ESRF | The European Synchrotron

How micro and nano-tomography can help industrial research ?

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The European Synchrotron | ESRF

CONTENT

- Introduction
- Tomography at the ESRF
- Examples
- Conclusions and perspectives

INTRODUCTION

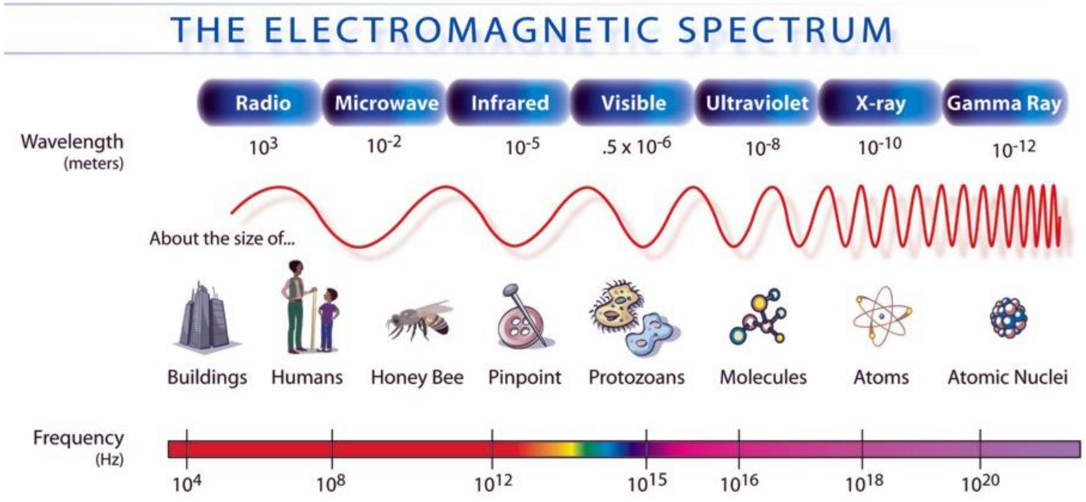
What is “tomography” ?

From ancient Greek:

- τόμος *tomos*, "slice, section"
- γράφω *graphō*, "to write"

Wikipedia definition:

- **Tomography** is imaging by sections or sectioning, through the use of any kind of penetrating wave.



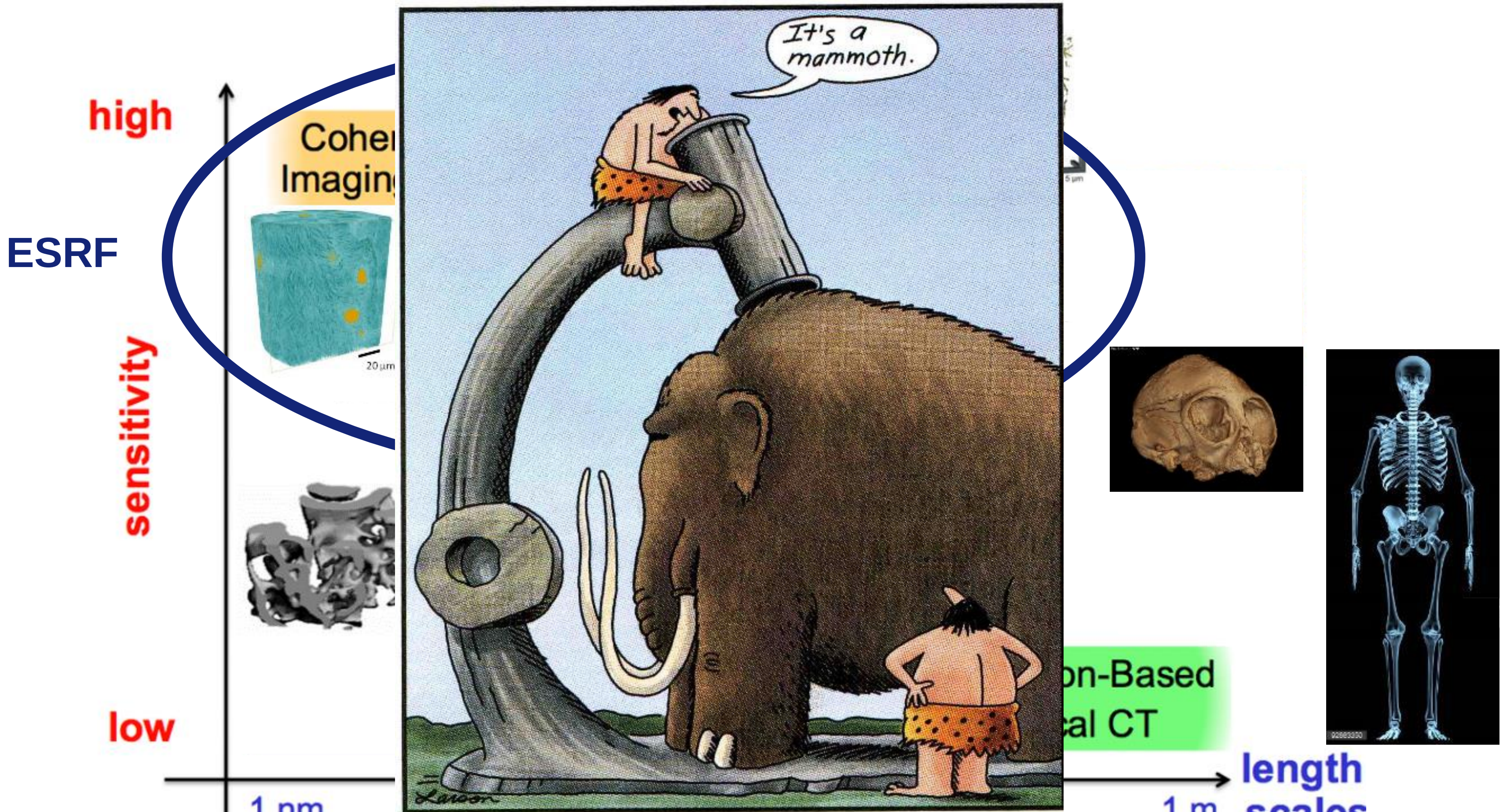
INTRODUCTION

Why are people doing tomography ?

- To see a feature in the middle of a larger sample (Non-destructive)
- To analyze a microstructure
- To characterize a sample evolution (4D in-situ)
- To understand a failure / a mechanism
- …

INTRODUCTION

From MACRO to NANO imaging



Early microscope

http://www.buzzingwire.org

Select the technique adapted to the need

Tomography at the ESRF

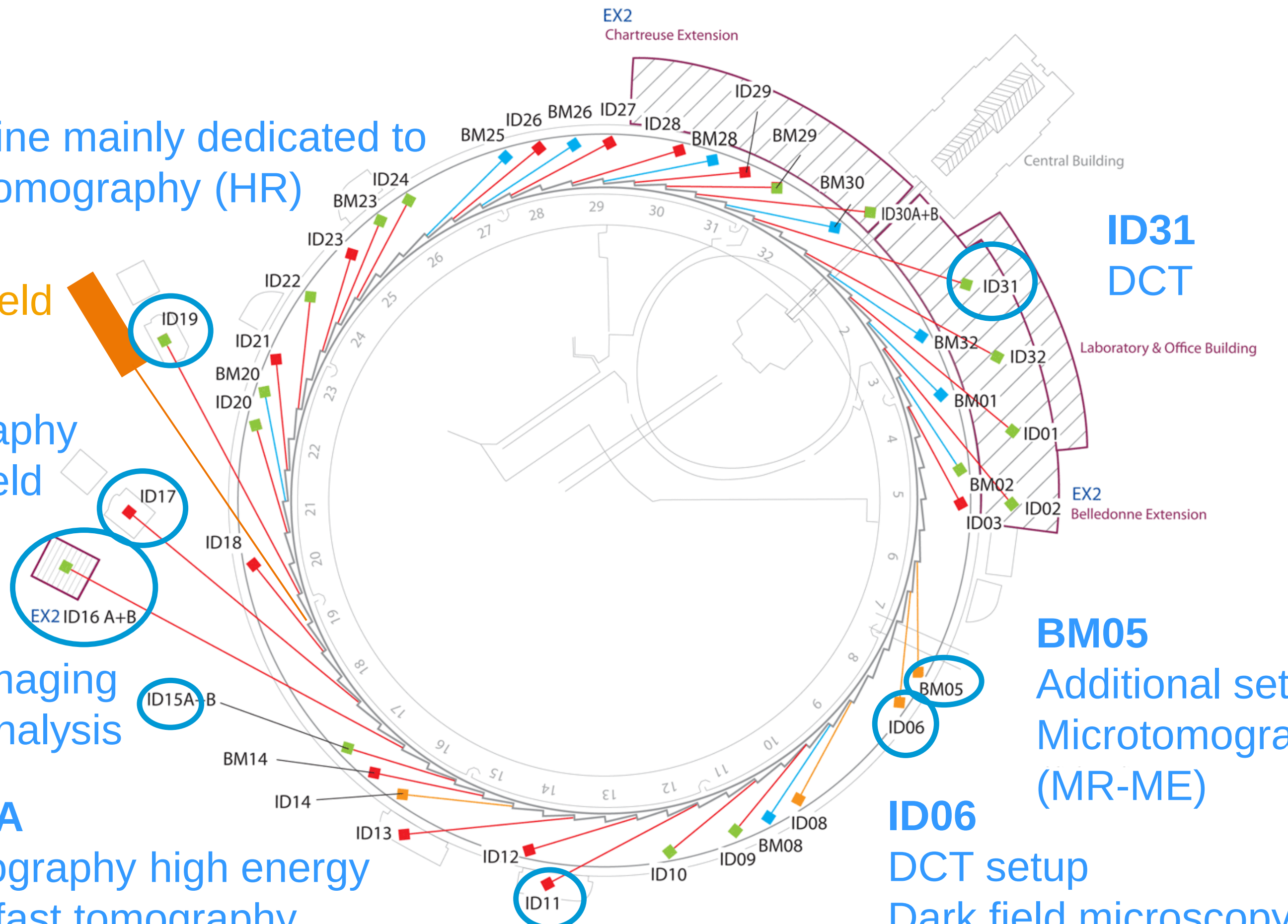
ID19
Beamline mainly dedicated to Microtomography (HR)

Future BM18 (2021)
Tomography very large field HE

ID17
Tomography Large field

ID16
Nano-imaging
Nano-analysis

ID15A
Tomography high energy
Ultrafast tomography



ID31
DCT

BM05
Additional setup for Microtomography (MR-ME)

