## T.1: Commissioning of Angle Dispersive X-ray Diffraction beamline on Indus-2

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The Indus-2 [1] is a 2.5 GeV Synchrotron radiation source at Raja Ramanna Centre for Advanced Technology (RRCAT), Indore, emitting radiation, from far infrared to hard x-ray with critical energy at 6.2 keV. Currently it is being operated at 2 GeV and three beamlines have been commissioned and operational, while several others are at different stages of installation. The Angle Dispersive X-ray Diffraction (ADXRD) beamline (BL-12) is amongst the first to become operational on a bending magnet source. This will be used for carrying out structural characterization on a host of different types of materials.

The beamline has been designed indigenously and the optics consists of an adaptive (bendable) optics for focusing the photon beam in both sagittal as well as meridional planes. The polychromatic x-ray from the synchrotron will be monochromatized using double crystal monochromator (DCM) based on two Si (311) crystals in (+, -) orientation. The beamline can be operated in both collimated as well as focused modes. In collimated mode, the beam size is large but the energy resolution ($E/\Delta E$) is $\approx 10^4$ (at 10 keV), whereas, in focused (high flux) mode the energy resolution is moderately lower. We can switch between the modes using bendable optics. The experimental hutch consists of two experimental stations in tandem. Station A has a six circle diffractometer and sodium iodide (NaI) scintillation detector for single crystal diffraction. Experimental station B consists of an Image plate area detector for x-ray diffraction on powder/polycrystalline samples. The beamline is a multipurpose one and various types of experiments like wide angle x-ray diffraction on single crystal and polycrystalline samples, hard x-ray reflectivity (XRR) and x-ray fluorescence (XRF) can be performed. This newsletter describes details of design, commissioning and validation of the beamline. We also discuss details of photon beam characterization and a few typical results on wide angle x-ray diffraction on single crystal and powder samples are given.

### Beamline and Experimental Station

#### Beamline

The beamline is installed on the $10^0$ port of bending magnet (BM 5) of Indus-2. The rms electron size at the center of the bending magnet at this port is 0.187 mm x 0.190 mm (H x V). Vertical and the horizontal divergences at this port are

