NINA: the ESRF Upgrade Beamline for Nano-Imaging and Nano-Analysis

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ID17 Bio-Medical Beamline
BM05 Optics Beamline
ID19 Micro-Tomography

ID21 Micro-analysis / IR end-station (low E)
ID22/ID18F Micro-analysis (high E)
ID22NI Nano-Imaging end-station
Outline

- Scientific case of NINA
- Experimental methods
- Beamline description
NINA will provide nano-focused beams to address key societal challenges
- Health
- Environment
- Energy and Advanced Materials
Poly-functional nano-particles for cancer therapy

Quest for *Magic Bullet Nanoparticles*:
- Potential for *in vivo imaging* of targeted tumour cells
- Increased *target selectivity for cancer cells*
- Increase the dose *efficacy of therapeutic radiation*
- Ability to *inhibit DNA damage responses* in targeted cells

- **Limited (geno-)**toxicity

D Lewis et al, Nanomedicine, DOI 10.2217/NNM.10.96
Societal challenge: Health

X-ray fluorescence nano-imaging of internalized Lanthanide nanovectors

Pt nano-particles target the nucleus

Dr. B. Kysela
Genome Stability and DNA Repair Group
The Medical School, University of Birmingham
Project: MRC grant support

P. Cloetens ESRF
S. Bohic INSERM U836 / ESRF
Metallomics

= study of the metallome
= the location, identity and quantity of free and complexed metals within a given cell type

The trace metals are closely linked to (neurodegenerative) diseases: dysregulation in the homeostasis of one or several metals (e.g., Parkinson, Alzheimer’s, ALS, Friedrich ataxia, Wilson, Menkes)
We are only just entering the cell.

Smaller nano-probe required for studies at the true organelle level:

- Nucleus: 2-5 μm
- Mitochondria: 0.5-1 μm
- Ribosoma: 25 nm
- Chromatin fibers: 20 nm
- Secretory vesicles
- Bacteria

5.10^{-21} g (5 zeptog) / pixel 100 nm in thin neurite-like processes in PC12
Mechanisms of gold biomineralisation in the bacterium *C. metallidurans*  
F Reith et al PNAS 2009 106:17757-17762
Iron nano-precipitates as origin of pre-breakdown in silicon solar cells

Kwapil et al., APL 95, 232113 (2009)

Electrical charge near precipitates → Local electric field
Defect engineering of metal nano-defects

Iron silicide nano-precipitates (20-30nm)

Iron oxide inclusion (microns)

Buonassisi et al., Nature Materials 4, 676 (2005), APS
Main assets of hard X-rays in Nano-Imaging

- Combine **high spatial resolution** on 'thick' samples in 3D
- **Multi-modal approach**: natural and quantitative contrast from electron density, elements, chemistry and (crystalline) structure
- **In-situ experiments** on specimens close to their native state or under a controlled environment
- **Dynamics**: follow evolving systems with fast 2D and 3D techniques
Full characterization at the nano-scale of diluted heterogeneous samples

Multi-modal framework providing complementary information through:

- X-ray fluorescence (XRF)
- X-ray absorption spectroscopy (XANES and EXAFS)
- X-ray diffraction (XRD)
- X-ray excited optical luminescence (XEOL)
- Magnified holotomography
- Ptychographic Coherent Diffraction Imaging (PCDI)
- Generalized computed tomography

**incoherent**

**coherent**
Experimental Methods at NINA

**Scanning Transmission Microscopy - STXM**

*X-ray Fluorescence: element distribution* Slow; trace elements

*XAS, XEOL, Diffraction*

**Projection Microscopy - PXM**

= Ancient History! (dixit J. Kirz)

electron density Dose efficient, fast; magnified holo(tomo)graphy
X-ray diffraction tomography

N_y x N_ø Diffraction Images

Azimuthal Integrations

ID22
Energy = 18keV
Spot size = 1.6x2.3 µm (VxH)
81 steps / angle
90 angles / 360°