ID06
DCT setup
Dark field microscopy

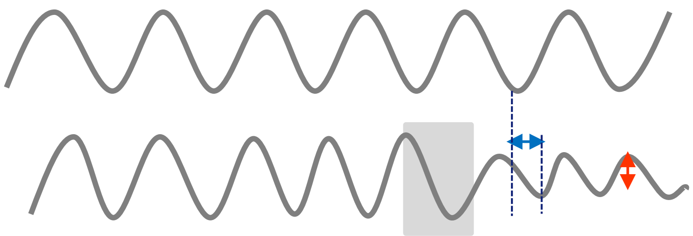
ID11
DCT setup

TOMOGRAPHY AT THE ESRF

Phase contrast imaging

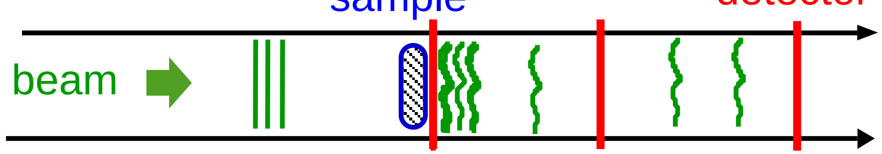
- Interaction of X-rays with matter: complex refractive index

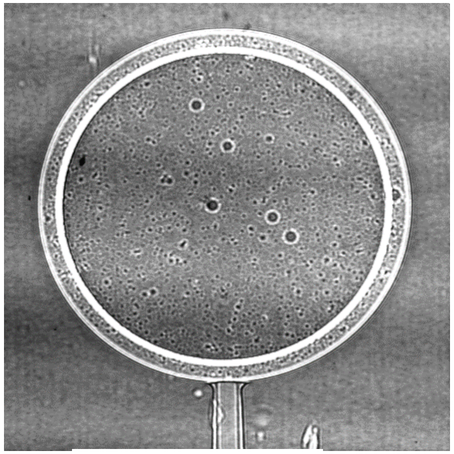
$$n = 1 - \delta + i\beta$$



Phase more interesting but not visible by direct observation

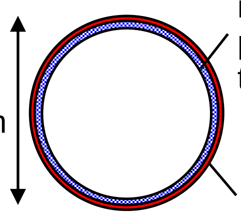
- Propagation based imaging





D_3 = 83 cm

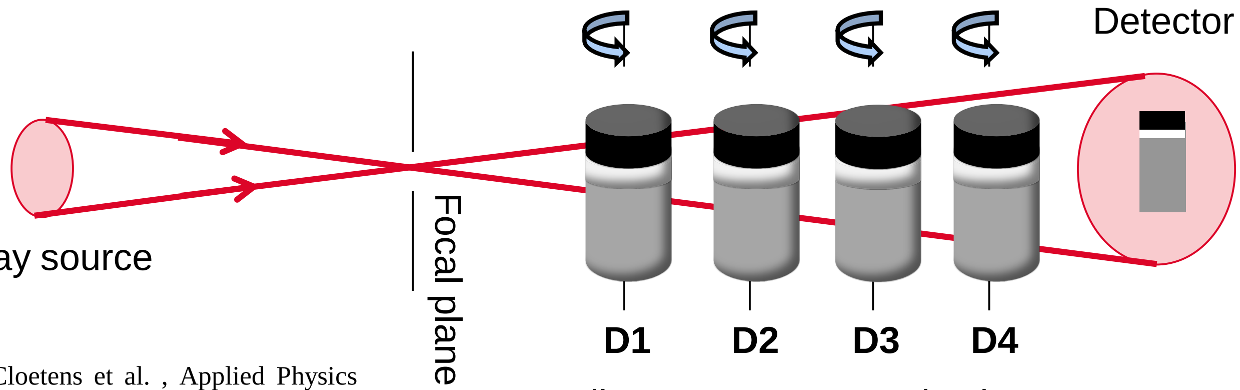
Example:
Polymer sphere with two layers



TOMOGRAPHY AT THE ESRF

Phase contrast imaging

- Nanotomography: magnified holotomography



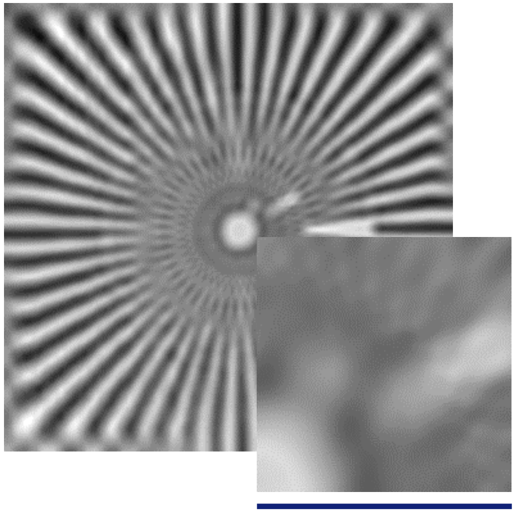
4 distances, ≈2000 projections

(1) P. Cloetens et al. , Applied Physics Letters, 75 (19) (1999) 2912-2914.

☼ 3D distribution of δ (electron density)

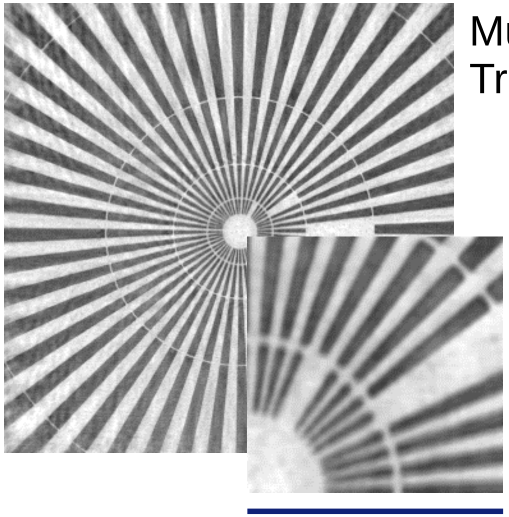
☼ Improved spatial resolution

Example:
Siemens Star pattern on ID16A



Single Distance Paganin

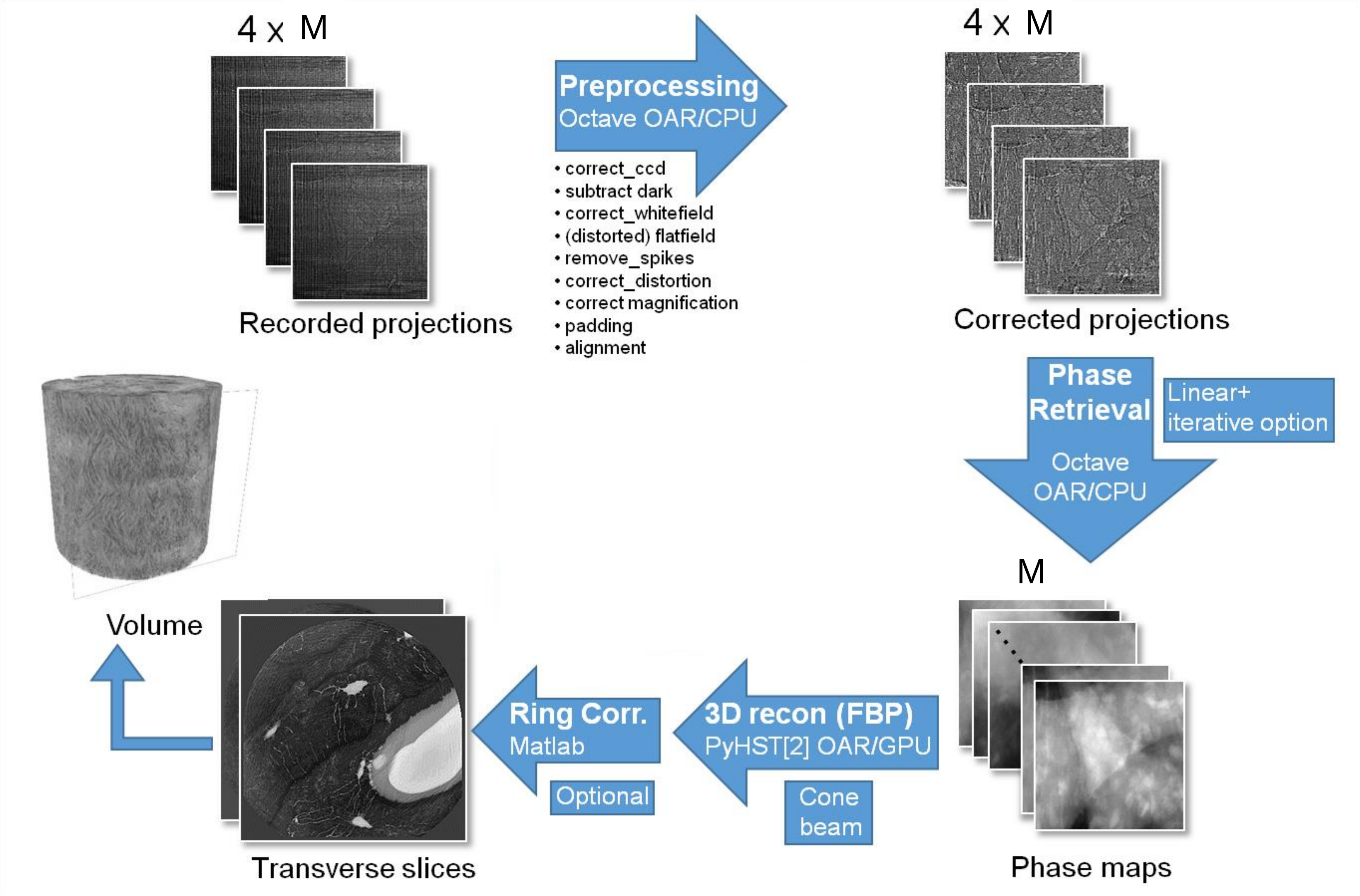
2.5 µm



Multiple distance Transfer Function

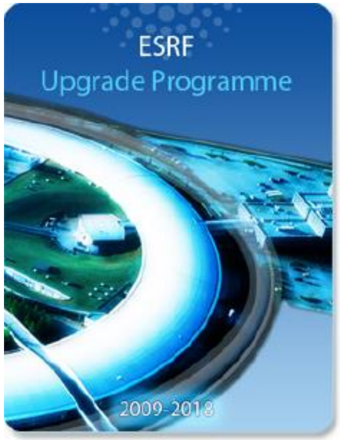
2.5 µm

Holotomography reconstruction



Specificities of tomography at the ESRF

- ☼ High energies: up to 250 keV on ID19 for MR, up to 80 keV for HR
- ☼ Phase contrast imaging: holotomography and Paganin approach
- ☼ High flux, high resolution, high Signal to Noise Ratio
- ☼ Short acquisition time: ultrafasttomo down to 50ms on ID15, 0.2s on ID19
- ☼ *In-situ* experiments (temperature, tensile, humidity,…)
- ☼ 2 end-stations dedicated to the nano-tomography



