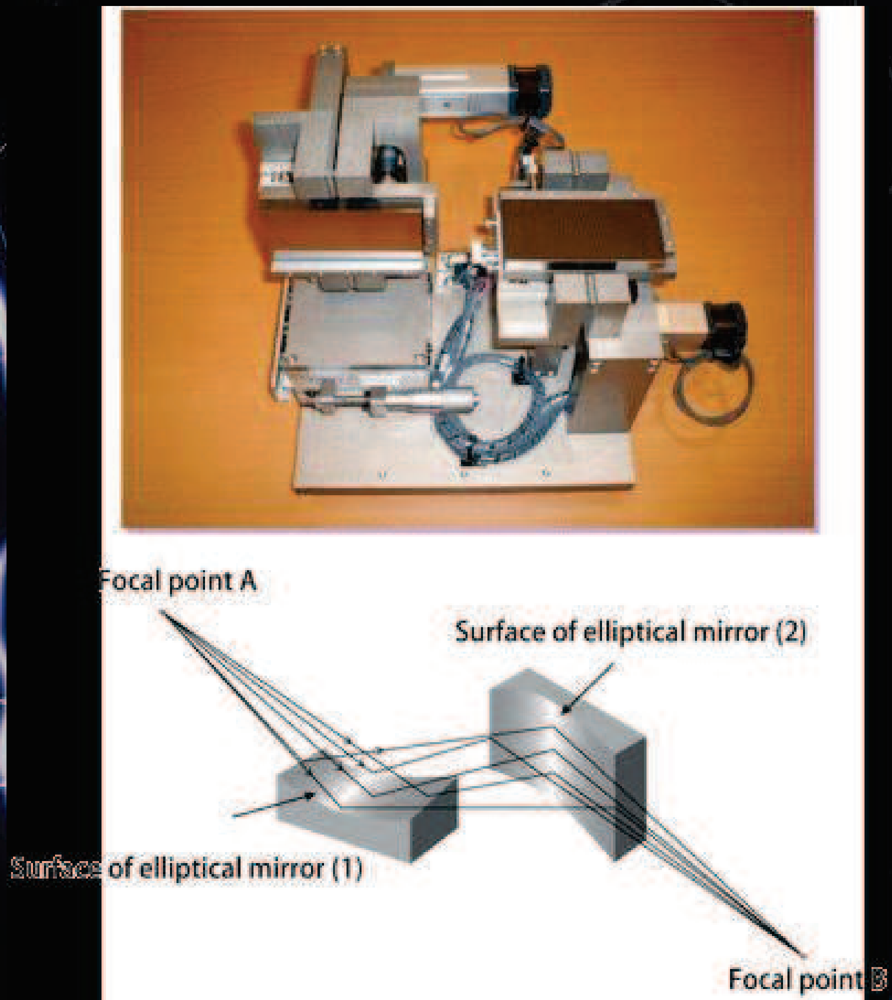
Compression axis Z

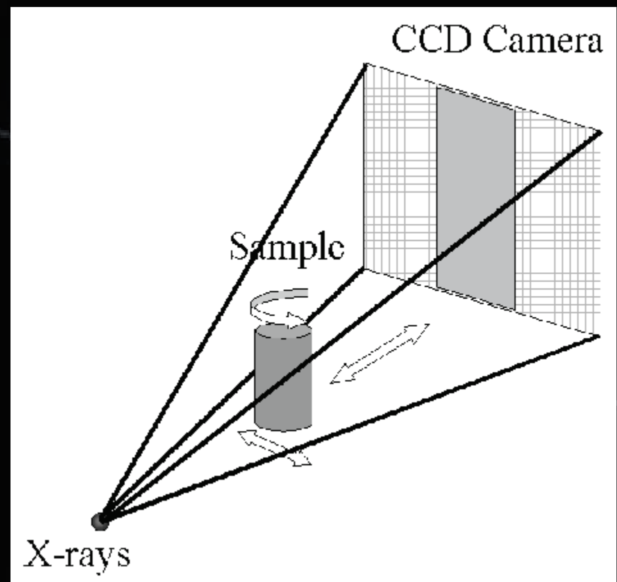
Prospects

Improve spatial resolution using KB mirrors

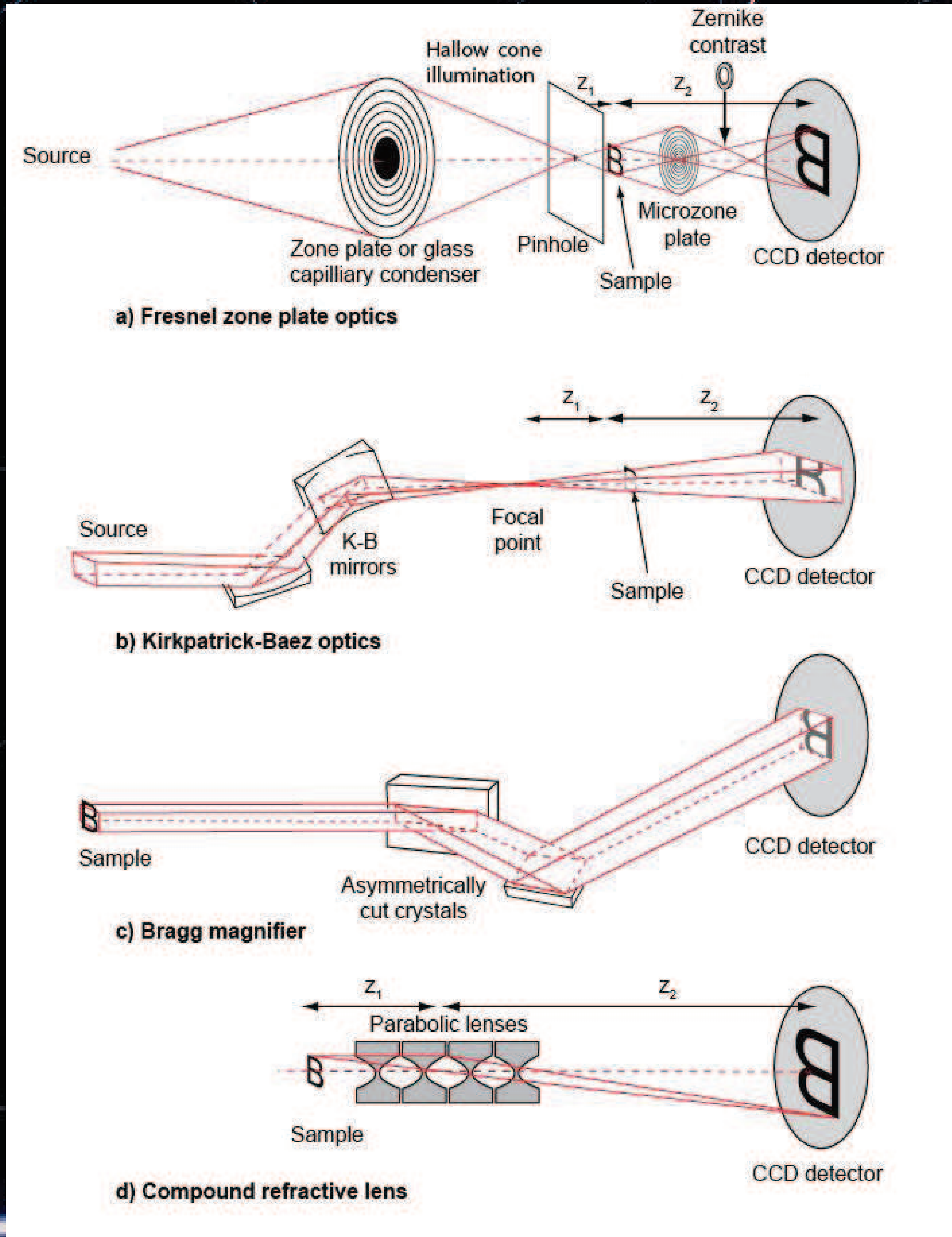


Use a conical beam on synchrotron to magnify



