Conceptual Design Report for the CRG-D2AM beamline

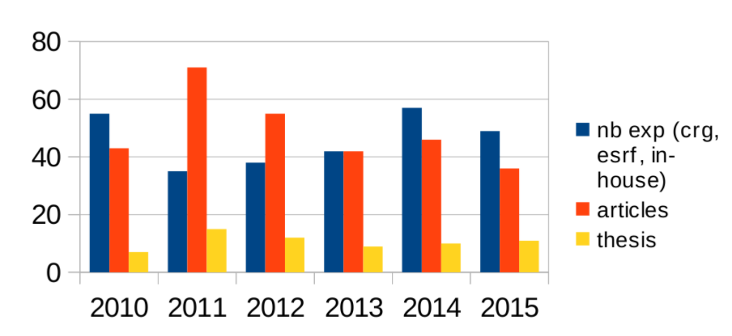
The scientific case for the beam line after the ESRF EBS phase

- The project history:

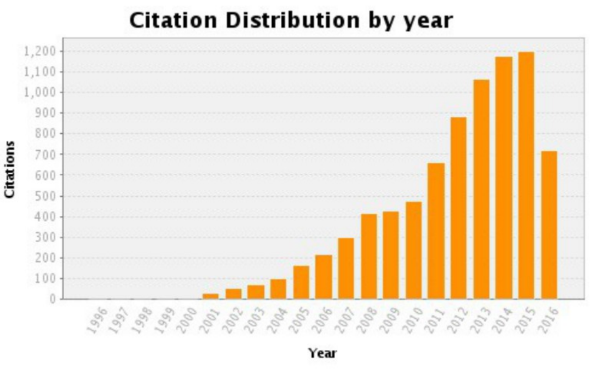
The BM02-D2AM Beam Line (Diffraction et Diffusion Anomale Multilongueurs d'onde), a French CRG dedicated to Materials Science studies with an emphasis on anomalous experiments, was one of the very first beam lines to operate at the start-up of the ESRF in 1994. It is dedicated to investigations into the microscopic structure of materials using two main techniques: (i) wide angle X-ray scattering, which provides information on long-range ordering at the atomic scale and (ii) small angle X-ray scattering, which provides information on the shape of objects at the mesoscopic scale (typically 0.1 to 1 micron). If relevant to the sample under study, both of these methods are coupled with "anomalous" X-Ray scattering, which yields additional chemical information by providing contrast between different atomic species.

Between 2010 and 2016, the beam line was completely refurbished in order to provide state-of-the-art fixed exit optics (including KB mirors) on a bending magnet beam line, with a XAFS monochromator couple to a heavy duty Kappa diffractometer, as well as versatile instruments where users' *in situ* apparatuses can be inserted. To be able to uncover new structural properties, recent developments in materials science demand the ability to perform X-ray characterization of heterogeneous, multi-scale samples under specific conditions, such as:
operando and time-resolved studies of advanced materials used for energy production and storage (batteries, membranes),
in situ mechanical stress applied to biopolymers,
in situ and time-resolved annealing of thin films, nanostructures... under controlled atmosphere

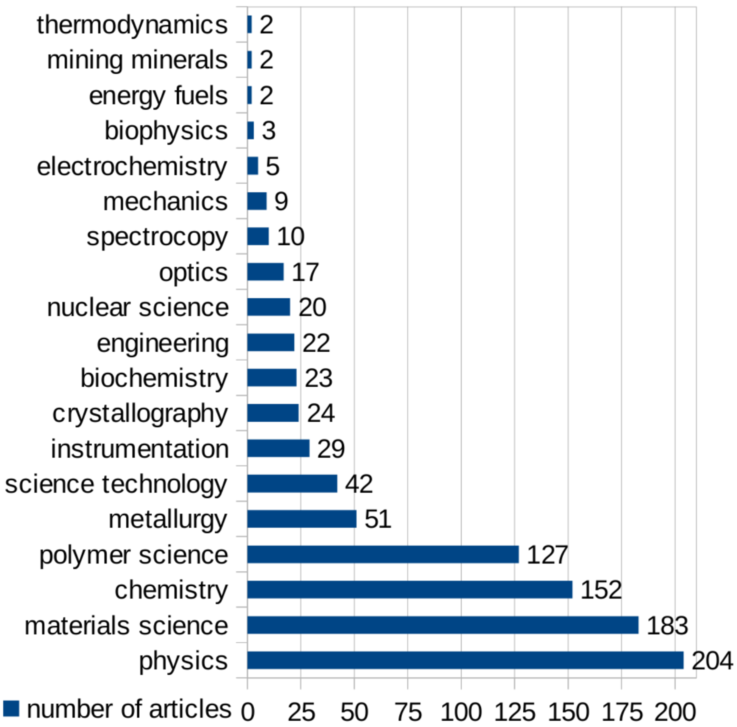
The high level of scientific activity of the beam line is demonstrated by more than 600 publications in peer reviewed journals since 1996. In the period 1998-2016, we recorded 515 publications, with an average citation per article of 15.6 (http://www.researcherid.com/rid/I-6445-2015). Moreover, experiments carried out at the beam line contributed to at least 150 PhD theses since 1998.



