A microprobe-XRF Beamline on Indus-2 Synchrotron Light Source

M. K. Tiwari1, S. R. Kane, A. K. Sinha, C. K. Garg, A. K. Singh, P. Gupta, S. R. Garg, G. S. Lodha and S. K. Deb

X-ray Optics Section, Indus Synchrotrons Utilization Division, Raja Ramanna Centre for Advanced Technology, Indore 452 013 India

1Email: mktiwari@rrcat.gov.in

Abstract. A microfocus x-ray fluorescence (XRF) beamline has been setup on Indus-2 synchrotron light source. The beamline works in the x-ray energy range of 4–20 keV. The optics of the beamline comprises of a Si(111) double crystal monochromator for energy tunability and a Kirkpatrick-Baez (KB) based grazing incidence focusing optics. Microprobe XRF scanning over a region of the sample is possible using a 5-axis sample scanning stage. The beamline provides an energy resolution ~ 10⁻³–10⁻⁴ with a photon flux density of the order of ~ 10⁸ ph/sec./mm²/100mA for the collimated unfocused beam. Measured performance, various attractive features and some initial commissioning results are presented.

1. Introduction

X-ray fluorescence (XRF) spectroscopy is a powerful “non-destructive” technique for the elemental analysis of materials at the micro and trace level. The technique finds several applications in a variety of fields viz. geology, archaeology, biomedical science and material science etc. [1-4]. Apart from the research applications, the XRF technique has potential usages in the industry especially in maintaining the quality control of ultra pure grade chemical reagents and products.

Considering several advantages of the synchrotron based XRF technique [5-6] and to fulfil the requirements of Indian research groups, we have built a microfocus XRF beamline (BL-16) on Indus-2 synchrotron light source. BL-16 beamline has been installed on the bending magnet source. It has been designed to work in the photon energy range of 4 – 20 keV. The beamline provides either microfocused or collimated monochromatic x-ray beams at the experimental station. The beamline enables a wide range of measurement capabilities which include micro-probe XRF examination of a specimen for spatial distribution of elements, energy dispersive x-ray fluorescence (EDXRF) analysis and total reflection x-ray fluorescence (TXRF) characterization of materials at ppb (parts per billion) levels for a short spectra acquisition time. In addition to the elemental mapping, BL-16 beamline also allows a user to perform other modes of XRF characterization, viz; grazing incidence x-ray fluorescence (GIXRF) analysis, chemical speciation, near-edge absorption spectroscopy and x-ray reflectivity characterization of thin layered materials etc.

BL-16 beamline started in user operation mode from mid 2011. Since then it has been used for a variety of user based research applications. We present design specifications, various salient features and some commissioning results obtained using the x-ray microfocus fluorescence beamline of Indus-2 source.
2. Beamline Details

The µ-probe XRF beamline (BL-16) has been installed on the 5º port of bending magnet. The optics of the BL-16 beamline comprises of a double crystal monochromator (DCM) with Si(111) symmetric and asymmetric crystals (mounted side-by-side), a Kirkpatrick-Baez (KB) focusing optics and the combination of slits in order to reduce the scattered x-ray background reaching the experimental station which usually emanates from different optical elements of the beamline. These slits also help in improving the collimation of the x-ray beam. The DCM is placed ~ 19m from the source and the KB focusing optics is placed ~ 4.7m apart from the DCM. The side-by-side mounting of the Si(111) symmetric and asymmetric crystals offers an advantage to use the DCM optics either in energy resolution mode or high flux mode with a minimal setup time. The KB focusing optics (Xradia, USA) comprises of a pairs of Pt coated (25nm) elliptical bendable mirrors. The measured r.m.s. slope errors (tangential) for the two elliptical mirrors were found to be better than 1.4 microrad whereas r.m.s. surface roughness was found to be ~ 0.3nm. The experimental stations of BL-16 beamline consist of a 5-axis sample manipulator for microprobe XRF–scanning applications and a 2-circle goniometer for total reflection x-ray fluorescence and grazing incidence x-ray fluorescence measurements for thin layered materials characterization. In addition, an optical microscope was employed for alignment of a sample in the x-ray beam path. Various detectors (Ionization chamber, Photodiode, single element SDD detector) are also available on the BL-16 beamline that makes possible to record high quality x-ray fluorescence data. Fig. 1a shows a schematic layout of the BL-16 beamline whereas Fig. 1b depicts the photographs for the beamline radiation shielding hutch and experimental station (inset). The beamline radiation shielding hutch and the personal safety interlock system, allow the safe operation of the beamline. The beamline operates under high vacuum condition (vacuum ~ 1×10⁻⁶ mbar).