N_y x N_ø Diffraction Patterns

Reconstruction

Glass
Ferrite
Calcite
sp3
Cubic

Algebraic Reconstruction Technique

Phase Sinograms

Sum Sinogram

Sum Pattern

XEOL in Nano-LEDs
Projection Microscopy

Magnified in-line holograms of Au Xradia test pattern
$E = 17.3$ keV

$D \approx 29$ mm
Projection Microscopy: phase retrieval

1) Linear, direct method
Linear least squares solution

\[ D_m \]

\[ \tilde{\Phi} (f) = \frac{\sum_m K_m(f) \cdot \tilde{I}_m(f)}{\sum_m K_m^2(f) + \epsilon} \]

+ 2) Iterative improvement (optional)
X-radia gold test pattern
Innermost line width: 50 nm
Energy = 17.3 keV
Field of view: 80 µm
Pixel size: 53 nm

Phase map

Au Fluorescence; 25 nm

9 µm
High energy nano-tomography

3D imaging of Ti-based samples
Sample diameter: 0.4 mm
Energy: 29 keV; voxel size: 50 nm
Similar resolution as FIB/SEM but non-destructive and larger FOV

Multiscale tomography → Ti6Al4V/TiB/5w

**FIB-tomography**

- **Step = 100nm**
- **10x8x8 µm³**
- **ID22- KB optics**
  - Vox=(50.7nm)³ Res ~ 180 nm
  - Destructive!
  - Vox=(13x13x100nm)³ Res ~ 100 nm
  - 10x8x8 µm³

**Summary Ti6Al4V/TiB/5w**

- **ID19- Parallel beam**
  - Vox=(0.3µm)³ Res ~ 1 µm
  - 150x300x546 µm³

- **FIB**
  - Vox=(13x13x100nm)³ Res ~ 100 nm
  - Destructive!
  - 10x8x8 µm³

- **41x41x20 µm³**
Nano-Laminography

Scanning geometry adapted to flat, laterally extended samples

Focus size \( \sim 100 \, \text{nm} \)

F. Xu, L. Helfen, P. Reischig, H. Suhonen et al
Coherent scanning X-ray diffraction

A

detector

specimen

focusing optics

UPBL4 is a long, canted, high β beamline with 2 branches:

- **NI**: ultimate pink beam focus for imaging and XRF
- **NA**: nanofocus monochromatic beam for spectroscopy

**X-ray ultra-microscopy** and **nano-spectroscopy**

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

Infrastructure

End-station NA Microscope NI & Nano-focusing optics

Control & (Online) Data Analysis

Detector Systems

Instrumentation & Stability

TDR Feasibility Study
- Long, **canted**, high beta beamline
- Supplementary 16 mrad deflection by ML optics
Optical Design: overall principles

- **Ultra-long** beamline:
  Usable working distance and target photon density

- **High beta**-section:
  Easier to make horizontal secondary source

- **Small number of optical elements, operate in horizontal plane:**
  Improve stability and robustness
  Preserve brightness, coherence properties

- **Multilayer or metal coated mirrors:**
  Achromatic beamline design (large bandwidth, energy scan)
  High efficiency

- **Primary optics close to the source:**
  Reduced effect on coherence

- **Nano-focusing optics integrated in sample stage:**
  Maximize demagnification
  Improved stability
Focusing geometry NI branch

**Horizontal**

\[ M_{h2} = \frac{q_{h2}}{p_{h2}} \]

\[ q_{h2} \approx 0.046 \quad p_{h2} \approx 145 \]

\[ q_{h1} \approx 11.8 \quad p_{h1} \approx 26.7 \]

\[ 50 \, \mu m \quad 940 \, \mu m \]

**Vertical**

\[ M_v = \frac{q_v}{p_v} \]

\[ q_v \approx 0.1 \quad p_v \approx 183.5 \]

\[ 22 \, \mu m \]
X-ray source: small vertical emittance!

