Review panel for BM8 (GILDA)

General note: The panel were impressed with the remarkable quality and quantity of science that the beamline has delivered over the reviewed period of operations.

Summary of recommendations

- The panel recognises that GILDA plays an essential role for a large user community. It provides valuable and much needed general spectroscopy and diffraction capabilities and the continuation of this provision has to be ensured. The multi-purpose beamline GILDA offers access to high-energy spectroscopy, which is only readily accessible at the ESRF.

- The panel strongly recommends a comprehensive refurbishment of the GILDA beamline to ensure reliable operation and to develop the beamline to a performance level consistent with the planned upgrade of the ESRF. This must be implemented in a systematic and timely fashion to avoid undue difficulties for the user community. Specific points to be refurbished follow in order of priority:

    a) Primary optics: 1st priority is the replacement of the current monochromator with a modern high quality spectroscopic grade device, and the installation of a new focusing mirror. This will ensure the provision of a high quality stable and homogeneous beam at the experimental stations of the beamline, and enable improved energy scanning capabilities.

    b) A new multi-element fluorescence detector has high priority. This will reinvigorate the capability of the beamline to study diluted systems which is particularly important, for example in the areas of materials, biological and surface science, chemistry and environmental science.

    c) Given the significant and increasing demand for ReflEXAFS at the beamline, the panel recommends the refurbishment of the reflectivity instrumentation.

    d) The panel recommends that the XRD capabilities of the beamline could be greatly enhanced by the provision of a complementary X-ray fluorescence detector that will give the opportunity of measuring diffraction and absorption at the same time.

- The two scientists that constitute the current staff are not sufficient for running the beamline in the most efficient manner. The permanent staffing level should be increased to include 3 beamline scientists and a technician. The panel then further recommends that the team be completed by the appointment of at least one PhD student and/or post doc.

- The panel encourages the continuation of the transition to the standard ESRF hardware and software.

ESRF, Grenoble, May 27, 2009

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Introduction
The General Purpose Italian Line for Diffraction and Absorption (GILDA) was established for investigating the local microscopic structure of matter and its relation with macroscopic physical properties, to elucidate problems in physics, chemistry and earth science. The review panel (chair M. Sandstrom) was impressed with the remarkable quality and quantity of the scientific output on this beamline, where the staff closely collaborates with many groups of the Italian user community, as was demonstrated in the excellent presentations of the staff.

Research activities
The recent research (2004-2009) performed on the beamline is extensively described in the comprehensive documentation presented to the review panel. The research topics were personally presented at the meeting of the panel and commented on by the staff members with the following emphasis: optoelectronics, nanoparticles, semiconductors and interfaces, archaeology and cultural heritage, followed by an overview of the diffraction activity in earth science and catalysis. The written report also covered studies of materials for energy production and transport, life science, soft condensed matter, catalysis, materials exhibiting special thermal expansion properties, phase transitions, local lattice distortions, instrumentation and data treatment methods.

The main components of the scientific activity are, according to the presentation, 36% solid state physics, 33% earth science, 21% in chemistry or physical chemistry. Materials science is the generally dominating activity, although many other topics are covered on this general purpose beamline with 82% X-ray Absorption Spectroscopy (XAS) and 18% X-Ray Diffraction (XRD) experiments. The results and publications are quantitatively and qualitatively on a very high level. The report lists 215 papers from 2004 to 2008 in international peer-reviewed scientific journals, with the average impact factor close to 3.0. This is a remarkably high output of the 199 experiments carried out in that period.

The high profile archaeometric studies have continued on glasses, lusters and pigments. ReflEXAFS studies have played an important role for the studies of thin films and catalytic reactions. Methods have been developed where e.g. surface and bulk sites for As dopants in Si have been distinguished by changing the penetration depth. New software dedicated to quantitative analysis of EXAFS data collected in total reflectance mode has been developed.

About 70% of the scientific studies are performed on dilute systems reflecting the niche of the beamline for providing high quality XAS spectra by means of fluorescence and total reflectance techniques. The energy range includes the K-edges from Cr to Er, with about 20% of the experiments carried out at energies above 20 keV. That wide energy range, from about 5 to well above 50 keV, offers opportunities for many potential user communities, also including the large ESRF community. Although many of the topics have industrial interest, e.g. the studies on optoelectronic glass, catalysts and hydrogen production processes, the involvement of industrial partners are mainly via University groups.

