

### Beamline Review Panel BM08

The panel would like to congratulate the GILDA staff for the remarkable quality and quantity of science that the beamline has delivered over the reviewed period of operations despite the challenging landscape for funding and staffing encountered during this period. The recommendations given by the previous panel review was partially fulfilled. The lack of funding did not permit the implementation of the recommended refurbishment of the optics. An improvement of fluorescence detection was found by the staff through taking equipment from Hasylab. It was regrettable that lack of resources resulted in the suspension of the diffraction capabilities of the beamline in 2012 together with the suspension of the operation of the beamline for Italian users through the CRG route for one year. Despite this "black" period, the scientific efficiency (ratio of publications to scheduled experiments) significantly increased compared to the previous review period and new scientific communities (cultural heritage and environment) have been attracted giving new opportunities for the renewal of the future science programme at GILDA.

The panel was very pleased to see the incorporation of GILDA into the CNR-IOM Italian research centre which provides a stable environment for operation, support and development. This involvement has also consolidated support from the Italian Research Ministry and resulted in a guaranteed investment of at least 1 M€ in the next two years and potentially a further 0.5 M€ in 2016.

#### **Summary of recommendations**

With the new investment from the Italian agency, a significant refurbishment of the GILDA beamline optics is now possible and we recommend the following priorities:

1. The 1<sup>st</sup> development priority is the purchase of a new monochromator with state of the art technology that is consistent with the level required by the future upgrade of the ESRF machine.
2. The installation of a new collimating mirror with appropriate mechanics and cooling to deal with the new source will optimize the energy resolution and flux available for X-ray Absorption spectroscopy. This is the 2<sup>nd</sup> priority.
3. For further increase of the flux and stability at the sample position that is necessary for planned scientific cases (2D mapping, ReflEXAFS, etc...), we recommend the installation of a second toroidal focusing mirror downstream of the monochromator as the 3<sup>rd</sup> priority.

These three recommendations are essential for a successful operation of GILDA in the coming years. The framework of the guaranteed budget expected in the next two years should cover this *mandatory* refurbishment of the optics.

The panel encourages the beamline team to continue the expansion of, and diversity in the user community to enhance the opportunities for long-term development projects. This will provide the necessary scientific cases to improve the end station with new ancillary equipment, cells and detectors. In particular high count rate fluorescence detection is a significant pressing need that is common to the majority of the research fields.

Following the first phase of the new investment in the beamline, further sustained investment from the Italian agencies is strongly encouraged. We recommend that this should take the form of:

4. The purchase of a new multi-element fluorescence detector and associated data acquisition electronics as the top priority.
5. If sufficient funding can be obtained, we then strongly recommend a significant refurbishment of the end-station including (i) a flexible sample space suitable for multi-technique measurements such as XAS+XRD, as well as *in-situ* or *operando* studies, (ii) a reliable sample positioning and manipulation system to facilitate mapping and grazing incidence experiments, (iii) the possibility to include specialist beam conditioning equipment (polycapillary optics)

It is important to note that the successful implementation of these two investment phases will at the very least require additional “peak load” technical staffing support during installation and commissioning periods, whilst the subsequent beamline operations will require additional scientific staff support if efficient operations are to be achieved. Specifically we would recommend the recruitment of first a technician and then an additional scientist.

## Introduction

The General Purpose Italian Line for Diffraction and Absorption (GILDA) is a multi-purpose high energy XAS and XRD beamline installed on a bending magnet source (BM08B) of the ERSF and operating for the Italian community since 1994. After 20 years of operation, no significant refurbishment of the optics has been undertaken. This critical issue was already identified during the previous review panel meeting. Due to the difficult financial situation throughout Europe and Italy during the 2009-2013 period it was not possible for the beamline to implement many of the recommendations of the previous review committee. Despite this lack of investment, the GILDA team should be applauded for maintaining the high level of research undertaken in many fields of research. The statistics show that the beamline has been highly productive in terms of the number of publications (more than one per experiment in the evaluation period) in leading international journals. A limited number of new technical developments were carried out by the staff in order to attract new communities, for example operando voltammetric measurements and 2D X-ray fluorescence mapping studies. This latter capability is particularly beneficial for the cultural heritage and environmental science communities working with highly heterogeneous samples on sub-millimeter length scales. The announcement of new funding from the Italian agency is a very positive development that was strongly welcomed by the review panel. This should ensure that the Italian community will continue to have access facilities available at the national low energy synchrotron radiation facility, ELETTRA, in Trieste.

