SYNCHROTRON X-RAY MICROTOMOGRAPHY FOR MATERIAL STUDIES

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CRISP 17/03/2014 - Elodie Boller
1 - Introduction

- Microtomography at ESRF
- ID19 beamline: dedicated to imaging techniques
  - Suitable instrument to study material microstructure

2 – Material applications using the different approaches:

- High/medium resolution (0.17-50 microns)
- Monochromatic / pink beam (high energy)
- Absorption microtomography
- Phase contrast imaging – Paganin approach
- Multiresolution
- Fast acquisition (1s / 3D image)

3 – Nanotomography

4 – Conclusion and perspectives
Introduction:

From MACRO to NANO

Macro-structure

Micro-structure

Ultrasound structure

Composition

Collagene, Crystal

Atomic structure

IMAGING TECHNIQUES

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Lambert-Beer law:

\[ \frac{I}{I_0} = \exp \int -\mu dx \]
FROM 2D TO 3D: TOMOGRAPHIC RECONSTRUCTION

N projections

Commonly 10 minutes

Commonly 3 minutes
Using GPU cluster
For $2048^3$ volume

Z reconstructed slices
MICROTOMOGRAPHY @ ESRF

ID22 Micro-XRF/XRD/XAS Combined to tomo → NiNa Project (ID16)
ID19 Beamline mainly dedicated to Microtomography
ID17 Tomography Large field
ID16 NiNa Available in May 2014
ID15 Tomography high energy Ultrafast tomography
BM05 Two additional setups for Microtomography
ID11 DCT setup 27/07/2011
# Microtomography: Comparison X-ray Lab/Synchrotron

<table>
<thead>
<tr>
<th>X-ray Lab</th>
<th>Synchrotron – ID19</th>
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<tbody>
<tr>
<td>Low flux</td>
<td>High Flux</td>
</tr>
<tr>
<td>Acquisition time decreased</td>
<td></td>
</tr>
<tr>
<td>Local tomography</td>
<td></td>
</tr>
<tr>
<td>Not really monochromatic</td>
<td>Monochromaticity</td>
</tr>
<tr>
<td></td>
<td>Huge energy range</td>
</tr>
<tr>
<td></td>
<td>Quantitative</td>
</tr>
<tr>
<td>No coherent beam</td>
<td>Coherent beam = holotomography</td>
</tr>
<tr>
<td>Cone beam = Magnification</td>
<td>Parallel beam = no magnification</td>
</tr>
<tr>
<td></td>
<td>But different optics available</td>
</tr>
<tr>
<td></td>
<td>Pixel size from 0.17µm to 50µm</td>
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</tbody>
</table>
MICROTOMOGRAPHY: COMPARISON X-RAY LAB/SYNCHROTRON

12 million years tooth

Beam hardening problem...
+ possibility to reach much higher resolution
+ much higher signal to noise ratio
PHASE CONTRAST IMAGING

\[ n \sim 0.999999 \]
\[ n = 1 - \delta + i \]

\[ \delta \Leftrightarrow \text{phase} \quad \beta \ll \delta \]
\[ \beta \Leftrightarrow \text{absorption} \]
4 distances: absorption + 0.2 m, 0.5 m and 0.9 m
800 angular positions
multilayer as monochromator: total time ≈ 40 minutes

E = 18 keV

Absorption  Direct Imaging  Phase contrast

β-map  Al/Si δ-map  Al

100 µm
Absorption vs. Phase contrast

**β-map**

\[ \frac{\Delta \rho}{\rho} \approx 2\% \]

**δ-map**

\[ \Delta \delta \approx 3.5 \times 10^{-8} \]

\[ \Delta \rho \approx 0.05 \text{ g/cm}^3 \]

Luc Salvo (SIMAP, Grenoble) Peter Cloetens (ESRF)
Microtomography – Phase contrast imaging  
Laminography – Interferometry  
Diffraction contrast tomography  
Compton scattering

Different « inhouse » groups:

- Bone (F. Peyrin)
- Fast imaging/Interferometry (A. Rack)
- Laminography (L.Helfen)
- Paleontology (P. Tafforeau/V.Fernandez)
- DCT (W. Ludwig) moved on ID11
- Compton scattering (A. Bonnin)

Long beamline: 145 m
Coherent beam
Beam size: 15*50 mm²
Homogeneous beam
Large energy range: 7 - 250 keV
Team of around 20 people
DEDICATED INSTRUMENTS

Detector
specially designed at ESRF
FReLoN CCD camera
Commercial Optique Peter device:
X/visible conversion
Objectives x20 x10 x4 x2
Eye-pieces x4 x3.3 x2.5 x2

Precise rotation 0-360° (cables integrated) and translation

Big translation

Beam

Sample

High resolution 2.8-0.17 microns
New sample stages for “big” samples:
- up to 50 kg
- up to 40 cm large
- z stroke of 50 cm

1) One in our experimental hutch

2) One in our monochromatic hutch, allowing longer propagation distance (13 m!) for medium resolution and high energies

3) One on BM05: additional setup

4) One on ID17 (very large sample) → standardisation
COMPATIBLE SAMPLE ENVIRONMENTS TO ADD A FOURTH DIMENSION

