BM01 ESRF Beamline Review

Introduction:

The Panel was impressed by the high quality and quantity of science as well as technical achievement since the last review. We formed the view that the activities at the SNBL are strongly science-driven and supported by an excellent well-motivated staff who are delivering a cohesive portfolio of techniques from the two member nations. In a multi technique environment it is clear that equal emphasis has been placed on software and hardware development. The scientific and technical achievements clearly benefit the ESRF user community and are all the more impressive in the context of a facility based on a bending magnet source. BM01 operates as an important interface between the various ESRF operations, offering state-of-the-art experimental facilities and data analysis for the chemistry community and more generally across the physical sciences.

On that occasion of the previous review in 2005, a variety of recommendations were made for this CRG including:

- Focusing on a smaller variety of experiments
- Developing stronger in-house research involving post-doc and PhD input
- Exploiting complementary relationships between SNBL and SLS and MAXLAB
- Attracting more users from Norway and Switzerland as well as other groups via ESRF Long Term Proposal Scheme, equipment sharing, scientific exchange etc

Over the last 3 years many of these recommendations have been realised creating now an excellent platform for future scientific success and technical development.

The Panel was introduced to the BM01 team of beamline scientists and received presentations from each of them. These included an overall summary of the beamtime, statistics on user distributions, budgetary information and the impact of scientific publications related to the beamline.

The primary strength of the beamline resides in the four different complementary but related techniques, single-crystal XRD, HRPD, EXAFS and more recently Raman spectroscopy. Combined techniques are increasingly popular, assisted by Raman spectroscopy and environment stages. The expertise and cohesive working environment of the in-house staff is directly responsible for much of this innovation.

Research by Users:

In the documentation and presentations the Panel formed the firm impression that SNBL users were performing some outstanding experiments, e.g. the work of Serre et al (Science 2007) on hybrid networks performed on BM01A and Baerlocher et al (Science 2007) on zeolites performed on BM01B. Taken together, these demonstrate the versatility and complementary nature of both branch lines as well as the ability of the SNBL facility to integrate measurement and analysis to provide leading-edge crystallographic tools for solving complex problems in sophisticated materials and processes. At the same time SNBL has mastered techniques for probing structural chemistry of catalysts under operating conditions (Vukojevic et al, Angew. Chem., 2005) and the thermodynamics of high pressure phase transitions (Dubrovinskaia et, PRL, 2005) relevant to the geosciences and materials science.

Elsewhere the user community has been able to publish increasingly in front line journals. 2005-2007 over 270 papers were published by SNBL users with the average impact factor rising from 2.5 to 3. There is now scope for following up solid-gas reactions in situ including hydrogen storage materials, and for analysing diffuse scattering, for example in multi-ferroic devices with potential for data storage. The SNBL team has constructively attracted user groups that have already made important contributions to sample environment facilities. Recently, for example, SNBL has imported industrial standard gas handling procedures to the ESRF for mixing and high pressure operations that will increase the academic and industrial user community. Since 2005 the scientific portfolio of BM01 has become better focused as recommended by the previous Panel, as testified by the current healthy oversubscription to both beamline branches. In addition, the reconfigured scientific programme has increased complementarity with the SLS. For example, a proposal for using modulation excitation spectroscopy on catalytic reactions on BM01 by ETH Zurich has been highlighted by the last Chemistry Review Committee.

Research by Staff:

Since the last review, a strong in-house scientific programme has been developed. We draw attention to the high pressure phase transition work of Dmitriev, Fillinchuk et al (PRB 76 (2007), PRB 77 (2008) and Angew. Chem. 47 (2008)) on light metal borohydrides, processes which are of great relevance in the design of practical hydrogen storage materials. Likewise, the analysis and theory of diffuse scattering by Chernyshov et al (PRB 76 (2007)) has been demonstrated through magnetic phenomena but has broader application to heterogeneous order-disorder transitions in many crystalline systems. At the present time, studies such as the visualisation of electron-phonon correlation by diffuse scattering rendering images of the Fermi surface in Zn appears to have great promise. More generally, the automation of data collection and analysis under in-situ conditions and in a continuous way will greatly benefit, in our view, the identification of important but subtle modifications in numerous systems.

Technical Status and Future Technical Developments:

BM01 is a mature bending magnet beamline where most of the optical components are of original design. The optical performance remains good for a 3rd Generation Source in all respects, particularly in terms of reliability and stability. Despite the age of these optics, upgrade tests showed that these optics can cope with the increased power load planned for 2011. Furthermore, all of the crucial electrical, vacuum and control systems have been updated recently. This includes the new ICEPAP controls and SPEC software which helps meet the overall ESRF standards and moreover will ensure user-friendliness on BM01. The completed refurbishment programme should also allow reliable operation for many years to come in its present configuration.

During the previous review, the beamline panel recommended concentrating on high-energy x-ray experiments. Judged by the current overview, the panel feel that the beamline scientists have exploited the full range of the bending magnet optimum energies, even above 20keV. Their choice, however, to restrain from exceeding energies above 25keV is well justified, given flux restraints and the otherwise major investment and new design that would be required. The high-energies are already available elsewhere at the ESRF where IDs are much better placed to complement the SNBL capabilities.