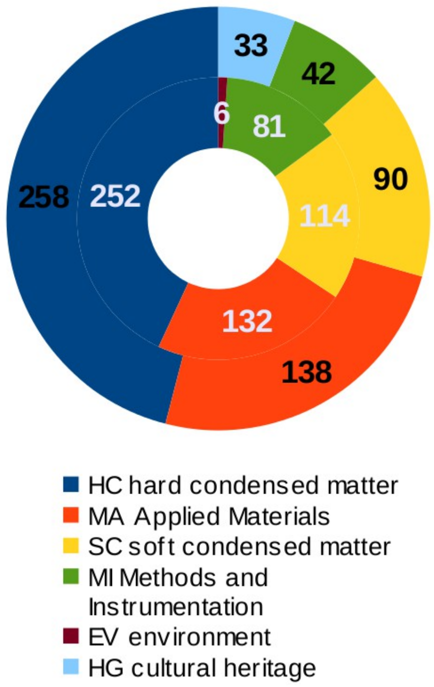
Number of experiments, peer-reviewd articles and PhD thesis recorded each year



Number of citations of D2AM publications from http://www.researcherid.com/rid/I-6445-2015



scientific fields of D2AM publications



Shifts distributed in 2015 (black figures) and in 2014 (white figures) by review committees and for in-house experiments.

- The science case

As a result of its recent upgrade, BM02-D2AM is, and will remain so after the ESRF upgrade, a beam line dedicated to ***in situ* or *operando* structural studies in materials science**: it provides tools to do wide reciprocal space explorations, to measure weak diffuse signals at small or high angles and weak/forbidden Bragg reflections and to take advantage of the anomalous effect close to absorption edges. This permits the study of materials in all their complexity, **from fundamental science to applied physics**: atomic structure, defects, chemical order/disorder, microstructures, scale hierarchy, heterogeneities… The new Kappa diffractometer and the new SAXS optical bench allow investigations of samples in **numerous different environments** such as low or high temperature, electrochemical cells, liquid vapour growth cells, tensile machine… The new X-ray optics delivers to users a clean and stable monochromatic beam from 6 to 40 keV of size 80 (Vert.) x 150 (Horiz.) microns^2 with a flux of about 10^{11} photon/s on the sample. A KB microfocussing system under vacuum will be commissioned on the beam line this autumn, with targeted beam size around 10 to 20 microns² and aiming for a 'reasonable' flux loss. The beam line is now equipped with **3 Silicon hybrid pixel 2D detectors from IMXPAD**: a 1.5 x 7 cm² 'S70' for structural studies and reciprocal space mapping, a prototype 7 x 15 cm² 'S540' for SAXS, GISAXS and X-ray diffraction over a larger angular range and a 9 x 15 cm² 'WOS' (WAXS Open for SAXS) for simultaneous SAXS/WAXS. A strong effort is now being applied to provide a user-friendly data analysis system based on the ESRF PYFAI package.

BM02 is also the beam line on which Diffraction Anomalous Fine Structure (DAFS) spectroscopy was developed [Proietti1999, Renevier2003, Favre-Nicolin, V., Proietti, M. G., Leclere, C., Katcho, N. A., Richard, M. I. & Renevier, H. (2012). The European Physical Journal Special Topics, 208(1), 189–216, Renevier, H. & Proietti, M. (2013). In Characterization of Semiconductor Heterostructures and Nanostructures, pp. 311–359. Elsevier]. A major improvement is the possibility of performing Multiwavelength Anomalous Diffraction (**MAD) and DAFS spectroscopy in grazing incidence**

geometry. The growing interest in the structural studies of 0D to 2D epitaxial nanostructures and heterostructures, thin films and more generaly nanomaterials, has lead to develop and apply MAD, DAFS (and XAFS) spectroscopy, in combination with *ab initio* calculations, to determine strains (in- or out- of the plane of growth) and atomic composition independently, long range and short range orders, as well as the crystallographic polarity [Katcho Europhys. Lett. 2011, Leclere JAP 2014, Hestroffer2011, Consonni2016]. Since, MAD and DAFS spectoscopy have been applied to systems of great technological interest, mainly semiconductor nanostructures as for instance GeSi quantum dots, nitrides nanowires, oxides thin films...