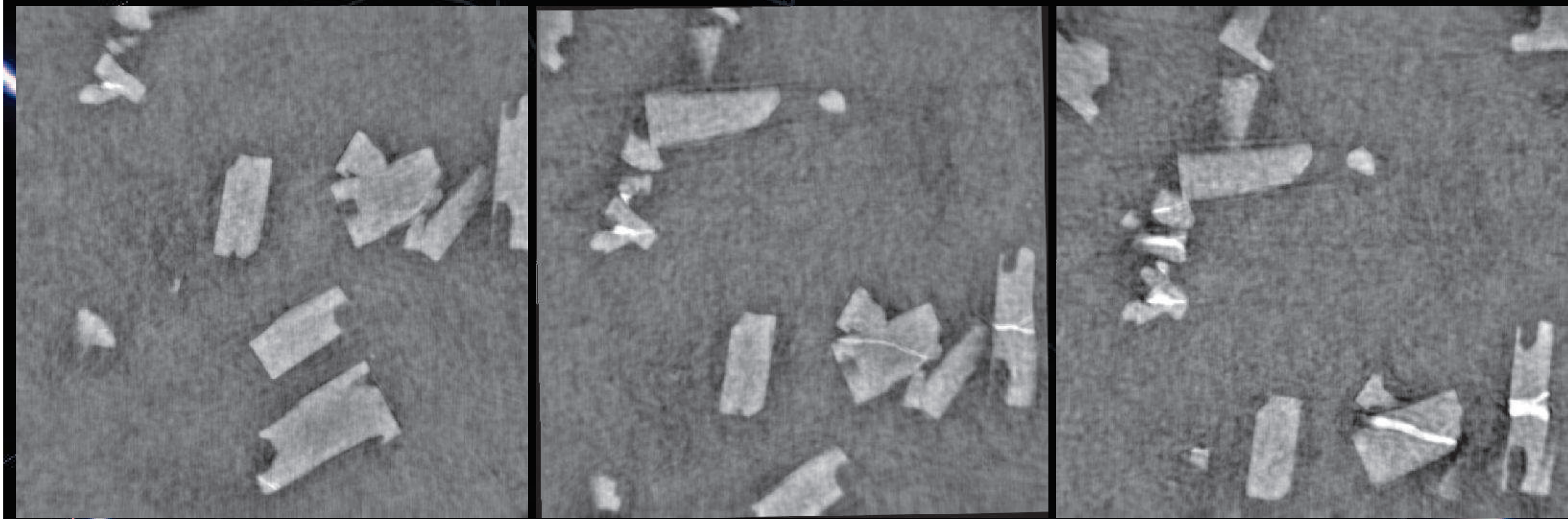


Spot size down to 20 nm


Zernike contrast
Hallow cone illumination
Source
z_1
z_2
Zone plate or glass capilliary condenser
Pinhole
Microzone plate
Sample
CCD detector
a) Fresnel zone plate optics
Source
K-B mirrors
Focal point
Sample
CCD detector
b) Kirkpatrick-Baez optics
Sample
Asymmetrically cut crystals
CCD detector
c) Bragg magnifier
Parabolic lenses
Sample
CCD detector
d) Compound refractive lens

Damage in Fe TiB_2 composites during tension (ex situ observation)

Voxel size = 50 nm !!!

