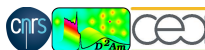


Proposed Timetable for May 26th afternoon

| | | | |
|-------|--------|--|--------------|
| 14:15 | 25 + 5 | Beamline Presentation | JF. Berar |
| 14:45 | 40 + 5 | Structural properties of nanostructures | H. Renevier |
| 15:30 | 25 + 5 | Activity in metallurgy and applied materials | F. de Geuser |
| 16:00 | 15 | <i>pause</i> | |
| 16:15 | 15 + 5 | Grazing Incidence Small Angle X-ray Scattering (GISAXS) at the CRG-D2AM beamline | M. Maret |
| 16:35 | 15 + 5 | Structure and physical properties of crystalline polysaccharides | Y. Nishiyama |
| 16:55 | 10 + 5 | Pixel detector development | JF. Berar |
| 17:10 | 20 + 5 | Perspectives | N. Boudet |
| 17:35 | 45 | <i>BeamLine visit and open questions</i> | |

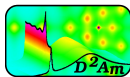




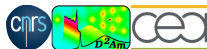
D2AM presentation to ESRF Beam Line Review Panel

J.-F. Bézar

An anomalous beamline from DAFS to SAXS.
Diffraction **D**iffusion **A**nomale **M**ulti longueurs d'onde



May, 26th 2009



1 Beamline Presentation

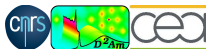
- a CEA-CNRS beamline
- User access
- Scientific production

2 Instruments and optics

- Optics
- Instruments

3 Scientific Results

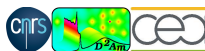
- D2AM Expertise
- Examples and Chosen talks
- From scientific project to upgrade project
- Expected result from upgrade project



Beamline Presentation

A CEA and CNRS beamline for materials science and biocrystallography, project leaders : J.P. Simon and M. Roth. Design optimised for Anomalous Diffraction and Diffusion

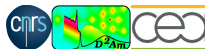
| | | |
|------|--|-----------------------|
| 1994 | <i>Beamline aperture : Material Science and Biocrystallography</i> | |
| 1997 | First Review panel | First FO-CCD |
| 1998 | <i>Fully dedicated to Material sciences</i> | |
| 2002 | | new Princeton FO-CCD |
| 2004 | Last review panel | |
| 2006 | | new goniometer circle |
| 2007 | First upgrade APS | |
| 2008 | | new GISAXS vessel |



a CEA-CNRS beamline

- a CRG at ESRF → No cost induced for ESRF
Management, local contacts and technical supports rely exclusively on CNRS and CEA personnel. Only very specific tasks are supported by the ESRF through the CRG-coordination staff.
- "Conseil d'Administration" (budget, orientation)
- "Structure d'exploitation" (staff, activities)

Following the opening of the SOLEIL synchrotron, the 2007 CRG-"Conseil d'Administration" decided to establish a common review committee for the French community, so that all French proposals could be "treated" on equal terms. As well as a single review committee, this also implies reimbursement for users coming to a CRG at the ESRF, which is not the case at present for CRG users. This will also induces some management modification, French CRGs becoming a "TGE"



Beamline Presentation : local staff

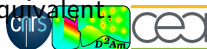
4 permanent CNRS employees of Institut Néel:

| | | | |
|------------------|--------------------|------------------|-------------------|
| <i>S. Arnaud</i> | <i>J.-F. Bézar</i> | <i>N. Boudet</i> | <i>B. Caillot</i> |
| technician | responsible | scientist | engineer |

Support from laboratories in Grenoble → cover various scientific topics.

| | | |
|------------------------------|---|---|
| NEEL CNRS-UJF | J.-L. Hodeau, E. Dooryhee | crystallography of functional materials |
| SIMaP Grenoble-INP | F. Bley, M. de Boissieu, M. Maret, J.-P. Simon | physics of metal and engineering |
| LMGP Grenoble-INP | H. Renevier | nano materials |
| CERMAV CNRS | C. Rochas | soft condensed matter |
| LSP UJF | E. Geissler, I. Morfin | |
| INAC CEA | V. Favre-Nicolin | nano materials |

10 scientists on a part-time basis \longleftrightarrow 2-3 full-time equivalent.



Beamline Presentation : user access.

Allocated : 1/3 by ESRF committees. 2/3 by French CRG committee.

Ratio requested/allocated shifts (or proposals) is about 2-2.5.

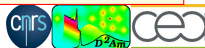
| year | Total† | BLC | IHR | Users* | ESRF users | | CRG users | |
|------|--------|--------|--------|------------|------------|-------|-----------|-------|
| | shifts | shifts | shifts | shifts | shifts | prop. | shifts | prop. |
| 2005 | 651 | 51 | 150 | 441 | 141 | 11 | 300 | 24 |
| 2006 | 678 | 48 | 153 | 456 | 132 | 10 | 297 | 25 |
| 2007 | 669 | 36 | 120 | 498 | 102 | 9 | 354 | 28 |
| 2008 | 690 | 30 | 153 | 486 | 195 | 11 | 291 | 19 |

†including pract. and indus. shifts, Users' total (*) includes shifts from "reserve" lists of ESRF and CRG **review committees** (above **420** shifts/year).