BM01A uses standard collimating and focusing mirrors which, together with the sagittally bent monochromator, delivers adequate focus for most of the experiments. However, especially for high pressure measurements, better focussing is needed. For this purpose, the beamline has commissioned multi-layer optics which affords a gain of a factor of 30. Given the natural limitation of a bending magnet beamline, the beamline should not invest resources in order to compete with extreme focusing objectives where other beamlines with insertion device sources are more optimal.

On BM01A, the single-crystal diffraction has produced a scientific champion, exceeding even the best of those at the ESRF. The technology behind these achievements lies, in part, with the import of specialised single-crystal diffraction equipment and associated software from Oxford Diffraction, backed up by stable and reliable beamline optics, and the particularly high motivation and local expertise of the scientists.

On BM01B, the set-up to do almost simultaneous HRPD, EXAFS and Raman spectrometry is unique. There are future plans to install a two strip focusing mirror to achieve truly simultaneous experiments. There is also strong local interest in developing simultaneous two-edge EXAFS capabilities by combining two split beams onto a single spot. The panel are keen for this area to be further explored and exploited as appropriate. There still remains the geometric constraints on BM01B that limit focusing of the tuned beam opportunities for fast time-scale studies.

On BM01A, the existing image-plate system and the CCD-detector are already producing excellent results, but are limited by read-out time. In order to cope with the recommended shorter time regimes, these need immediate upgrading, in our view. BM01B currently has a multi-element Ge detector for fluorescent EXAFS studies which represents state-of-the-art design. For the powder diffraction rig, there is one bank of high-resolution analyser crystals which give very narrow peak-shapes, and this is especially applicable to the refinement of low symmetry structures. However, this technique is slow in comparison to the EXAFS and Raman measurements; so installation of a multi-strip detector system must be regarded a very high priority.

The available sample environments at the beamline are excellent and well supported. Highlights include the Raman spectroscopy set-up, high-pressure and high-low temperature capabilities. The technical achievements of the gas catalysis rig are particularly noteworthy: its state-of-the-art design and implementation resulted in the ESRF adopting the system as a model for safe standard practice.

Regarding sample preparation, the increasingly technically complex experiments require specialist know-how, laboratory space, and off-beamline ancillary instrumentation. We believe that it is essential for them to capitalise upon the potential increased space advantages of the upgrade to obtain more appropriate space. This said, there is a peculiarity about high-pressure experiments that are increasingly attracting beamtime on BM01. In this regard exclusively, collaboration with existing ESRF high-pressure sample preparation facilities and manpower, should be investigated given the particular high specialism in this area.

Future scientific directions:

The beamline has a very bright scientific future building upon the impressive output to date and unique features of the beamline. We feel that the most significant impact will be realised when these unique features are used in combination to solve complex and difficult problems in crystallography, catalysis, and chemical reactivity. In these fields, further major impact is expected both for fundamental research as well as for applied research with industrial partners.

The Panel is pleased to note that SNBL is already very well nested in a network of collaborations with many national institutes in Switzerland and Norway, with SLS/PSI and MAXLAB, as well as with groups on-site. Projects like Insitu@SNBL and the Raman facility greatly profit from this network. On-site we mention the productive collaboration with the Dutch-Flemish CRG Dubble, which widens the options for the user base of both CRG's and enables more efficient use of the resources and personnel. It is unique as it is formalised in 2005 by signing a Memorandum of Understanding. Another fruitful scientific collaboration with ID28 is highly appreciated by the users. SNBL participates in a number of on-site projects.

It is clear that the future of the scientific priorities of the SNBL strongly depends upon the ESRF directions, especially concerning the upgrade. A dialogue on strategic future visions between the ESRF and the SNBL is needed in this regard.

Staff:

The panel was very impressed with the staff's response to the last review. The cohesiveness of the current scientific output reflects the significant effort of the beamline staff that provides highly specialized contributions. We recommend that this in-house collaboration continues and is expanded where ever possible by joint appointments, visiting staff, and especially student and post-doc programs. We feel that the present staff level should be maintained to avoid any compromise in the scientific diversity and output.

Summary of Recommendations:

- The beamline should continue its very successful phase of operation, consolidating on its considerable achievements.
- Priority should be given to those areas which really benefit from the simultaneous combination of techniques (single-crystal XRD/HRPD/XAS/Raman/Mass Spectrometry).
- Improve the speed of powder and single-crystal diffraction (sub-second) to push the frontiers of chemical crystallography by detector upgrades.
- Preserve the capability for stand-alone powder diffraction after the ESRF upgrade as a strategic resource.
- Given the outstanding performances achieved at SNBL in extracting maximal information from 2D re-construction of reciprocal space, extra effort should be put into developing software codes to model the non-Bragg contribution.
- The ESRF and SNBL should discuss a strategic and more productive way to accommodate the protein crystallography interests of the member states of SNBL which do not specifically require the unique features of SNBL.
- There is a lot of integrated systems development at the SNBL which could benefit the ESRF especially during its upgrade phase, and vice versa, and these should form the basis of a much closer collaboration.
- Continue and expand upon student and post-doc programs especially considering collaborative methods of working and joint appointments with ESRF and SNBL.
- During the upgrade phase, SNBL should re-optimize its off-line laboratory and storage space (including the use of ESRF facilities) in order to maximize the efficiency of multi-technique experiments in the future.
- Continue with the very fruitful Memorandum of Understanding between DUBBLE and SNBL and look for similar opportunities with other CRG beamlines and the ESRF.

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