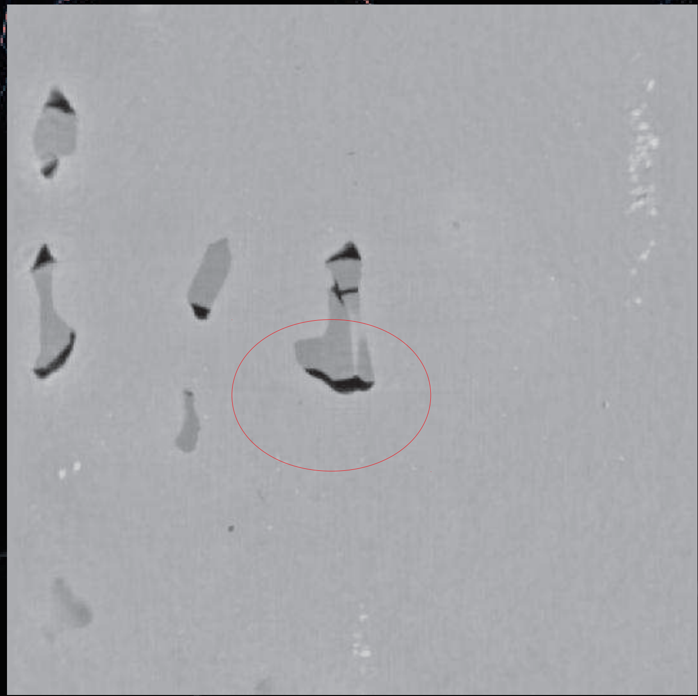


5 microns

P Cloetens and Hekki Suhonen

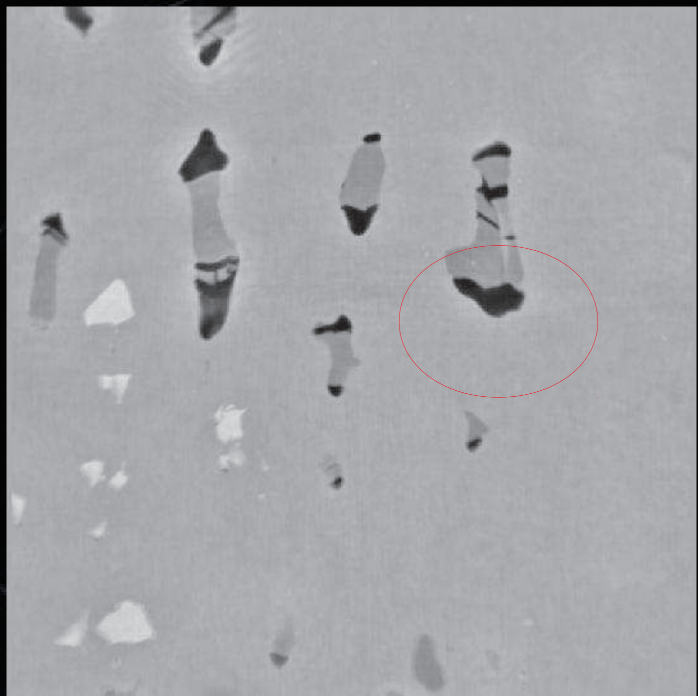
Seen from the top of the sheet

1

