A NEW X-RAY MICROPROBE FACILITY:

THE MICRO-XAS PROJECT AT THE SWISS LIGHT SOURCE

Messaoud Harfouche, Daniel Grolimund, Andre M. Scheidegger, Markus Willimann and Beat Mayer

Paul Scherrer Institut

Nuclear Energy and Safety Department

Waste Management Laboratory

Swiss Light Source

18th ICXOM 2005 - Frascati
OUTLINES

- Introduction
- SLS Machine
- Micro-XAS Beamline
  - optics, detectors, ...
- Active Samples
- Applications
  - $\mu$-XAS, $\mu$-XRF, $\mu$-XRD
  - Time Resolved measurements
- Conclusion and Perspective
**MICROPROBES**

<table>
<thead>
<tr>
<th>BEAM</th>
<th>E [keV]</th>
<th>SIZE [μm]</th>
<th>DEPTH [μm]</th>
<th>METHODES</th>
</tr>
</thead>
<tbody>
<tr>
<td>Laser</td>
<td>$h_ν$</td>
<td>&lt;10 - 50</td>
<td>~ 10</td>
<td>Laser-Ablation ICP-MS</td>
</tr>
<tr>
<td>Electrons</td>
<td>5- 50</td>
<td>&lt;0.1</td>
<td>1 - 10</td>
<td>TEM, SEM, EMPA, …</td>
</tr>
<tr>
<td>Protons</td>
<td>~ 2000</td>
<td>&lt;0.3 - 5</td>
<td>5 – 100</td>
<td>PIXE, …</td>
</tr>
<tr>
<td>Ions</td>
<td>10 – 30</td>
<td>&lt;0.5 – 10</td>
<td>&lt;0.1</td>
<td>IMP, SHRIMP, …</td>
</tr>
<tr>
<td>X-rays</td>
<td>&lt;1 – 80</td>
<td>&lt;0.5 – 10</td>
<td>~10 – 1000</td>
<td>μ-XRF, μ-XAS, μ-XRD, …</td>
</tr>
</tbody>
</table>

**SYNCHROTRON RADIATION**

- Intensity, Brillance
- Polarization
- Energy tunability

→ Sensitivity
→ Resolution
→ “Unique” technique
SLS MACHINE

- 2.4 GeV
- 350 mA
- Top Up
Winter 98/99

First Light
Dec. 2000
SLS Machine
SLS Machine

SLS by night
SLS Machine
M**I**C**R**O-**XAS** **B**EAMLINE **A**T **SLS**

**X-ray Beam:**
- **Photon flux:** ~ $10^{12}$ photons/s on sample; brilliance, flux density and source size.
  - Energy range: ~ 4.5 - 20 keV
- **Monochromator:**
  - Energy resolution $dE/E$ of ~ $10^{-4}$
- **Focusing optics:**
  - $1 \times 1 \ \mu m^2$ spatial resolution
  - Focus adjustable to problem

**Investigation of:**
- heterogeneous and dilute samples
- small samples
- active samples

XAS, XRF and XRD at Spatial and/or Time Resolution
MICRO-XAS BEAMLNE AT SLS
Vertical: collimating
Horizontal: 1.4²: 1
OPTICS: Key Components

Rh Torroidal Mirror

Radius = 5.44 km

Vertical = Collimats
Horizontal = Focus

23.0 μm

1.0 m
**OPTICS: Key Components**

**Rh Toroidal Mirror**
- Radius = 5.44 km
- 23.0 μm

**Double Crystal Monocromator**
- CAM
- Fixed-exit
- Cryogenic
- Energy resolution is within a few percent of the theoretical value (Darwin width)
**STATUS:** *Towards 1 μm² X-ray beam*

- **Half clipped beam**
- **V. Focused beam**
- **V./H. Focused beam**

**SCHEDULE**

Till 05/2006
- commissioning
- pilot users

Starting 05/2006
- user operation
EXPERIMENTAL INFRASTRUCTURE

�� **Detectors**
- Ion Chambers
- Ketec (Si)
- 32el. GeSS
- WDX
- CCD

�� **X-Ray Eye**

�� **Electronic Microscope**

�� **Samples Stages (6 axes)**

�� **Detector Portal**
Active specimen containment system consisting of a specimen holder, its safety case with a massive base and a safety base cover, and a steel cover used for transport.
**ACTIVE SAMPLES:** at the micro-XAS Beamline

- **Ge Detec.**
- **CCD**
ACTIVE SAMPLES: at the micro-XAS Beamline

- Ge Detec.
- Pb shielding
- Microscope
- CCD
- 6 axes manipulator
- KB System
**APPLICATION:** μ-XRF, μ-XAS and μ-XRD

- Co oxidation during cement hydration
- Formation of Co(OH)2-, CoOOH- &/or Co(III) phyllomanganate-like phases
**APPLICATION:** Time Resolution

**micro-XAS @ SLS: 08/2005**

\[
\lambda_{\text{pump}} = 400 \text{ nm (350\,\mu J)}
\]

\[
E_{\text{probe}} = 7.121 \text{ keV}
\]
**APPLICATION: XRF**

Measuring ambient air filter samples at the $\mu$-XAS beamline

1 mark represents a 1-hour aerosol accumulation spot
Coarse mode (2.5 – 10 μm) urban aerosol

Average M7A (Average of 130 1-hour coarse mode aerosol samples)

Counts per 15s livetime

Energy (keV)

SLSμXAS, monochromatic 11.5 keV
HASYLAB L, monochromatic 15 keV
Applications:

μ-XRD

Micro-Pillar of Au model (Simulation)

Synthesized Au samples

size = 40 - <100 μm

Data under reduction

Corundum

Au Sample

microXAS, SLS; 09/2005
**CONCLUSION AND PERSPECTIVE**

**μ-XAS BEAMLİNE WILL:**

- Optimal use of the high brightness of SLS complex, heterogeneous and dilute systems
- Offer the combination of XAS, XRF and XRD at the micro scale on the same sample “spot”
- Offer a “unique” opportunities for time resolved studies

**FUTURE:**

- 3D imaging & Tomography techniques
- Free Software for data reduction
- ...
ACKNOWLEDGEMENTS

MICRO-XAS TEAM

PSI:
- M. Vespa
- R. Daehn
- N. Bukowiecki
- E. Van

SLS:
- S. Johnson
- P. Heimann

EPF LAUSANNE:
- Ch. Bressler
- M. Saes
- M. Chergui