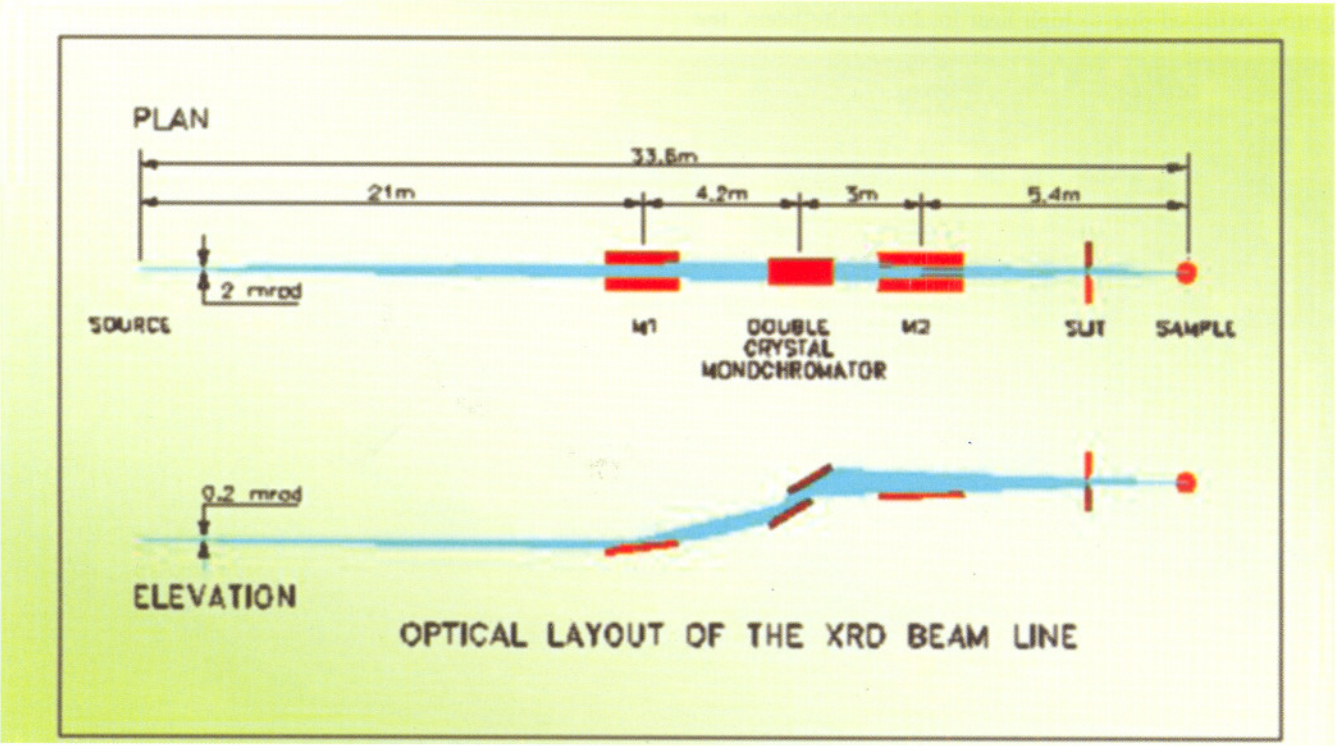


***Fig. T.1.1:** Plan and elevation of the optical elements used for estimation of the performance of the beamline using ray tracing program 'RAY'*

0.29 and 0.05 mrad, respectively. Photon size at the tangent point is estimated to be approximately 0.5 mm (H) × 0.5 mm (V). The beamline acceptance is 2 mrad (H) × 0.15 mrad (V) and the photon energy range is 5-25 keV. The optical layout of the beamline is shown in Fig. T.1.1. The beamline optics consists of a plane, bendable Si mirror (M1) coated with about 500 Å thick Pt layer. The mirror is bendable in the form of a meridional cylinder and is used to collimate/focus photon beam at the experimental station. The monochromatisation of the beam is done using Si (311) based DCM, with bendable second crystal in the sagittal direction. Thus the DCM serves dual purpose of monochromatisation as well as focusing the photon beam in the sagittal direction. A combination of two translation motions, along and perpendicular to the photon beam propagation direction, to the second crystal ensures a fixed exit of beam, when the Bragg angle is changed from $49.2^0$ to $8.7^0$ for 5 keV to 25 keV operations, respectively. The DCM is designed for a fixed exit of 35 mm. Si (311) crystal has intrinsic Darwin width of 6" in comparison to 11" for Si (111) crystals, at 5 keV. Therefore the monochromator has a higher energy resolution than the monochromator based on Si (111) pair. Finally a post mirror (not integrated in the beamline at present), similar to M1 is used to focus the monochromatic beam in the meridional direction. The absorption in air of hard x-ray radiation is not significant, but scattering of lower energy spectrum in ambient air is high, leading to loss of photon. Also, to save optical elements from getting oxidized due to high heat load of white beam, the beamline is operated in high vacuum ($<10^{-6}$ Torr). The experimental station, however, is in air. Fig.T.1.2 shows a photograph of the beamline. The photograph of the beamline was taken before the erection of radiation hutch.

In addition to the optical components, the beamline consists of wire and beam position monitors for accurate determination of beam position, slits and apertures for definition of photon beam and water cooled fluorescent screen along with CCD camera, for beam viewing. The vacuum is terminated using an un-cooled Be window of 200 μm thickness.