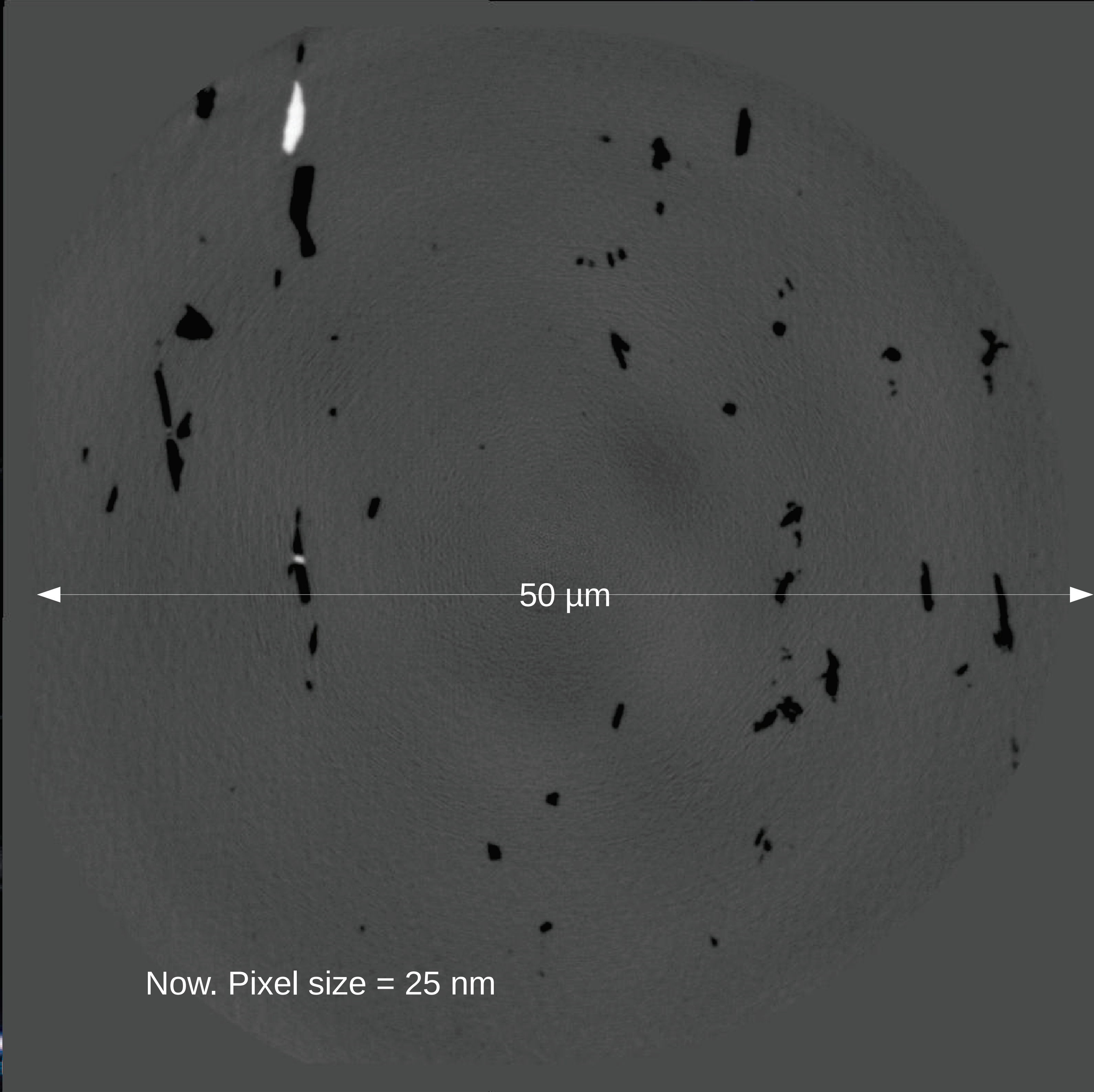


3

2



4


50 µm
Now. Pixel size = 25 nm

Temporal resolution

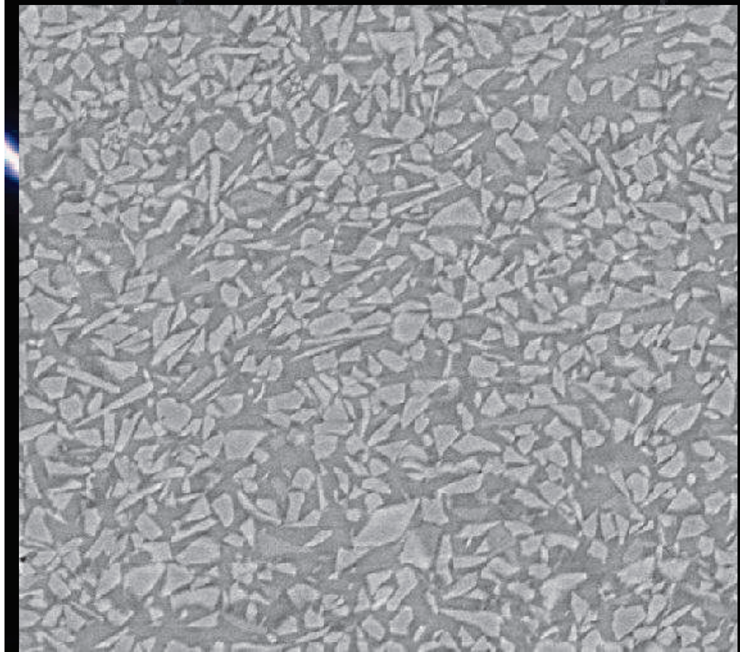
Using ID15 beamline (pink beam = high flux)