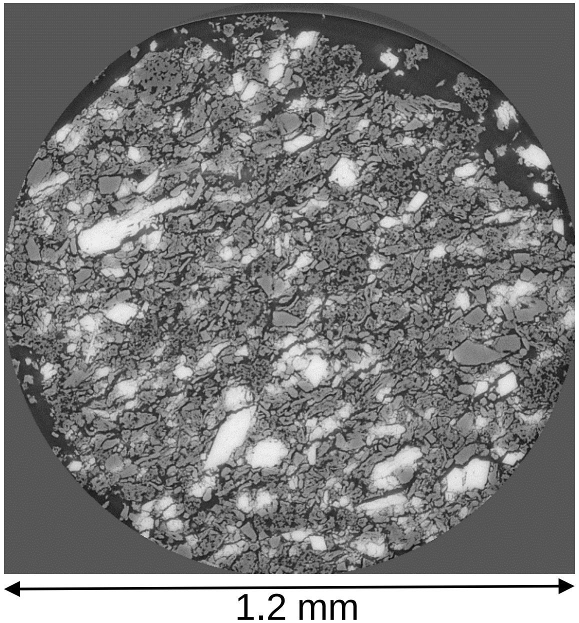
EXAMPLES

HR

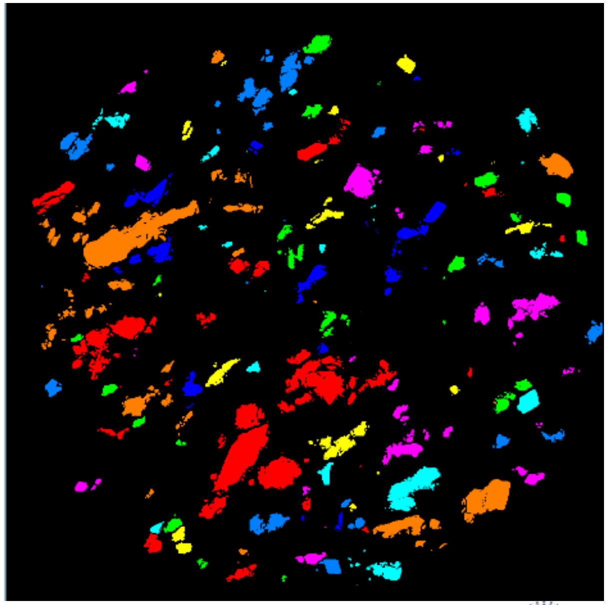
Pag

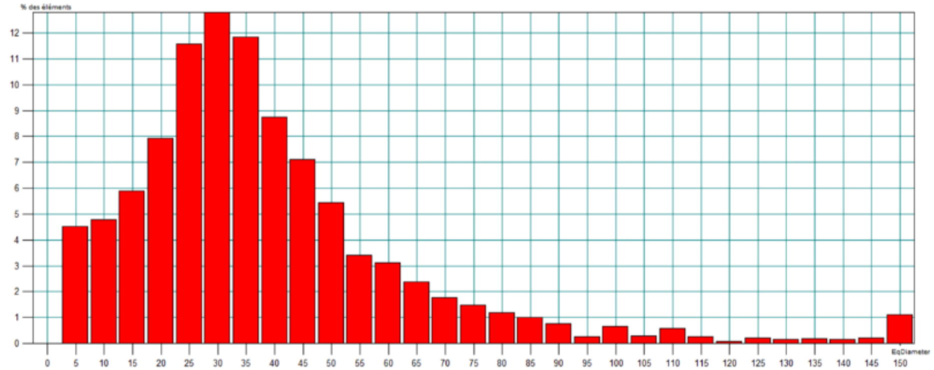
Pharma sample with 3D parameter extraction

Particle shape



Shape and size distribution of API grains (active ingredients)





EXAMPLES

MR

Pag

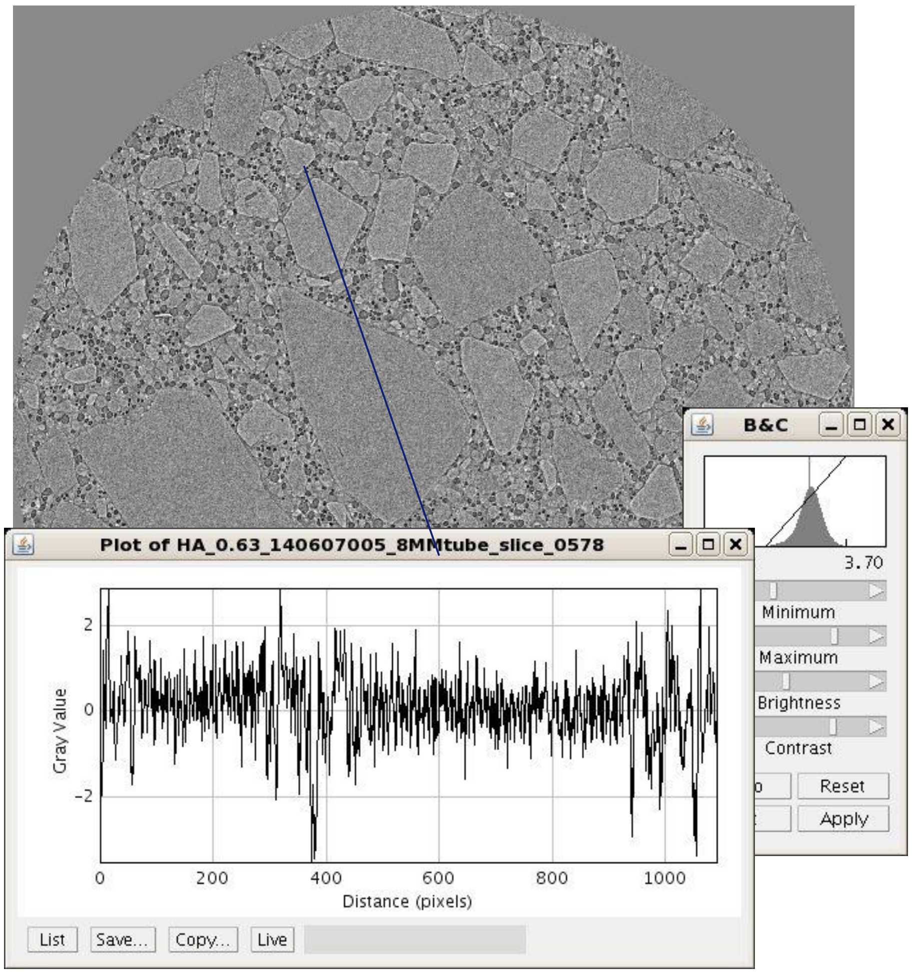
Example in Food industry: bouillon cube imaging