MAD and DAFS in grazing incidence, by using (or not) the KB mirrors, will definitely take advantage of the smaller emittance. Such a specificity and skill are unique in Europe. We definitely want the many interested users to have the possibility to combine MAD and DAFS with a state of the art instrument.

The beam line has been used in a very wide range of **Materials Science**, characterizing short-range and long-range order from gels [Zhang L., J. Colloid Interface Sci. 2016] and amorphous materials [Hosokawa, Stellhorn, J. Non-Crystal Solids 2016] to quasi-crystals [Takakura, Nature Mat. 2007] and nanostructures [Delachat F., ACS Nano 2015], or nanoparticles [Hajiw S., ACS Nano 2015].

D2AM is also widely recognized for its **versatile SAXS** set-up into which it is easy to insert various sample environments (temperature, mechanical tests [Xiong B., Macromolecules 2015], pressure [Nishiyama 2010]) for in situ/operando studies. In 2008-2009, a GISAXS chamber was adapted to the SAXS bench to allow *in vacuo* transfer of users' samples [Babonneau, 2009]. It is now even possible to perform a GISAXS experiment with the sample aligned on the KAPPA diffractometer [PhD C. Matringe, 2016 ; Aissou K., ACS Nano 2016].

After the EBS phase, the D2AM beam line will continue to provide **stable, reproducible, precise** and **low noise characterisation tools** for *in situ* or *operando* structural studies in materials science with strong support from the local contact in the **on-line data analysis**, a key point for the success of in-situ experiments. The present and future main domains of activities were defined during a meeting with local contacts and close collaborators :

- nanostructured thin films and nano-materials: structure, strain, chemical composition of nanowires, magnetic alloys, microelectronic and technological materials, thin oxide films...
- multi-scale and heterogeneous materials: strain, segregation, microstructure, defects, chemical composition of metallic alloys under annealing or mechanical stress, ceramics, cultural heritage materials, materials for energy production and storage…
- polymers and solutions: morphology, organisation, aging and stress, cavitation of charged polymers, nano-precipitation processes, polymeric self assemblies, nano-vectors...

Many studies will benefit from the expected reduced beam size, and gain in spatial resolution such as mapping metallic alloy welds, X-ray diffraction tomography of large cultural heritage samples, structural study of bones. All the grazing incidence experiments will also benefit from the reduced beam size, including MAD and DAFS in grazing incidence.

Choice of the new source

After several iterations with the 4 proposed wiggler configurations, we selected the "**Super Bent Magnet**" configuration, which provides the **cleanest beam** on the sample. Indeed many experiments driven on the beam line require a clean sharp beam with **no tails**.

Steps required to adapt the beam line to the new source

- Modification of the beam line optics and experimental equipment

Since the SBM configuration will supply the same photon flux to our optical elements, no **mandatory modifications** are expected for the beam line optics and experimental equipment.

Nonetheless, **two highly desirable modifications** are already planned: we intend to undertake a new study to reduce and optimize the size of the second crystal of the monochromator (in collaboration with CRG-IF beam line), as well as its bender, which was originally designed to focus a 6mrad BM fan. The second point, raised during a CRG coordination meeting, is the possibility of reducing the size of the Front End Beryllium window. This was designed for 6 mrad, but because we shall receive only 2 mrad, the modification will also allow us to reduce its thickness and thus to gain flux at energies below 8keV.

The last anticipated modification, but at a higher cost, will be to equip our instruments with larger and more efficient **CdTe pixel detectors**. At present we share a 7 x 15 cm² S540 CdTe prototype pixel detector with our colleague from CPPM in Marseille and the detector group at SOLEIL. We are starting to seek financial support to purchase our own.

- Modification of the beam line general infrastructure (hutches, networks,…)

The only expected modification of the beam line general infrastructure will be the realignment of all our elements on the new beam axis. This is possible within the existing hutches.

<u>Support requested from ESRF services for these measures</u>

We will need the support of the ESRF alignment group to localize the new beam path in our optical and experimental hutches, and also of the ISDD and safety to modify the shielding. Furthermore, during a CRG coordination meeting, all CRGs asked that the opportunity of reducing the size and thickness of the Beryllium Front End window should be studied by the relevant ESRF staff.

<u>A timeline and list of milestones</u>

The BM02 staff will realign all beam line components whenever the ESRF alignment group is able to localize the new beam path in the hutches. The new second crystal monochromator and its bender will be installed during the 2019 shutdown at the latest. The same applies to the thinner beryllium window if an appropriate solution is proposed by ESRF.