≈ 8 days/year : teaching practicals (HERCULES, ESONN,... , UJF, Grenoble-
INP, GISAXS S, HSC...).

≈ 1-2 days/year : industrial companies under contract with the ESRF.

Very few beam line failures (motors, cryostat), any lost time replaced in the following months by using beamtime reserved for maintenance.

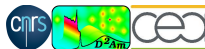


Beamline Presentation : scientific production 2005-2008

197 publications \Leftrightarrow **137 experiments from committees** \Rightarrow **IHR effect.**

| journal | 2001 2004 | 2005 2008 | journal | 2001 2004 | 2005 2008 |
|-----------------|--------------|--------------|------------------|--------------|--------------|
| Nature Mat. | - | 2 | Acta Materialia | 1 | 7 |
| Phys. Rev. Let. | 3 | 5 | Appl. Phys. Let. | 2 | 2 |
| Phys. Rev. B | 5 | 11 | J. Appl. Phys. | - | 9 |
| J. Appl. Cryst. | 6 | 8 | Polymer | 2 | 7 |
| Macromolecules | 2 | 10 | Biomacromol. | - | 5 |
| Langmuir | 5 | 5 | Carbon | - | 3 |

D2AM is widely involved in **training activities** (HERCULES, ...).
At least **17 PhD theses** based on experiments defended since 2005.
More than **12 invited lectures at international conferences** directly related to experiments carried out at D2AM.



anomalous Optics with low parasitic scattering

Mirrors : platinum-coated silicon, low pass filter, focuses in the vertical plane.
Double crystal **monochromator** : Si(111) or Si(311), focuses sagittally.

Si(111) Optic summary

5 - 25 keV $\Delta E/E = 10^{-4}$

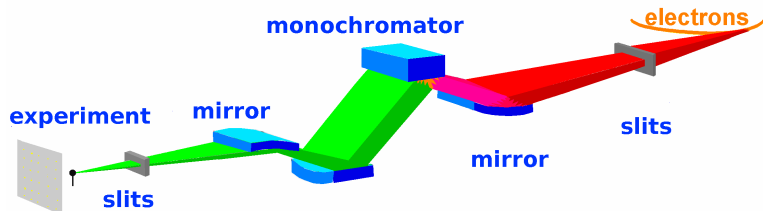
BM02 bending magnet, $E_c = 20.6$ keV

Cr K-edge \rightarrow Pd K-edge Ti used

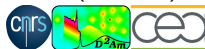
Demagnification of the source 3 / 1

High harmonic rejection

Stability : coherent measurements over 24h, no feed-back needed



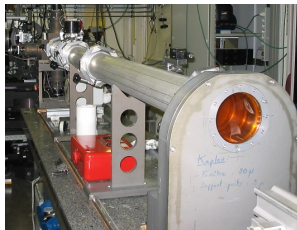
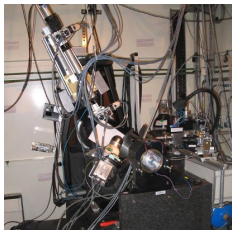
Delivers photons from 5 to 25 keV (35 keV using Si(311)) to the experimental hutch in a spot of a few hundred microns, spots of $70 \times 100 \mu\text{m}^2$ can be attained.
Flux at the sample position : $\approx 2 \cdot 10^{10}$ ph/s for 1 mm^2 at entrance slits (200 mA).



Instruments : goniometer and small angles camera

"7-circle" goniometer : Euler cradle for the sample, detector arm, analysers, optional GI circle and ancillary equipment.

Si or Ge (ΔE lower than 5 eV), curved graphite ($\Delta E \approx 200$ eV at 10 keV)



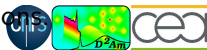
Small angle camera : sample-detector distance (0.3 - 2 m).

Q range $3 \cdot 10^{-3} - 1 \text{ \AA}^{-1}$ (8-15 keV)

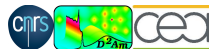
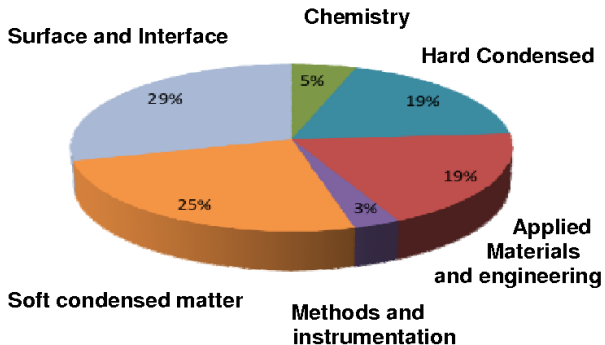
Ultra SASX $\rightarrow 4 \cdot 10^{-4} \text{ \AA}^{-1} \Rightarrow$ overlap with visible light scattering.

High pressures, high temperatures $\Rightarrow 20$ keV : Q range $\rightarrow 2 \text{ \AA}^{-1}$.

GISAXS : dedicated vessel, secondary vacuum, sample motions.