**Experimental Station**

The primary aim of the beamline is to perform high resolution x-ray diffraction experiments on polycrystalline and single crystal samples. So to facilitate these goals in most optimum way, we have two experimental stations in tandem. The first experimental station consists of a six circle diffractometer (Huber model 5020) and a NaI scintillation detector. Four out of six circles are the $\theta$, $\omega$ ($2\theta$), $\phi$ and $\chi$ (Eulerian cradle). $\theta$ and $\omega$ ($2\theta$) circles are for the Bragg scan and contain the sample and the detector respectively. For Bragg scan, the sample stage ($\theta$ circle) rotate by an angle $\theta$ and the $\omega(2\theta)$ stage rotate by an angle $2\theta$. The Eulerian cradle consists of circles $\varphi$ and $\chi$ to give yaw and roll manipulations

*Fig. T.1.1: Photograph of the beamline (taken before the hutch was erected), shows the DCM an the diffractometer* to the sample, respectively. These four circles form the main diffractometer. The rest two circles are in the detector arm to place analyzer crystal in the diffracted beam. This improves the resolution in reciprocal space and is used in the case of high resolution single crystal work.

The second experimental station consists of 345 mm diameter image plate area detector (mar 345) and a desktop beamline (dtb) consisting of a pair of ionization chambers and two slits. This experimental station is primarily used for x-ray diffraction measurements on powder samples in either transmission or reflection modes. It may be noted that the x-ray beam can be focused at the sample kept at either of the experimental stations because we use adaptive focusing using bendable optics in both meridional as well as sagittal planes.

**Simulation of Beamline Performance**

Simulation of the beamline performance has been done using the ray tracing program 'RAY' [2]. The ray tracing program requires the following steps:

a) Generation of source and calculation of heat load.
b) Start with an optical layout
c) Judicious decision on surface errors like rms roughness, flatness, slope errors etc. on optical elements. Very tight specifications come with high cost and may not improve the performance of the beamline, proportionally.
d) Calculation of slope errors on optical elements because of heat load absorbed on them and putting the data in ray tracing program
e) Ray tracing and evaluation of beamline performance. Flux, energy resolution and beam size are estimated in this step and are compared with the desired values.
f) If the estimated values are inferior to the desired values, the optical design is modified and ray tracing is performed again, till the performance is acceptable.

In table I, we summarize the design parameters (desired for the experiments) of photon beam at the experimental station with which design of the beamline was carried out.

***Table I: Design parameters of photon beam at 10 keV***

| Photon beam parameters at the experimental station | Value |
|---|---|
| Flux (Photon/s) | $10^{10}$ |
| Energy resolution ($E/\Delta E$) | $10^4$ |
| Photon beam size ($H \times V$) | 1.0 mm × 0.5 mm |
| Angular resolution (Arc Sec) | 20 |
| Polarization | No specific requirement |
| Energy range (keV) | 5-25 keV |

Iterations are done in the ray tracing program to achieve the required design parameters. In addition to the manufacturer's errors (rms roughness and slope errors) in optical elements; thermal deformations are given in terms of thermal bump height and FWHM of thermal strain. In the case of pre-mirror, the bump height of 0.4 μm and FWHM of 200 mm was estimated for bending magnet source, using finite element software, ANSYS [3]. Thermal deformation is also significant in the case of the first crystal and the deformation values are Bragg angle dependent. Typically at 15 keV the deformation is maximum. The slope error due to thermal deformation is less for side cooling of crystal, compared to bottom cooling. The details are published elsewhere [4].

Fig. T.1.3 shows the spot diagram indicating intensity distribution for 10 keV photons. This gives the size the beam and transmission efficiency of the proposed design. Thus the size and flux (photons per sec at the experimental station) can be estimated from this figure. The size of the photon beam source at the tangent point is estimated to be 500 μm x 500μm.

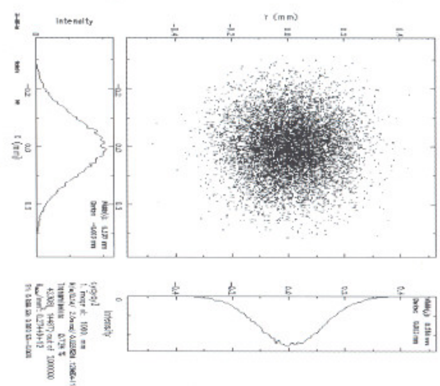

*Fig. T.1. 3: Spot size obtained using ray tracing. The beam size is* 500 μm *(H)* x 500 μm *(V).*

Table II shows final simulated beamline parameters at representative energy (10keV). More details about the beamline design are given elsewhere [5].