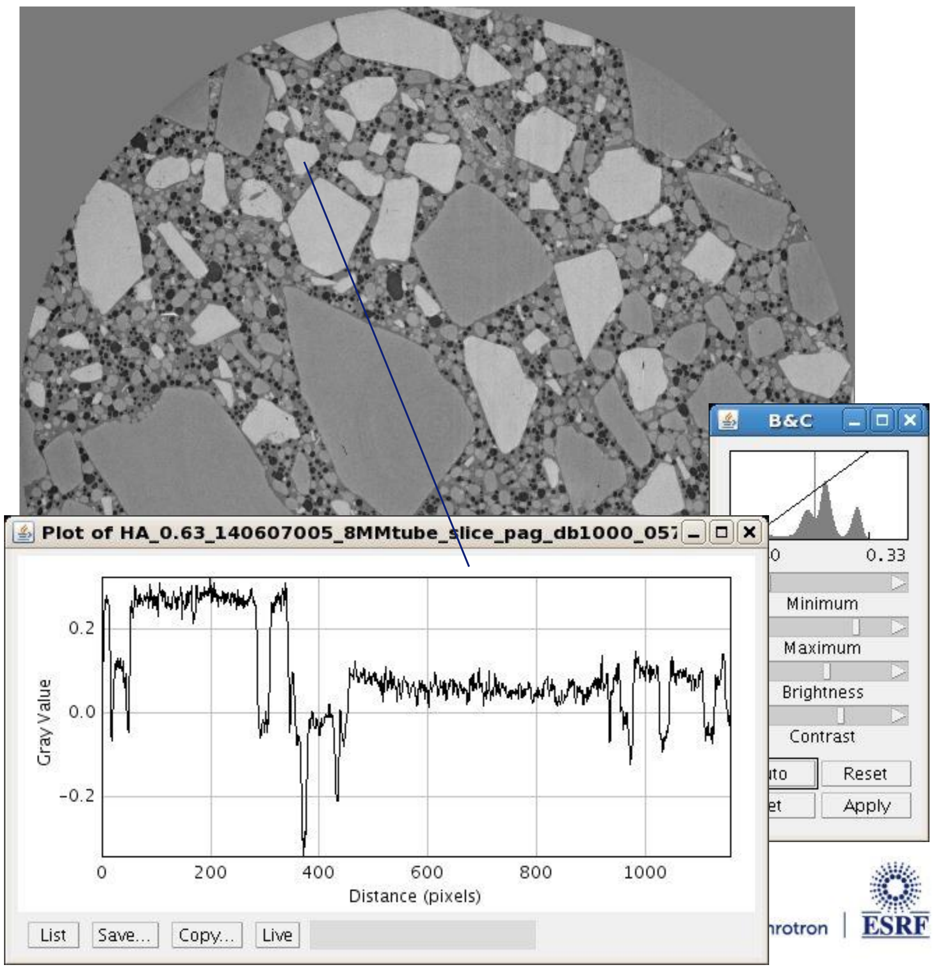
ID 19

3D distribution of the different compounds

Segmentation of Sugar or MSG, Salt, Starch, Porosities, Fat, Herbs

1.5mm





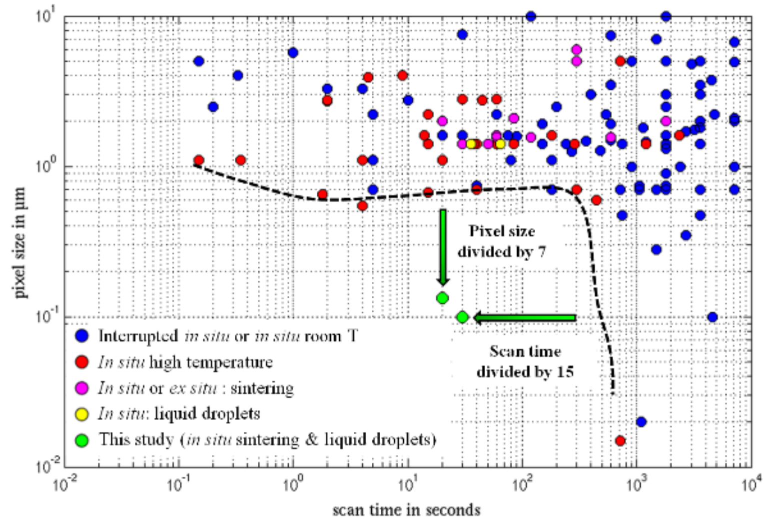
ESRF

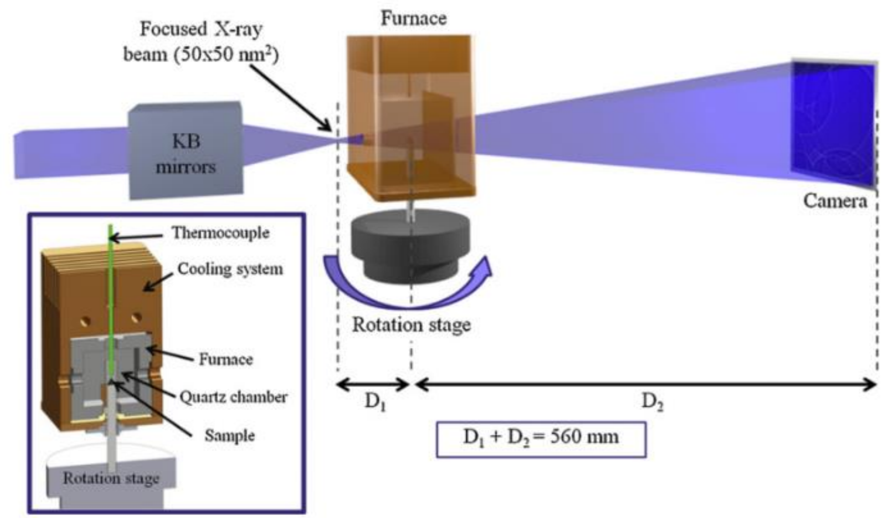
EXAMPLES

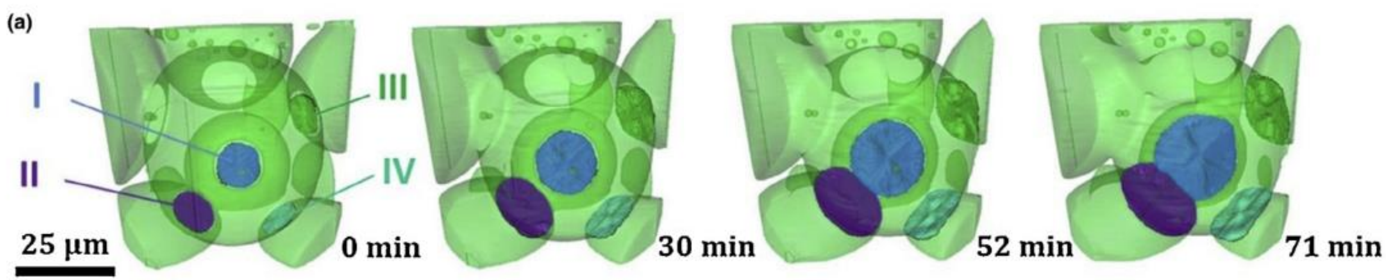
ID16B

High temperature *in-situ* X-ray nano-tomography

To understand the sintering mechanism







670°C
Voxel size: 100nm

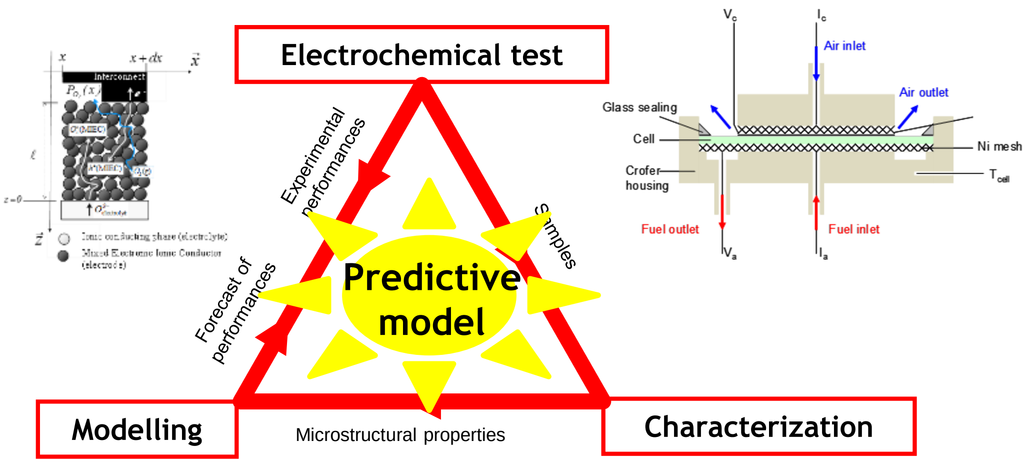
Publication:
J. Villanova et al., "Fast in situ 3D nanoimaging: a new tool for dynamic characterization in materials science." Materials Today (2017)

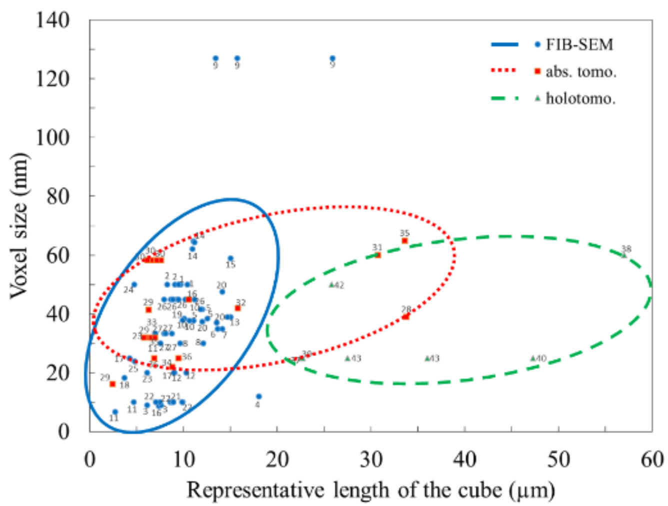
EXAMPLES

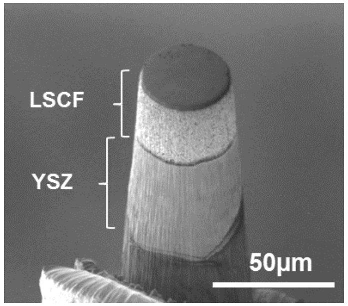
ID16A

Strongly absorbent material: Solid Oxide Fuel Cell

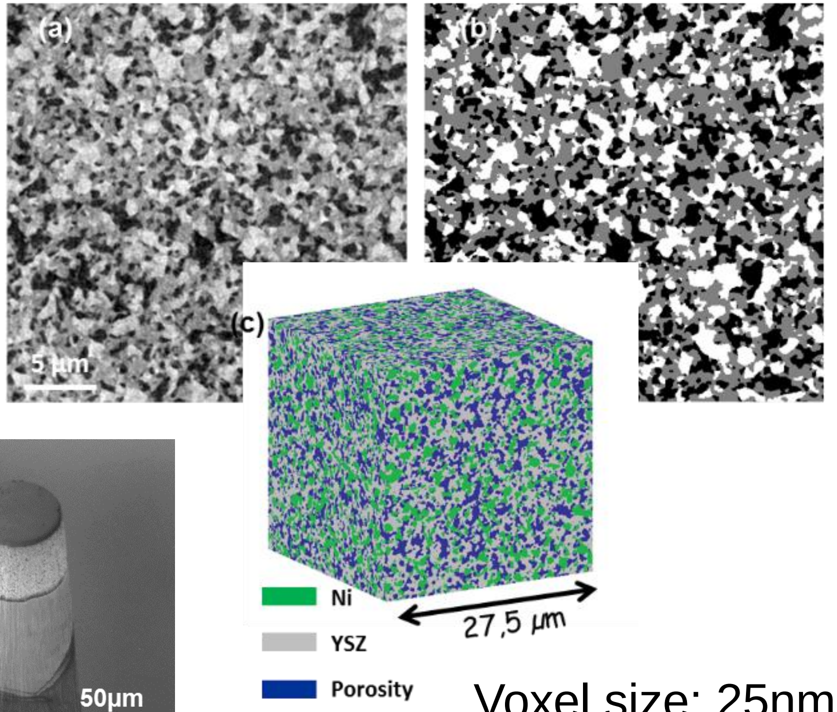
To understand the degradation over time







Contribution of Ni agglomeration on the degradation



Voxel size: 25nm

Publications:

J. Laurencin et al. , Electrochimica Acta, 174 (2015) 1299-1316.

M. Hubert et al. , Solid State Ionics, 294 (2016) 90-107.

CONCLUSION AND PERSPECTIVES

Synchrotron Radiation enlarges considerably the applicability and sensitivity of the method

- High resolution
- Short acquisition time
- Phase contrast imaging: Paganin or holotomography
- Versatility of beam energy
- Sample environment: temperature, tensile, humidity,…

Access to proprietary beamtime at the ESRF

- Quick access
- Full service provided
- Confidentiality
- Image processing in collaboration with 3D data analysis specialists

NOVITOM

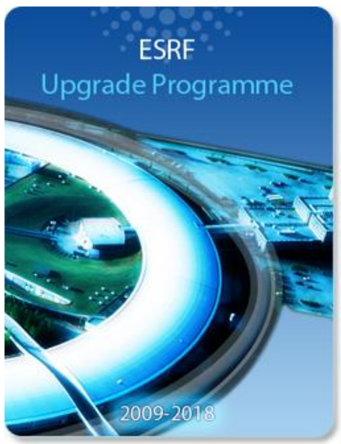
CONCLUSION AND PERSPECTIVES

Instrument in constant evolution

<u>ESRF Upgrade Phase 1</u>: 2008-2015

☼ ID19 refurbishment (transfocator, new multi-modal tomo, 2 new sample stages, …)

☼ New ID16 long beamlines dedicated to Nano-Imaging, Nano-Analysis



ESRF-EBS

<u>ESRF Upgrade Phase 2</u>: 2015-2022

☼ New source SOON !