Scientific fields



- **Diffraction and diffuse scattering**

- ▶ crystal structure \rightarrow 1st structure of a quasi crystal
- ▶ multilayers

- **Anomalous scattering**

- ▶ nano object characterization : semi conductors and oxides
- ▶ amorphous radial distribution : metal bulk glasses

- **DAFS**

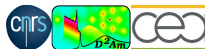
- ▶ from spectroscopy (Fe_3O_4) to nano object characterization

- **GISAXS**

- ▶ feasibility of quantitative anomalous result establish

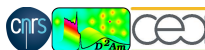
- **SAXS** - Soft and hard condensed

- ▶ quantitative anomalous results available for both soft and hard condensed
- ▶ in situ characterization (time scale : few seconds) with quasi online treatment



Which results to present : chosen talks

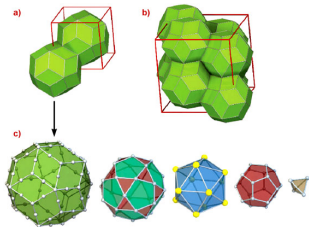
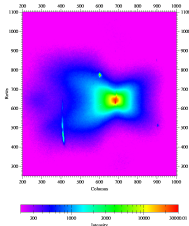
- Focussed on hard condensed and nano : **recognised expertise, stones for our upgrade project**
- Related to the availability of collaborators
- **2 scientific main topics**
 - ▶ H. Renevier :
Structural properties of nanostructures
 - ▶ F. de Geuser :
SIMaP activity in metallurgy and applied materials
- **2 spotlights on experiments**
 - ▶ M. Maret :
Grazing Incidence Small Angle X-ray Scattering (GISAXS) at the CRG-D2AM beamline
 - ▶ Y. Nishiyama :
Structure and physical properties of crystalline polysaccharides
- **1 spotlight on development**



Other material science spotlights

Synchrotron X-ray radiation provides some of the most powerful tools to study these structures and thereby gain an understanding of why materials behave as they do, and how properties can be tailored and improved for specific applications. The examples presented below cover only the very tip of the iceberg as far as materials research at D2AM is concerned.

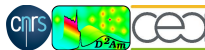
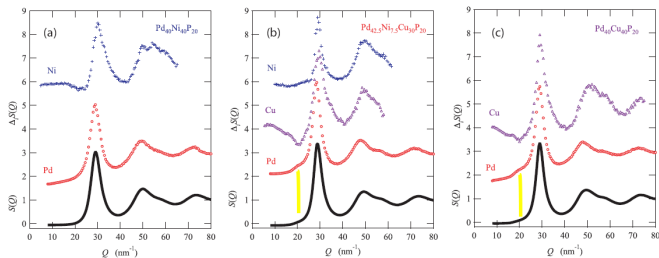
Quasicrystals structure : bi-atomic quasicrystals have been produced, allowing this question to be answered and giving insight into their cluster structure.



and diffusion : quantitative measurements allow determination of phason elastic constants

Zeolites and amorphous bulk metal

- **Powders:** properties of zeolites depend strongly on the location of the small amount of ions that favour the insertion of specific gases : anomalous diffraction \Rightarrow identify and locate them *in operando*
X-zeolite reaction with Xylene gas.
New hybrid materials obtained by incorporating molecules in zeolites.
- **Bulk metallic glass :** very demanding experiment, beam stability and reproducibility
Analyser improvement : curved graphite crystal \Rightarrow collection efficiency

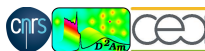


From scientific project to upgrade project

- Increasing demand in nanostructures characterization.
- Materials are no more standalone but in working condition.
- Real materials are often multiphases thermodynamic equilibrium.

⇒ **New performances for the beam line instruments : an upgrade project with its aim on "multi scale materials"**.

- Refurbishment of out of age optics, progress made in manufacturing mirrors and the new surface quality obtained, allow to build a better optic in term of focussing and difuse scattering.
- New monochromator needed to allow dynamic experiments like spectroscopy in diffraction condition (DAFS) and not only static as anomalous ones.
- New goniometer allowing rapid motion and dedicated sample environements
- To take into account the ESRF upgrade (source, flux).



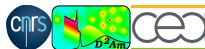
Expected result from upgrade project

- To be able to characterize new nanostructures with High Z elements : apply anomalous and DAFS methods to In, Cd, Te, ... inside dots
- To be able to perform in-situ SAXS characterization, together with WAXS, on new alloys and polymers during thermal or mechanical elaboration process
- To be able to characterize surface objects organisation up to mesoscale
- To reinforce our expertise in applied anomalous scattering

To summarize :

structure of real materials in situ in operando in working devices

We hope the BLRP recommendations will help us in funding this upgrade !



Thanks for your attention

D2AM is the result of a collective work and special thanks :

- to all those working to improve BM2 (local contacts...)
- to our users for their results and suggestions
- to the CRG office
- to my colleagues working on (close to) the beamline
 - ▶ ⇒ *without them no experiment could be done*
 - ▶ ⇒ *they are not listed in order to avoid to forgot some of them*