***Table II: Comparison between the design values and the measured values of beamline parameters***

| Parameter | designed/ simulated value | Measured | Remarks Value |
|---|---|---|---|
| Spectral Range | 5-25 keV | 5-25 keV | |
| Flux | $1\times10^{10}$ ph/s | $8\times10^{8}$ ph /s | See text |
| Resolution ($E/\Delta E$) | 10,000 | 4000 | See text |
| Beam size ($H \times V$) | 1.0 mm ×0.5 mm | 0.6 mm × 0.7 mm | |
| Angular Resolution | 20" | 17" | discussed later in this section |

**Results and Discussion**

Here we present some representative results obtained during the commissioning of the beamline. The beamline has been used for a) x-ray diffraction studies on single crystal and powder samples and also for b) surface and interface characterization of thin films and determination of their optical constants in hard x-ray region using x-ray reflectivity measurements. First, we present the results related to characterization of photon beam parameters at the experimental station. Representative measurements on various applications of the beamline are presented later in the section.

## Characterization of Photon Beam at the Experimental Station

After commissioning of the beamline, detailed measurements were carried out to evaluated the performance of the beamline. Several vertical scans of a gold wire position monitor during one injection and over various injections reveal that the beam stability at the entrance of the beamline (after the frontend) is within 50 μm, which is acceptable for x-ray diffraction and other applications of the beamline. The beam size was estimated using of x-ray CCD camera (20X) and was found to be 0.6 mm (H) × 0.7 mm (V). The beam size was further confirmed by exposing yellow paper (A special paper sensitive to x-rays) in the beam.

The beam intensity (photon/s) was measured using a calibrated x-ray photodiode (deep depletion junction photodiode AXUV-100) having good quantum efficiency (0.126 A/Watt at 10 keV) in the spectral region of interest (5-25 keV). Fig. T.1.4 shows intensity (photons/Sec/100 mA) normalized to 100 mA of ring current as a function of photon energy for 2 GeV electron energy. We find maximum intensity of 8 x $10^8$ photons/s at around 11 keV, as compared to the design value of $10^{10}$ photon/s. The lower values are because of lower electron energy (2.0 GeV as compared to the design value of 2.5 GeV) and lower normalized current (100 mA against the design value of 300 mA).

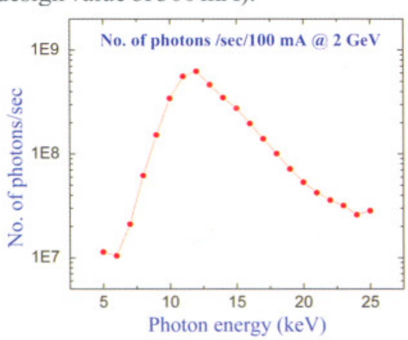


*Fig. T.1.4: Flux plotted as a function of photon energy. A maximum intensity around 10keV is expected.*

The spectral resolution was determined at an absorption edge, using 70 μm thick copper foil and NaI scintillation detector. Fig. T.1.5 shows transmission as a function of photon energy across the edge. In the inset, we show the first derivative of the curve. Maximum energy which gives the edge energy as 8819.8 eV, has ~159 eV shift from the actual energy of the copper K-edge (8978.9 eV). This shows that the DCM has an offset of 0.39° with respect to the beam. The peak (inset of Fig. T.1.5) was fitted with a Gaussian and FWHM was found to be 2.6 ± 0.1 eV. This gives spectral resolution (E/ΔE) of 4000 (as compared to designed value of 10,000). So, the measurement is used for the dual purpose of measuring the spectral resolution as well as energy calibration of DCM.