☼ New beamline (BM18) with High energy (350 keV), large beam (300x15 mm^2), sample up to 300 kg, full automation

THANK YOU FOR YOUR ATTENTION

boller@esrf.fr

http://www.esrf.eu

KEY FIGURES

22 partner countries

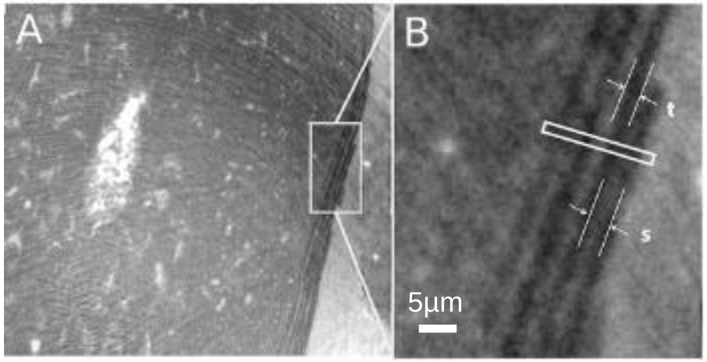
9000 scientific visits per year

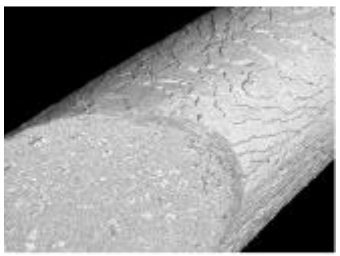
44 beamlines

2000 publications per year

EXAMPLES

Multi-technique analysis: hair

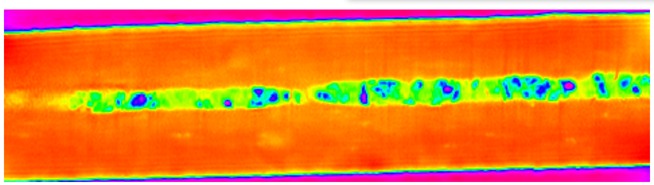




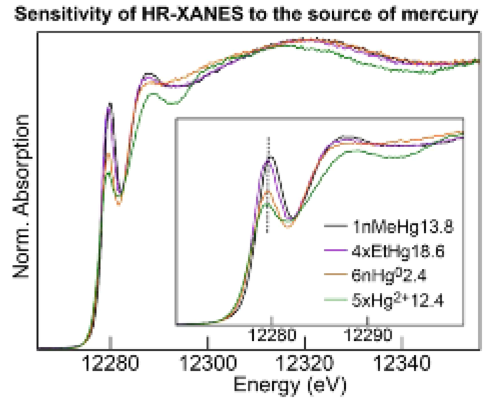
Old ID22 → **ID16B**

Structure imaging

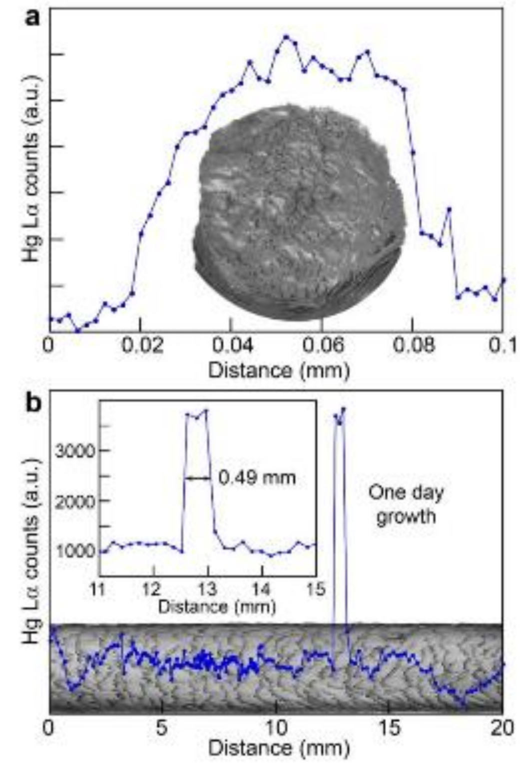
3D density mapping



ID19



XANES **ID26**



Nano-XRF **ID16B**

Origin of mercury contamination

<u>Publication</u>:

P. Cloetens et al., "Absorption and phase imaging with synchrotron radiation" Europhysics news (2001)

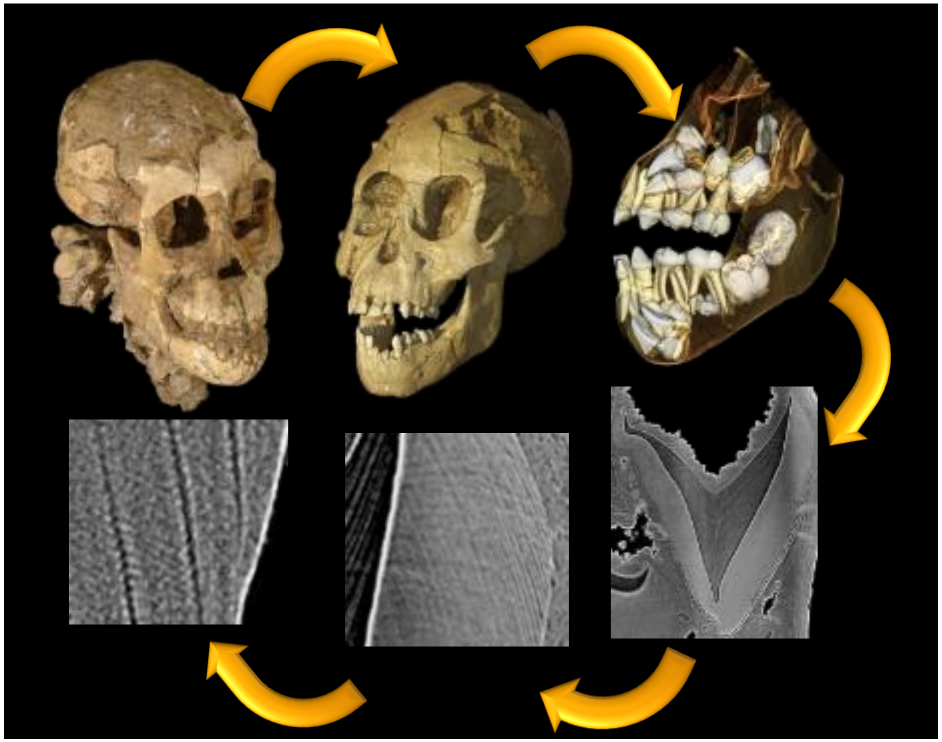
A. Manceau et al. "Chemical forms of mercury in human hair reveal sources of exposure“ Environmental science & technology (2016)

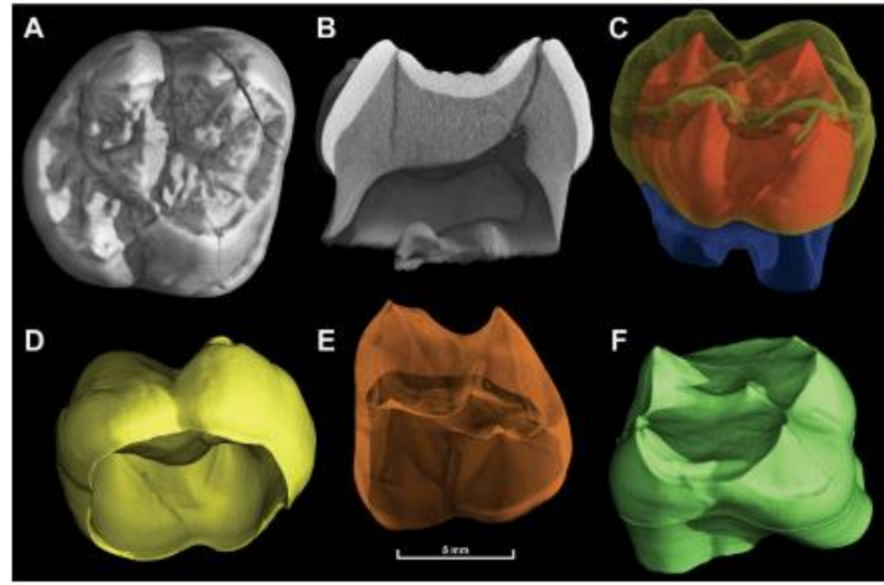
EXAMPLES

Multi-scale imaging: teeth

ID19

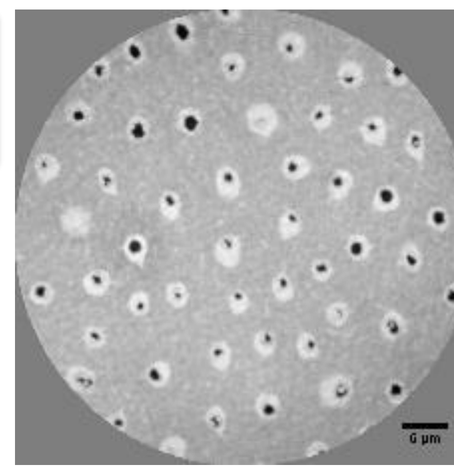
Age determination





Imaging dentine

ID16A



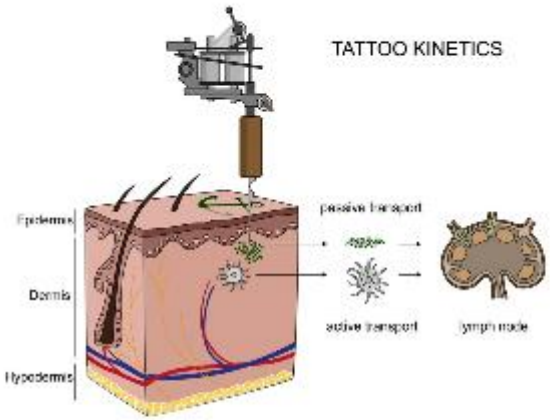
Publication:
T.M. Smith and P. Tafforeau, "New Visions of Dental Tissue Research: Tooth Development, Chemistry, and Structure" Evolutionary Anthropology (2008)
JB Forien et al. " Compressive Residual Strains in Mineral Nanoparticles as a Possible Origin of Enhanced Crack Resistance in Human Tooth Dentin " Nano Lett. (2015)