## Research by Users

This review period has seen significant evolution of the research community that makes use of GILDA. In the previous review period, the majority of research came from studies in solid state physics, earth science and chemistry/physical chemistry. In this review period, there has been significant growth in studies coming from the biological science, environmental science and cultural heritage communities. The results and publications continue to be quantitatively and qualitatively on a very high level with a slight, but significant increase in the quality of the publications measured by average impact factor.

Several experiments have been noted to be outstanding, these include: (i) the study of the interaction of cisplatin with human superoxide dismutase (Banci), published in JACS (2012), which is important for understanding the biomedical behaviour of cisplatin in the treatment of amyotrophic lateral sclerosis, (ii) the structural characterisation of a high affinity mononuclear site in the copper(II)- $\alpha$ -synuclein complex (Bortolus), published in JACS (2010), which is a factor that plays a role in our understanding of Parkinson's Disease, (iii) the surface science approach to the solid-liquid interface: surface dependent precipitation of Ni(OH)<sub>2</sub> on  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> surfaces (Tougeriti), published in Angew. Chem. Int. Ed. Eng. (2012), which was a new approach for investigating catalysts.

The review panel found it pleasing to see that the research programme in semi-conductor systems continues to be an active area of research on the beamline, although now the activity has transferred from the beamline staff to the user community.

## Research by Staff

Research highlights coming from the activities of the GILDA staff include (i) the work on the enhancement of the Er<sup>3+</sup> luminescence in Er-doped silicate by a few-atom metal aggregates (Maurizio), published in PRB (2011), where co-doping a rare-earth containing glass sample with small clusters of gold atoms was found to induce a non-linear enhancement of the rare-earth element luminescence, and (ii) the discoloration of the smalt pigment: experimental studies and *ab-initio* calculations (Cianchetta), published

in JAAS (2012), which identified the underlying cause of the discoloration of the smalt pigment used in Renaissance paintings.

In particular, the interest of the local staff in cultural heritage investigations is strongly supported by the review panel and we recommend that they continue to pursue this kind of activity in the future. This work has stimulated technical developments at GILDA involving 2D-mapping of large samples.

### **Technical Status of the Beamline**

As detailed in the introduction, the review panel clearly recognise that the lack of investment in the past decade has severely limited the present competitiveness of GILDA compared to other comparable beamlines at third generation sources.

### **Future Technical developments**

The panel advise that the comprehensive technical design required for the major refurbishment of the optics be initiated as soon as possible. This is essential as the re-development funds will be made available by the Italian funding agency in the 2014 and 2015 financial years. This plan is also needed to underpin the renegotiation of the CRG agreement with the ESRF at the end of 2014.

The key elements of the optics refurbishment are:

- (a) The purchase of a new spectroscopy grade monochromator capable of operating in the 4keV to 50keV range, and matched to the enhanced requirements of the new X-ray source that will be delivered by the ESRF machine upgrade, finalized in 2019.
- (b) The purchase of a new collimating mirror to match the source to the monochromator to ensure spectroscopic energy resolution and flux throughput.
- (c) The purchase of a new toroidal focussing mirror to deliver an intense and stable beam to the sample position.

Each of the new optical components must be designed taking into consideration the parameters of the new X-ray source, and consequently the review panel recommend close liaison between the beamline staff and the ESRF machine development team.

A second phase of investment should then focus on enhancing the user end-stations. Discussion with the user community could be fruitful to establish the needs and priority order of the developments. At the current time, the panel advises investment in the purchase of a new multi-element fluorescence detector and associate electronics, as a common theme seen in the existing science programme is the study of dilute systems.

Subject to the delivery of the upgrade funds, the panel recommends that Phase 1 of the development programme be completed by the end of 2016, and Phase 2 be implemented as soon as possible thereafter.

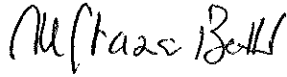





Long term investment in the experimental research infrastructure is an important factor if the beamline is to remain competitive. The staff are strongly advised to explore a range of collaborative activities with the user community, with the aim of attracting further investment through long-term projects.

**Staff**

The realisation of the recommended refurbishment programme will require additional staff resources to be made available. Phase 1 of the refurbishment programme will require additional technical support, in particular at "peak load" times (installation and commissioning of new optical components). Ideally, the provision of the additional staff required for these installations will be through coordination with the CNR-IOM framework.

Anticipating the growth of the user community after the beamline upgrade the panel strongly recommend that additional resident technical and scientific staff are employed to support operations. A particular need is the provision of routine technical support for integration of experimental setups to the beamline and support for small scale developments and maintenance.

**Signatures:**

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