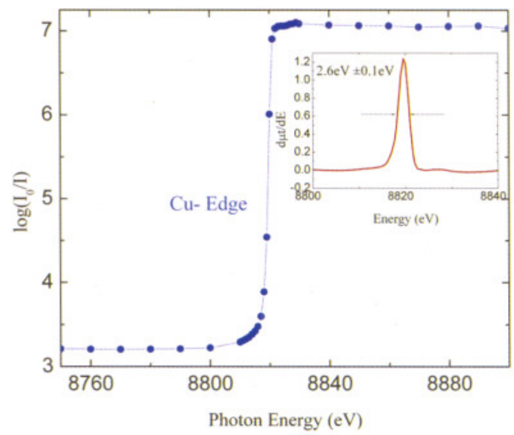


*Fig. T.1.5: Transmission as a function of photon energy across the Cu edge measured using Cu foil of thickness ~ 100 μm.*

The lower spectral resolution may be due to the following reasons:

(i) The photon beam has not been collimated in vertical direction and

(ii) Intrinsic width of copper (~1 eV) has not been subtracted from measured value of 2.6 eV.

Calibration of DCM energy was further verified by measuring x-ray diffraction on standard $LaB_6$ powder (to be discussed later). Table II shows a comparison of designed and measured photon beam parameters for the beamline.

## X-ray Diffraction Measurements

**Single Crystal Diffraction**

HOPG (Highly Oriented Pyrolytic Graphite) is an oriented crystal, and Fig.T.1.6 shows XRD pattern at 15 keV photon energy for HOPG oriented along (002). The peaks correspond to (002), (004), (006), (008), (0010) and (0012)

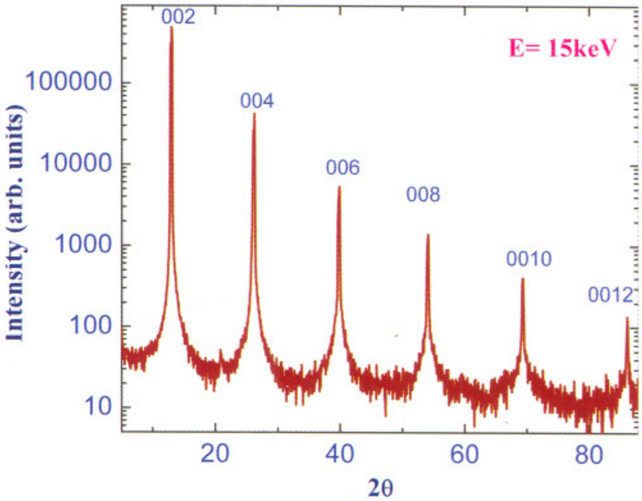


*Fig. T.1.6: XRD pattern of HOPG oriented along (002) direction. Higher order peaks up to (0012) are obtained*

planes are indexed. In order to measure the angular resolution, we have used a GaAs single crystal. Fig. T.1.7 shows the single crystal diffraction taken at 10.25 keV (uncalibrated). From gaussian fit to the measured curve, FWHM estimated to be ~17".

So, the instrumental angular resolution is <17". The angular resolution can be further reduced by using a single crystal in the diffracted beam.

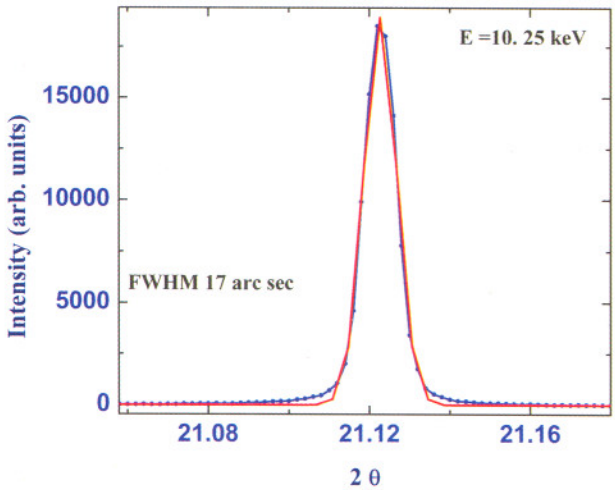


*Fig. T.1.7: Single crystal diffraction pattern of GaAs (111), gives the angular resolution*