Beam time applications
The delivery of the beam time with 2184 shifts allocated in 10 semesters is in accordance with the average of the ESRF public beamlines. In agreement with the ESRF-CRG contract $^{2}/_{3}$ of the shifts have been allocated to the Italian community by a National Scientific Committee, and $^{1}/_{3}$ by the ESRF beam allocation panels. Totally more than 80% of the shifts on this beamline were allocated to Italian user groups.

For the CRG's part of the beam time the factor for the requested to available shifts is about 1.5 to 2. The oversubscription ratio is significantly higher for the public (ESRF) beam time.

Highlights
There were several impressive highlights covering a wide field of applications and a wide energy range. They include the studies of Er-doped Si nanoclusters, a promising light emitting material in Si-based optoelectronics (Phys. Rev. 2006), and of In-dopants in pure or C-doped Si (Appl. Phys. Lett. 2006). The aggregation of Fe in a GaN-matrix elucidates the ferromagnetic features of magnetically doped semiconductors (PRL 2008). The structure of the Fe/NiO(001) interface with increased spin magnetic moment of the Fe atoms have been characterised (PRL 2006). Depth-selective ReflEXAFS measurements, combined with theoretical DFT studies, were used to study the penetration of As atoms into Si surfaces (J. Appl. Phys. 2007). The peculiar phase transitions of Ga in nanodroplets and thin films were studied with XAFS fluorescence (JACS 2007). Sub-nanometric metallic Au particles that sensitise Er photoluminescence (Appl. Phys. Lett., 2006) were investigated, as also the cause of the negative thermal contraction for Au particles (Phys. Rev. B 2008) and in CuCl (Phys. Rev. B 2007). The redox behaviour of Pt nanoparticles on ceria-zirconia support, a three way catalyst, has been studied (J. Phys. Chem B, 3006), as also a catalytic process to split methane and water to produce hydrogen (J. Appl. Cryst. 2005). An archaeometric application revealed the reaction causing blackening of the copper pigment azurite in a XV century painting (Appl. Phys. 2008).

Technical aspects of instrumentation
The beamline constitutes an excellent facility for XAS studies, particularly for dilute systems, including the capability of grazing angle geometry. A new cell has been developed to allow *in situ* studies of gas-solid chemical reactions. Feasibility studies of a superconducting Transition Edge Sensor, widely used in astronomy with a very high energy resolution, have been performed.

At present some refurbishment is taking place regarding the beamline stepper motors and vacuum control system. Standard ESRF software and instrumentation for vacuum control is being installed.

However, after about 15 years of operation the bulk of the instrumentation has overcome its reasonable operating lifetime. A major refurbishment is necessary to continue reliable operation for the next 5 years and beyond. Especially the optical components, the

monochromator and the mirrors, need replacement in the near future. Plans for beamline refurbishment with design criteria particularly adapted to total reflection/glancing angle mode as well as to conventional high quality EXAFS measurements, were presented by the beamline staff. Those plans take advantage of the Upgrade Plan of the ESRF that will provide an increase in the high brilliance of the source as well as increased stability.

A new multi-element fluorescence detector to replace the present 10 year old system still in use will increase the capability of the beamline to study diluted systems. This is particularly important for example in the areas of materials, biological and surface science, chemistry and environmental science. The panel also recommends an enhancement of the XRD capabilities of the beamline by the provision of a complementary X-ray fluorescence detector that will give the opportunity of measuring diffraction and absorption at the same time.

Future scientific directions
With projected technical refurbishments this beamline will not only maintain but also significantly improve its status for the foreseeable future (next 5 years and beyond). The panel recognises that the multi-purpose beamline GILDA plays an essential role for a large user community. It provides valuable and much needed general spectroscopy and diffraction capabilities with access to high-energy spectroscopy. The continuation of this provision, which is only readily accessible at the ESRF, has to be ensured.

Staff
The scientific quality of the staff is of a high standard. The support provided to the user groups is professional and friendly, and staff members play important scientific roles in most projects. However, the two scientists that constitute the current staff are not sufficient for running the beamline in the most efficient manner. The permanent staffing level should be increased to include three beamline scientists. A technician is urgently needed. After those additions to the staff, the panel then further recommends an appointment of at least one PhD student and/or post doc. The beamline has a proven record of being an excellent training ground for PhD students and young scientists.