Resolution 1.5 µm

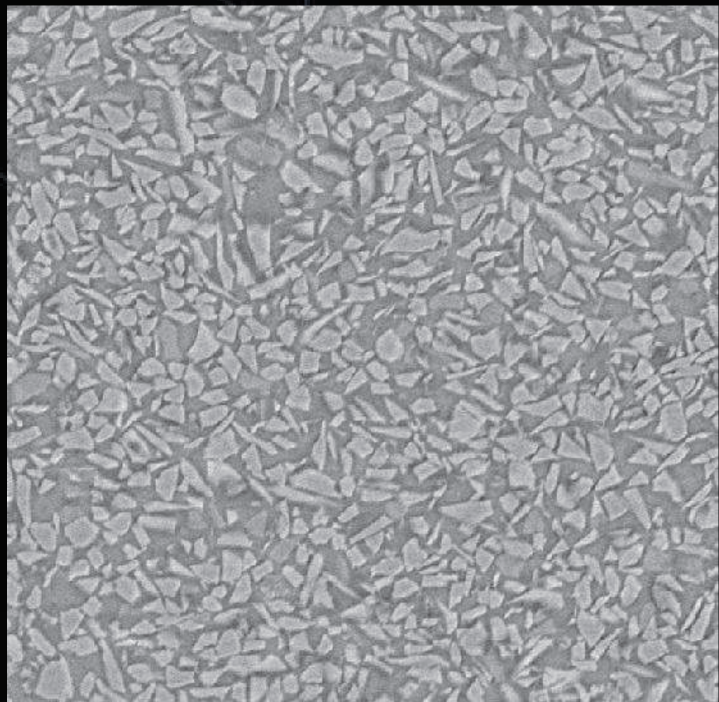
See previous movies

Drastic reduction of the exposure time !
Time for one scan :