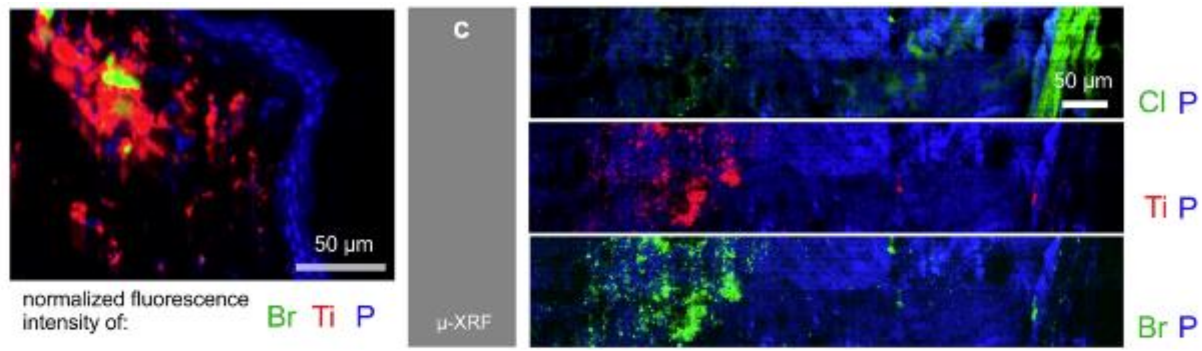
EXAMPLES

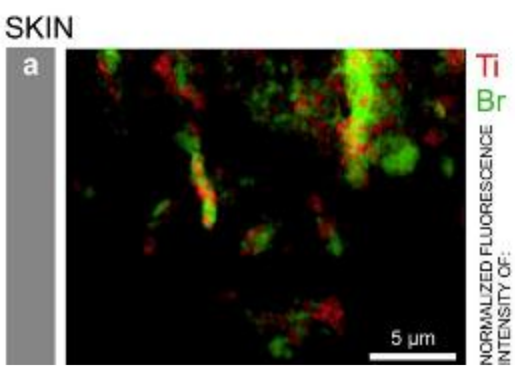
ID21 / ID16B

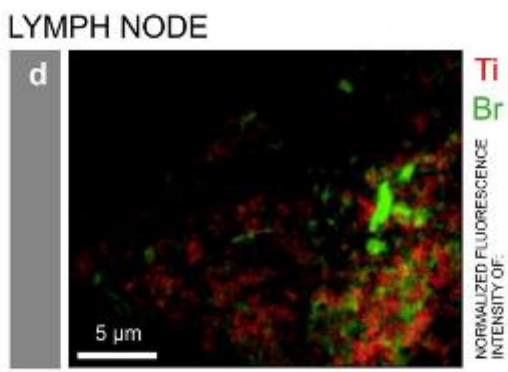
Nano-Fluorescence on biological sample

Migration of Tattoo pigment under the skin









Micro- and nano-XRF in Lymph node reveal the tattoo pigment migration and long-term deposition of toxic elements

<u>Publication</u>:
I. Schreiver et al., "Synchrotron-based ν-XRF mapping and µ-FTIR microscopy enable to look into the fate and effects of tattoo pigments in human skin" Scientific Reports (2017)

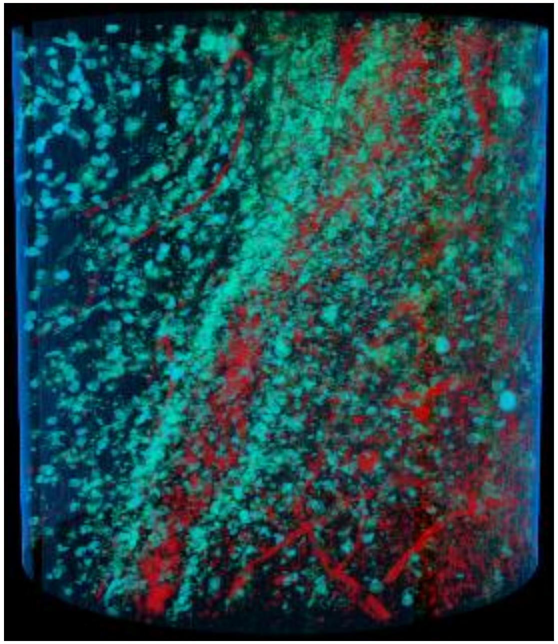
EXAMPLES

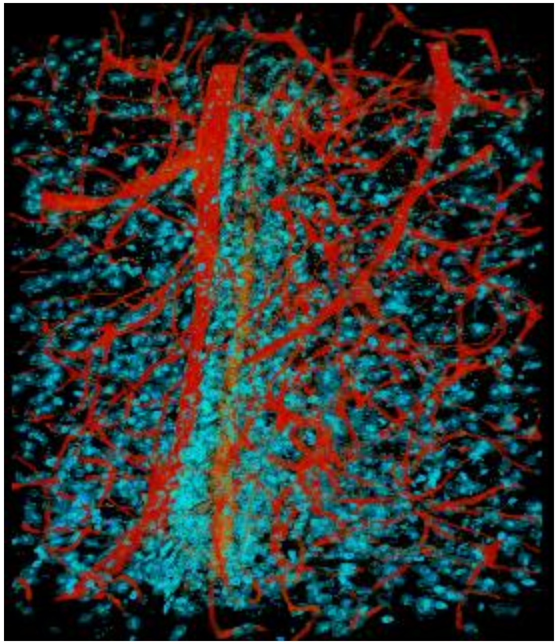
Nanotomography on biological sample

Efficiency of an advanced therapy

ID17 / ID16A

(a) (b)





Magnified phase nano-tomography applied to a mice model of multiple sclerosis: 3D rendering of the vascularisation (**red**) and cell population (**blue**) in the spinal cord of diseased mice without treatment (a) and treated with mesenchymal stem cells (b)

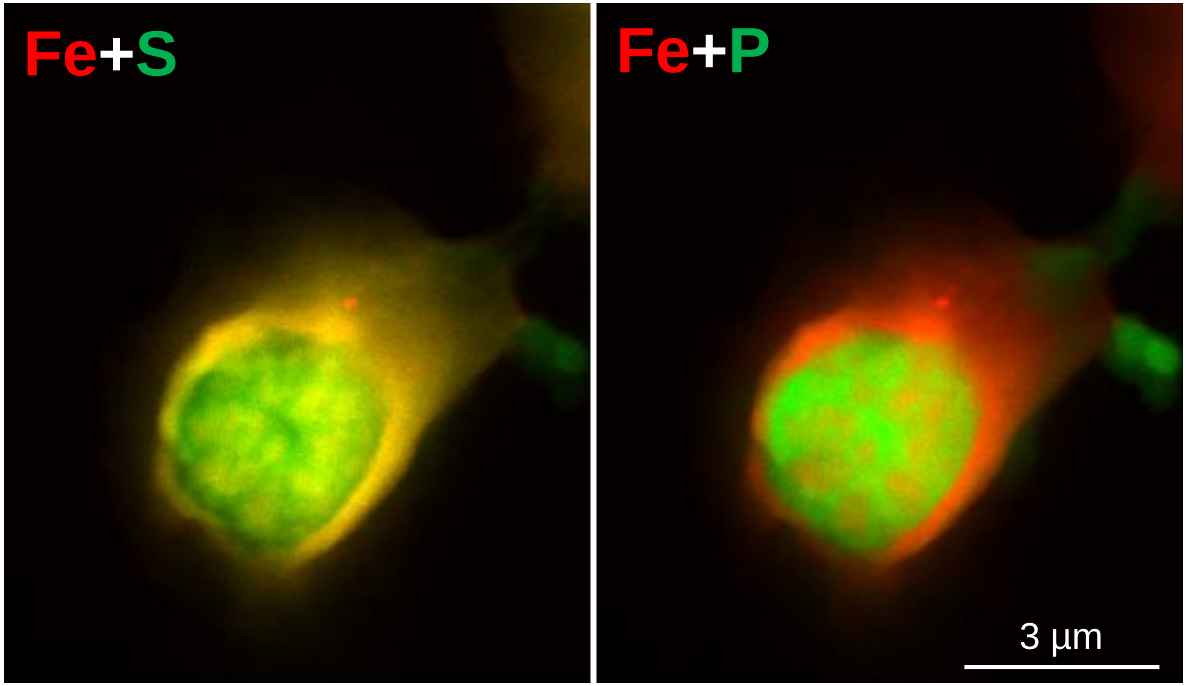
Publication:
A. Cedola et al., "X-Ray Phase Contrast Tomography Reveals Early Vascular Alterations and Neuronal Loss in a Multiple Sclerosis Model " Scientific Reports (2017)

EXAMPLES

Nano-Fluorescence on biological sample

Distribution of S, P and Fe within cell nuclei

ID16A



Fe is co-localized with S, less with P (chromatin)

Images suggest Fe is incorporated in nuclear membrane regions and FeS enzymes

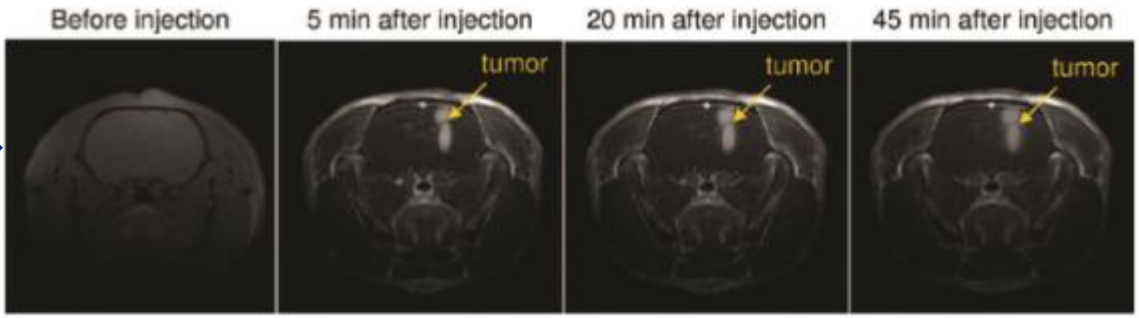
Human lymphocyte cell nucleus
Step size 30nm
E = 17keV