“Record brilliance at the ESRF achieved thanks to ultra small vertical emittance”

Horizontal emittance: **4 nm**
Vertical emittance: **40 pm → 5 pm** (achieved in Users mode) → **2 pm** (future)
### X-ray source

#### Revolvers U18.3/U22.8

<table>
<thead>
<tr>
<th></th>
<th>Revolver IDs NI</th>
<th>In-vacuum ID NA</th>
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<tbody>
<tr>
<td><strong>Period [mm]</strong></td>
<td>18.3</td>
<td>22.4</td>
</tr>
<tr>
<td><strong>Length [m]</strong></td>
<td>2.8</td>
<td>2.8</td>
</tr>
<tr>
<td><strong>Magnet material</strong></td>
<td>NdFeB</td>
<td>NdFeB</td>
</tr>
<tr>
<td><strong>Minimum gap [mm]</strong></td>
<td>11.5</td>
<td>11.5</td>
</tr>
<tr>
<td><strong>Peak field at min. gap [T]</strong></td>
<td>0.285</td>
<td>0.420</td>
</tr>
<tr>
<td><strong>Deflection parameter at min. gap</strong></td>
<td>0.486</td>
<td>0.895</td>
</tr>
<tr>
<td><strong>Fundamental energy [keV]</strong></td>
<td>16.9</td>
<td>10.85</td>
</tr>
<tr>
<td><strong>Total power emitted at min. gap (I=0.2 A) [kW]</strong></td>
<td>1.05</td>
<td>2.28</td>
</tr>
<tr>
<td><strong>Power density at min. gap (I=0.2 A) [kW/mrad²]</strong></td>
<td>125.2</td>
<td>148.4</td>
</tr>
</tbody>
</table>

- **NI branch**: in-air revolver undulators optimised for specific energies
- **NA branch**: in-vacuum undulator, period aims at compromise high energy flux and energy tunability
Intricate layout due to the two branches
Solved. Maximum in-dependency of the two branches.
Nano-Analysis end-station

- Based on setup of the ID22NI pilot project

New capabilities:

- XAS measurements over the full energy range
- Extended energy range: 5 - 70 keV (today: 17 - 29 keV)
- Smaller spot sizes (< 100 nm)
- In-situ experiments (longer working distance when needed)

Commissioned April 2010

transfer of granite table, multilayer coated KB, sample stage, detector stages
Many technological challenges have been solved ...

- Experimental environment (stability) and instrumentation compatible with routine 3D experiments at 50 – 100 nm scale
Nano-Analysis end-station

Mostly inhouse (ISDD) development, integrated design

ID22NI end-station

Large stroke, high resolution scanner

Metrology results – TY axis

Stroke: 40 mm
Resolution: 12.5 nm
ICEPAP driven

12.5 nm

PIC air bearing spindle

Metrology with 3 x capacitive sensors (Lion Precision)

Bi-directional radial error 5 turns

Max. radial error: 33 nm

Y. Dabin, P. Bernard, M. Nicola (ISDD)
Full FEA modelling for shape optimisation

**ID22-NI horizontally focusing bender**

- Bonded optics
- Mirror width profile machined to +/-3µm
- Close to fracture stress limit
- Optimized Si orientation

**Complete KB**

- Should provide 35 nm x 35 nm focus
- Flux > 10^{12} ph/s (pink) on NA branch

R. Barrett, B. Baker, L. Zhang, Ch. Morawe (ISDD)
Ultimate dynamic focusing KB

- **Focus size**: 60 - 80 nm (user mode ID22NI)
- **Flux**: up to $10^{12}$ ph/s
  \[\rightarrow 1.5 \times 10^8 \text{ ph/s/nm}^2!\]
- **Energy range**: 17-29 keV
- **Tunable bandwidth**: $\Delta E/E \sim 1.5 - 7\%$

Kirkpatrick-Baez Optics

**Knife-edge scan**

Counts

Analytical derivative:

FWHM: 58 nm (V)

S. Gorelick
Ch. David
NI cryo-microscope

- Coherent imaging techniques (magnified holo-tomography, pCDI)
- 2D/3D XRF analysis
- Optimized for biological samples (frozen hydrated state)

- Change in the way to conduct an experiment / measurement
- Change of technology
- Change of type of users

Large solid angle fluo detection
Metrology correction
Spindle
Vacuum vessel
Sample cooling
Transfer line
Sample storage