![Figure 1A](image1a.png)

**FIGURE 1A:** A schematic layout of the microfocus XRF beamline showing its major optical elements.

![Figure 1B](image1b.png)

**FIGURE 1B:** Photographs of the BL-16 beamline radiation shielding hutch and experimental station (inset).
3. Results and Discussions
For precise determination of focused beam dimensions, the beam was scanned with a step of 0.5µm using a knife edge setup consisting of a 200 µm diameter gold wire mounted in the cross geometry and followed by a Si photodiode detector at the upstream of the KB optics. The measured vertical and horizontal profiles of the microfocused beam are shown in Fig. 2. The derivatives of the vertical and horizontal scan profiles provide a focus beam dimension of ~ 4.3µm (v) × 7.5µm (h). The measured photon flux in the collimated beam was found to be ~ 10^6 ph/sec./mm^2/100mA at 10 keV x-rays whereas for microfocus beam it was found to be one order of magnitude less compared to collimated beam.

**FIGURE 2:** Measured vertical (A) and horizontal (B) profiles of the microfocused beam obtained using a knife edge scan setup giving a focus beam of size ~ 4.3µm (v) × 7.5µm (h). The open circles are the measured data whereas solid lines are the fitted Gaussian profiles.

Fig. 3A shows measured EDXRF spectrum of a standard reference sample obtained from National Institute of Standards and Technology, USA (NIST-610) containing several elements in a glass matrix. Almost all trace elements have been detected in the NIST sample. The best minimum detection sensitivity of ~ 200ppb to 3ppm could be achieved for elements of atomic number z ranging from 22 to 82. Fig. 3b shows the TXRF spectrum of a NIST-1640 (trace element in natural water).

**FIGURE 3A:** EDXRF spectrum from a NIST 610 (trace elements in glass matrix) at 15 keV energy.

**FIGURE 3B:** TXRF spectrum of a NIST 1640 (trace elements in natural water) at 15 keV energy. The spectral background increases at low energy region < 4keV, due to re-scattering effects of fluorescent and sample scattered x-rays in the detector element.
The micro fluorescence mapping capabilities of the BL-16 beamline have been examined by measuring a few test pattern structures (grid structures). Fig. 4 demonstrates one such example where a two-dimensional elemental fluorescence map of a Cu grid has been recorded. The map was generated by measuring the net area intensity of Cu-Kα fluorescence line. The observed dimensions of the Cu grid structure determined from the µ-fluorescence mapping measurements were found to match closely with the optical measurements that were done independently.

![Cu-Kα fluorescence Image](image)

**FIGURE 4:** Measured fluorescence micrograph of a Cu grid structure using the BL-16 microfocus beam.

4. Conclusions
Design, construction and realization of a microprobe x-ray fluorescence beamline on the Indus-2 synchrotron radiation facility are presented. The BL-16 beamline allows various re-configurable operational modes (normal XRF, TXRF and µ-XRF modes) with a minimal setup time which enables wide range of experiments to be performed on it. We expect that with the existing beamline instrumentation it is possible to cater needs of a wide synchrotron user community in particular in the region of Indian sub-continent.

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