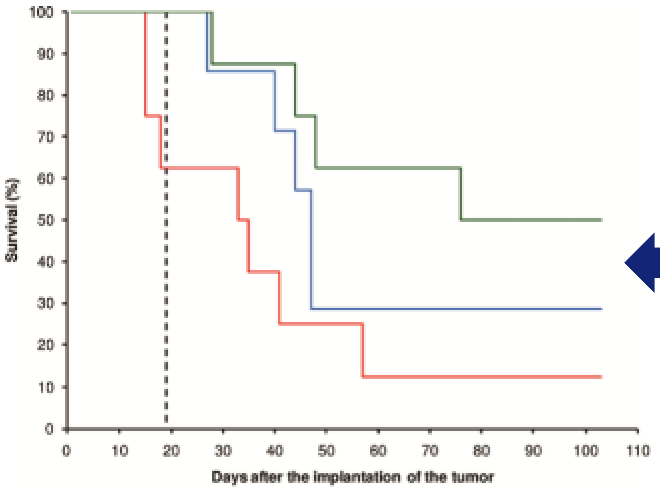
Publication:
I. Robinson et al., "Nuclear incorporation of iron during the eukaryotic cell cycle" J. Synchrotron Rad. 23 (2016)

EXAMPLES

Medical beamline: Microbeam Radiation Therapy (MRT)

T1-weighted images of the brain of a 9LGS-bearing rat before and 5, 20, and 45 min after intravenous injection of GBNs





9LGS bearing rats, without treatment (black dashed curve, only treated by MRT (blue curve) and treated by MRT 5 minutes (red curve) and 20 minutes (green curve) after NPs intravenous injection.

In-vivo radiosensitization and novel microbeam irradiation regimens

Publication:

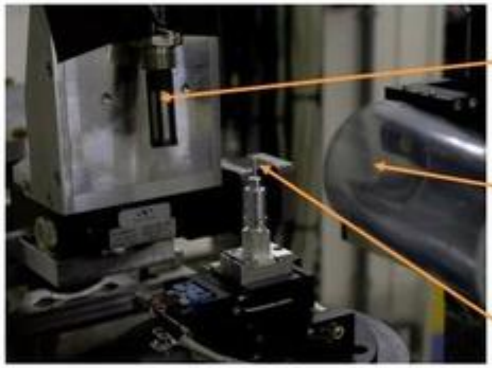
G. Le Duc et al. " Toward an Image-Guided Microbeam Radiation Therapy Using GadoliniumBased Nanoparticles “ ACS Nano (2011)

S. Dufort et al., "The High Radiosensitizing Efficiency of a Trace of Gadolinium-Based Nanoparticles in Tumors" Sc. Reports (2016)

SAMPLE ENVIRONMENT

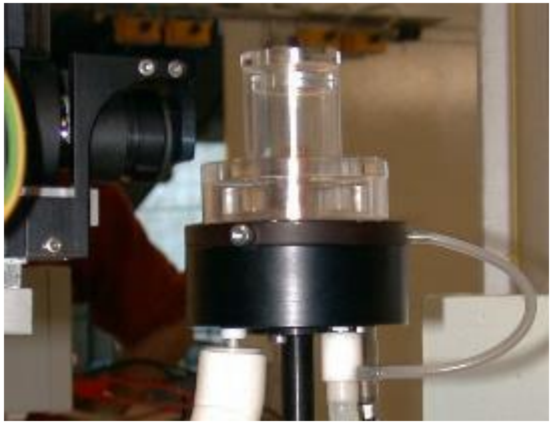
Furnace
T_{max}>1600ºC

Hygrometry

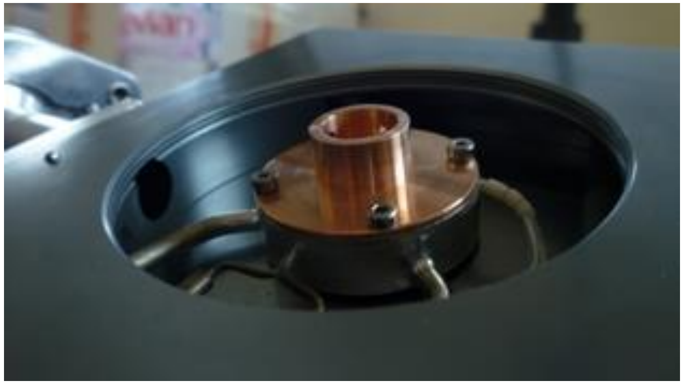


Mechanical load

Cryo
Cold cell (-150ºC)

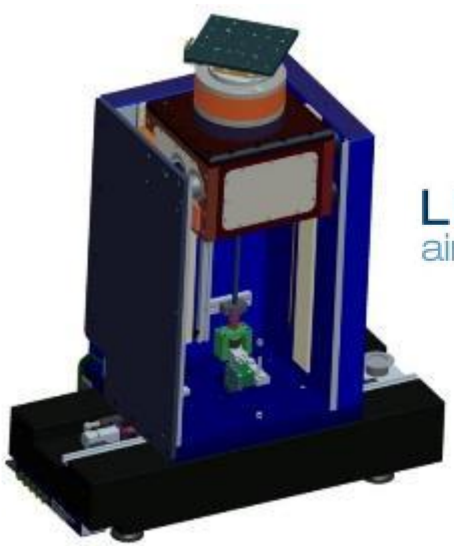


Cooling/heating cell

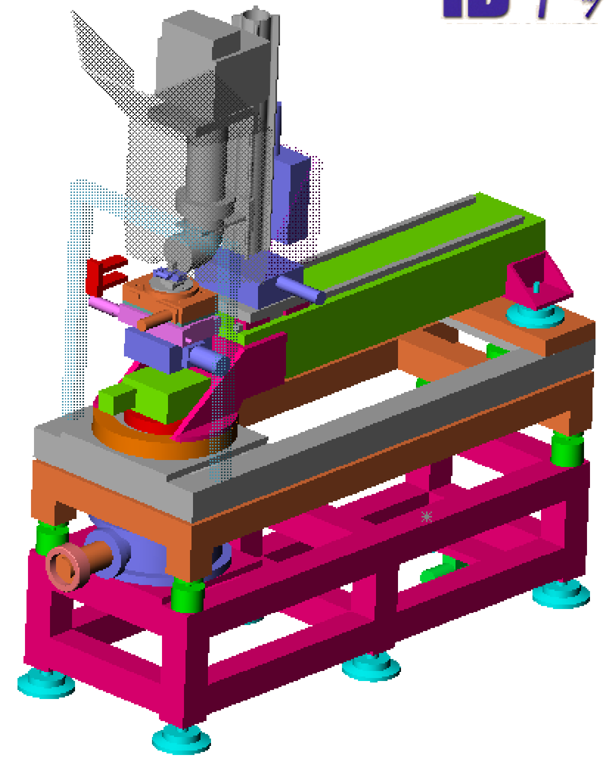


SAMPLE ENVIRONMENT

ID 19



LEUVEN
air bearings



2 sample stages for “big” samples:
- up to 30 kg
- up to 50 cm large
- z stroke of 50 cm

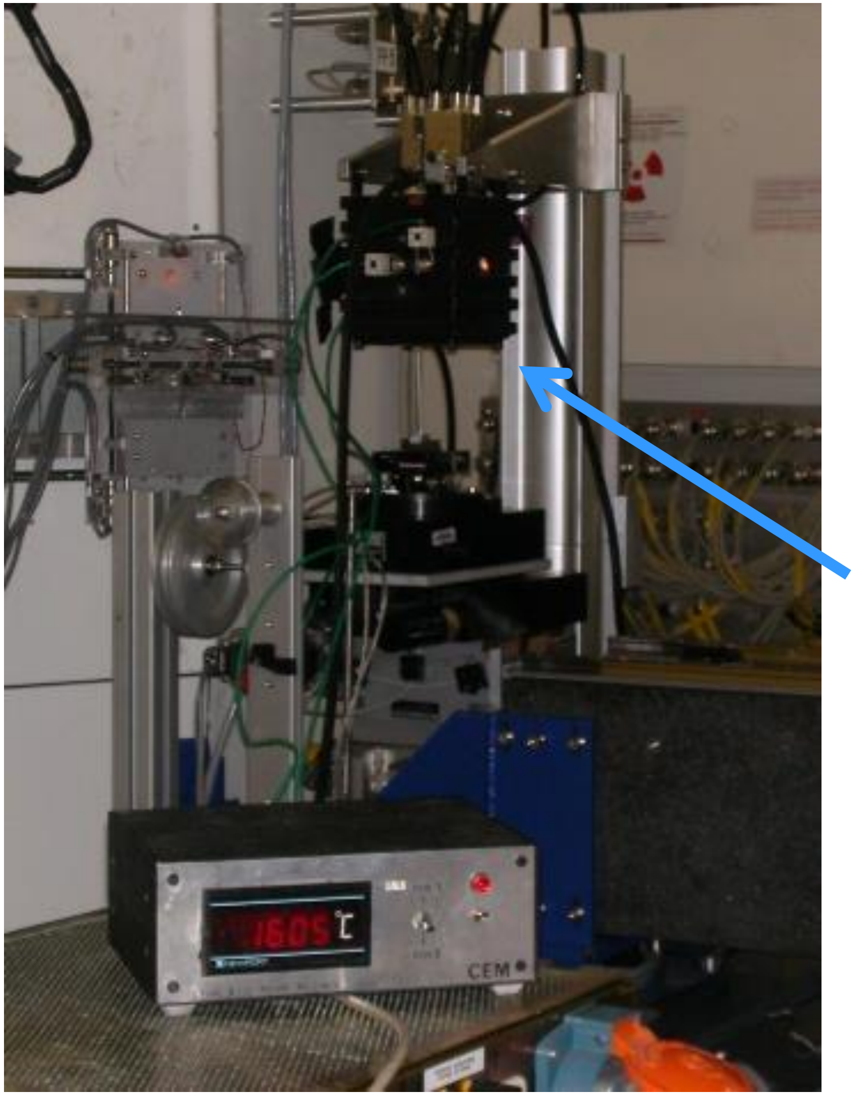
One in our experimental hutch

One in our monochromatic hutch, allowing longer propagation distance (14 m!)