2 Furnaces

Collaboration with SIMAP
$T_{\text{max}} = 800^\circ\text{C}$
Al alloys

Collaboration with ENSMP
$T_{\text{max}} = 1600^\circ\text{C}$
Ceramics, glass solidification

Minimum scan time for a 3D image on ID19 using CMOS camera: 0.2s
1 ms on ID15 beamline!
Collaboration with CEN Météo France

Cryostat, cryostream

Snow, ice, ice cream, trees

Hygrometry control device

Paper
Collaboration with MATEIS

Tension/compression stage
Fatigue stage
Hot traction device
AI/Mg alloys, steel

a new cooling/heating (-20°C/200°C) cell developed with the ESRF sample environment laboratory
Composite for aerospace, soap

→ Possibility of real in situ experiments
2- MATERIAL APPLICATIONS: SEVERAL APPROACHES

- Absorption contrast
  - A
  - Holotomography
    - At least 3 distances
    - Quantitative Density
    - In situ
    - Sample sensitive to dose
  - Paganin approach
    - 1 distance
    - Fast acquisition
    - In situ
    - Sample sensitive to dose
  - Phase contrast
    - \( \phi \)
- Pink beam
  - Qualitative analysis
  - Fast acquisition
- Monochromatic Beam
  - Quantitative analysis
  - Fast acquisition
- Medium resolution
  - 50 - 3.5 microns
Absorption contrast

Monochromatic Beam Quantitative analysis

Pink beam Qualitative analysis Fast acquisition

Phase contrast

Holotomography At least 3 distances Quantitative Density

Paganin approach 1 distance Fast acquisition In situ Sample sensitive to dose

High resolution 2.8-0.17 microns

Multiresolution: 3 different pixel sizes Fully automatized

Holotomography

At least 3 distances

Quantitative Density

In situ Sample sensitive to dose

Monochromatic Beam Quantitative analysis
2- APPLICATIONS

Polyurethane foams (mattresses)

Multiscale application

Abs

Multires

30 mm

7 mm

1 mm
TABLET SWELLING

Abs
Medium resolution
Pink beam

Courtesy of Dr Pete Laity
University of Cambridge

35 keV - 2 minutes per scan
30 micron pixel size

Glass microspheres
to understand the process
NOVITOM
ADVANCED 3D IMAGING

Biscuit

Abs
High resolution
Pink beam

500 µm

Air

Biscuit
3- NANOTOMOGRAPHY : NINA ON ID16

Long beamline with 2 independant branches:
- **ID16A-NI**: ultimate pink beam focus for imaging and XRF
- **ID16B-NA**: nanofocus monochromatic beam for spectroscopy

<table>
<thead>
<tr>
<th></th>
<th>NI</th>
<th>NA</th>
</tr>
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<tbody>
<tr>
<td><strong>Length</strong></td>
<td>185 meters</td>
<td>165 meters</td>
</tr>
<tr>
<td><strong>Spatial Res.</strong></td>
<td>10 – 100 nm</td>
<td>50 nm -1 m</td>
</tr>
<tr>
<td><strong>E/E (%)</strong></td>
<td>1</td>
<td>0.01</td>
</tr>
<tr>
<td><strong>Energy range</strong></td>
<td>Discrete 11 – 17 – 33 keV</td>
<td>Scanning 5 → 70 keV</td>
</tr>
<tr>
<td><strong>Main goals</strong></td>
<td>XRF, coherent XRI-2D/3D</td>
<td>XAS, XRD, XRF, XRI-2D/3D in-situ experiments</td>
</tr>
<tr>
<td><strong>Cryo environment</strong></td>
<td></td>
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<tr>
<td><strong>Main fields</strong></td>
<td>Biology &amp; Life Sciences, Nanotechnology &amp; Nanomedicine</td>
<td>Biology, environmental sciences, geoscience, materials sciences, ...</td>
</tr>
</tbody>
</table>
ID16A-NI: NANO-IMAGING

Scientific drivers

Anti-malarian drugs Hemozoin crystal

Nano/Micro-Technology:
3D Integration

Bone ultrastructure; Langer et al, PloS One
EXPERIMENTAL SETUPS

With P. Thibault et al. (translations and rotation)
3 - CONCLUSIONS

Synchrotron Radiation enlarges considerably the applicability and sensitivity of the method
High Intensity – resolution
Short acquisition time
Coherent beam – phase contrast imaging
Huge energy range
Sample environment

For industrials: microtomography experiments mainly at ID19/BM05
Full service provided, quick access, total confidentiality
Other BLs also accessible (ie ID15, NiNa soon)

Image processing in collaboration with 3D data analysis specialists
ESRF UPGRADE

ESRF Upgrade Programme: 2009-2015 Phase I

To keep/improve our specificities:

- Possibility to image bigger samples at higher energies
- Higher flux at high resolution (transfocator)
- New multimodal monochromator
- Graphical user interface
- Fast acquisition: online reconstruction, …
- Detector efficiency improvements: scintillators, optics, camera coating, …
- Sample environment
- Nanotomography: NINA project (Mid 2014)

Instrument in constant evolution

To reach our goal:
Providing new imaging techniques with best possible image quality