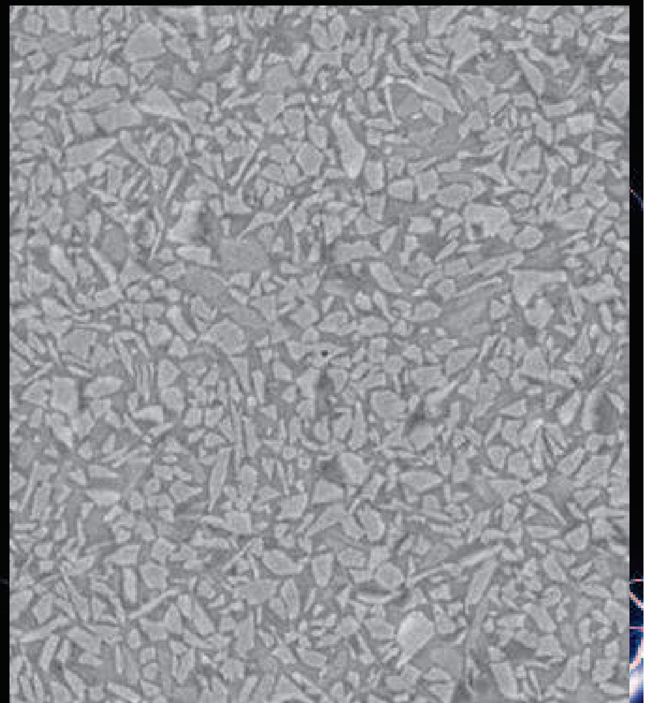
5 minutes



20 seconds

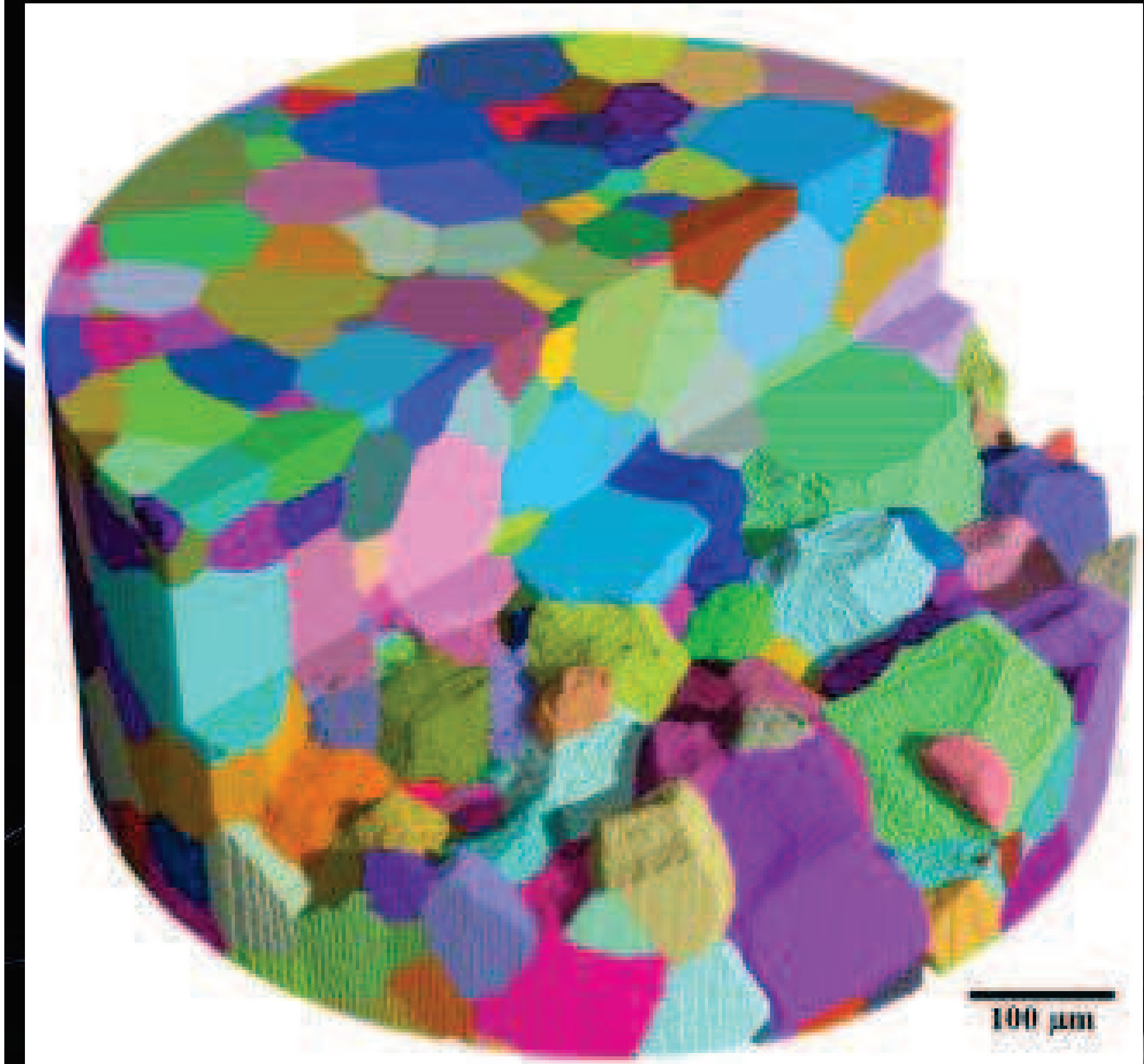


0,16 second : tensile test with $d\varepsilon/dt=10^{-3}\,s^{-1}$



500 microns

DCT on a Ti alloy. PhD M Herbig JY Buffière – W Ludwig



Metastable β-titanium alloy

'Timet®21S'

Chemical composition:

15 wt% Mo, 3 wt% Nb

1008 grains

Laminography

- For large objects (sheets)
- T Morgeneyer CDM

Challenges

- A bit of incremental development to bring the latest developments to routine
- Ready for metallurgy
- Spread the info
- Have access to beam time
- A “metallurgy” beamline
  - High energy
  - Diffraction + imaging
  - Hermès Equipex project (on ID15)

Acknowledgements

- JY Buffière, J Adrien, W Ludwig, S Dancette, A Pelletant,... MATEIS
- L Salvo, M Suery, P Lhuissier,... SIMAP
- P Tafforeau, E Boller, ... ESRF ID19
- M Dimichiel, M Schell, ESRF ID15
- P Cloetens, H Suhonen, ESRF ID22
- S Roux, F Hild, LMT