Medium resolution 2.75-47 microns

High resolution 0.12-2.8microns

SAMPLE ENVIRONMENT



Several furnaces:

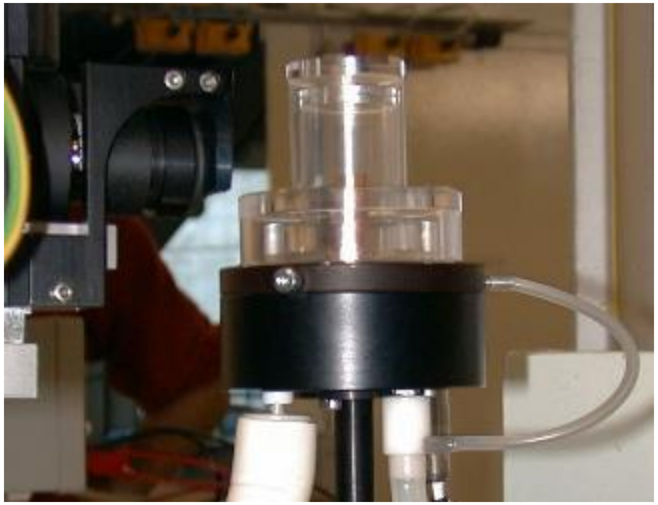
Collaboration with SIMAP
Tmax=800°C
Al alloys

Collaboration with ENSMP
Tmax=1600°C
Ceramics, glass solidification

ESRF, induction with atmosphere controlled
Tmax>1600°C
Slip ring for fluids available

Minimum scan time for a 3D image on ID19 using SCMOS camera: 0.2s
50ms on ID15 beamline!

SAMPLE ENVIRONMENT



Collaboration with CEN Météo France

Cold cell (-150°C/50°C)

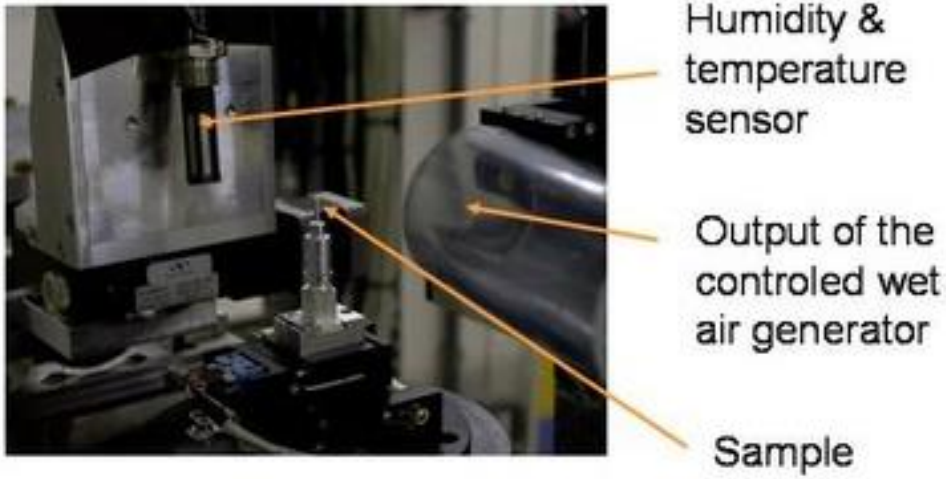
Cryostream

Snow, ice, ice cream

Collaboration with EFPG-3SR Laboratory

Hygrometry control device

Paper



SAMPLE ENVIRONMENT

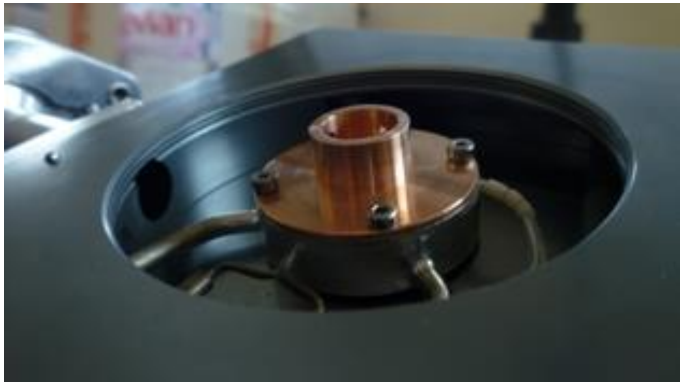
Collaboration with MATEIS

Tension/compression stage

Fatigue stage

Hot traction device

Al alloys, steel



Collaboration with ENSMA

a new cooling/heating cell developed with the ESRF sample environment laboratory, based on a Linkam commercial device

Composite for aerospace, soap

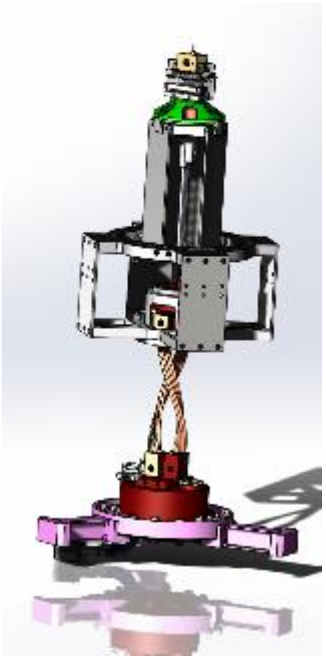
→ Possibility of real in situ experiments

SAMPLE ENVIRONMENT

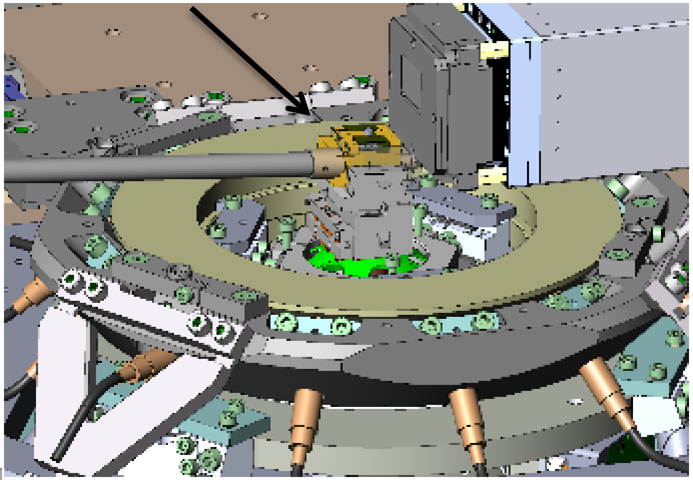
ID16A

Cryo loading chamber and transfer system

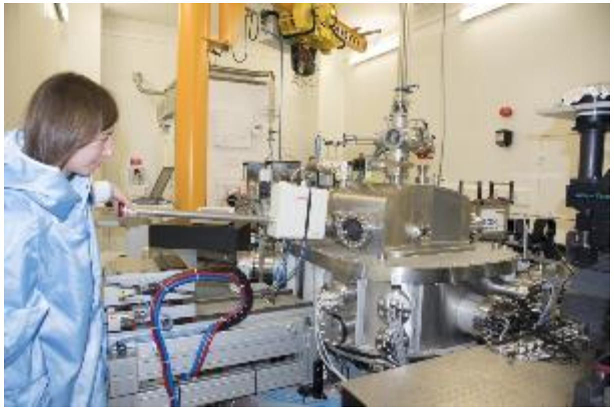
Cryogenic cooling of the sample stage



Leica EM-VCT Cryo-transfer system



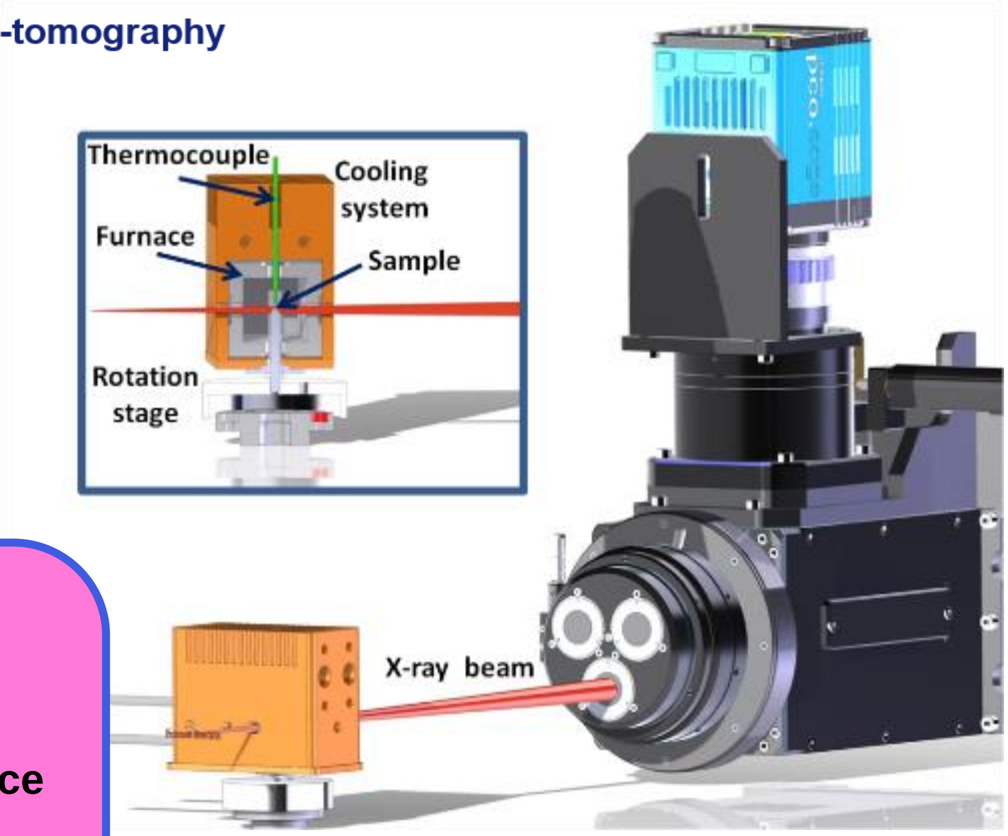
Integrated to the "Hexapiezo" end-station



SAMPLE ENVIRONMENT

ID16B

Technique:	Phase contrast nano-tomography
Energy range:	17.5 and 29.6 keV
ΔE/E :	10^{-2} (pink)
Photon flux:	10^{10} to 10^{11} ph/s
Detector:	PCO edge
Pixel size:	100 nm
Time scan :	16 s
Temperature range:	400°C to 1400°C



Key points:

→ Temperature stability in the hutch
→ Temperature stability inside the furnace
→ Fast acquisition speed
→ Continuous acquisition (multi-turns)

Next step:
→ Multi-scales

Collaboration with Luc Salvo and Pierre Lhuissier from SIMAP