## Powder (polycrystalline) Diffraction

X-ray diffraction of standard Lanthanum Hexaboride ($LaB_6$) sample was done in the θ-2θ geometry using diffractometer and also using the image plate area detector in transmission geometry. Fig. T.1.8 shows the measured X-ray diffraction pattern at 15.3 keV along with the simulated pattern at the same energy. Good agreement is found between the two results. X-ray diffraction on the same sample was recorded on area detector in transmission geometry. The pattern on the image plate at energy 14.34 keV is shown in Fig. T.1.9a. The recorded data is converted to intensity vs. 2θ graph using Fit2d software and is reproduced in Fig. T.1. 9b.

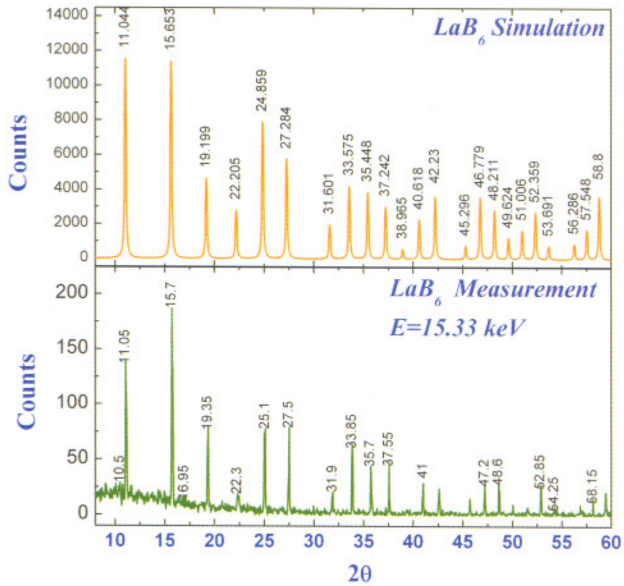


*Fig. T.1.8: Powder diffraction pattern of $LaB_6$ obtained using diffractometer and the simulated pattern at the same energy.*

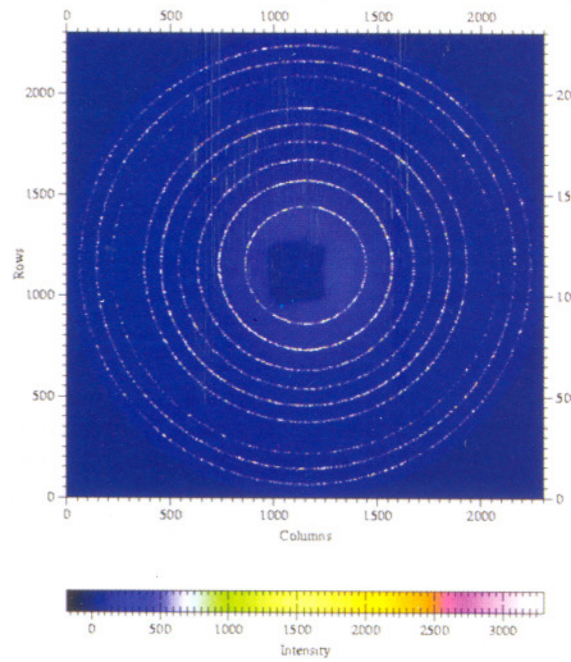


*Fig. T.1.9a: The diffraction pattern of $LaB_6$ powder.*

A good agreement between X-ray diffraction pattern with the diffraction pattern obtained using diffractometer (Fig. T.1.8) and simulated data using literature d values shows consistency of the measurements in two experimental stations. The advantage of using area detector for powdered samples are that the amount of sample needed is very small (few mgms) and also the measurement can be done fast (few minutes), as compared to diffractometer measurements; which takes about 1 hour.

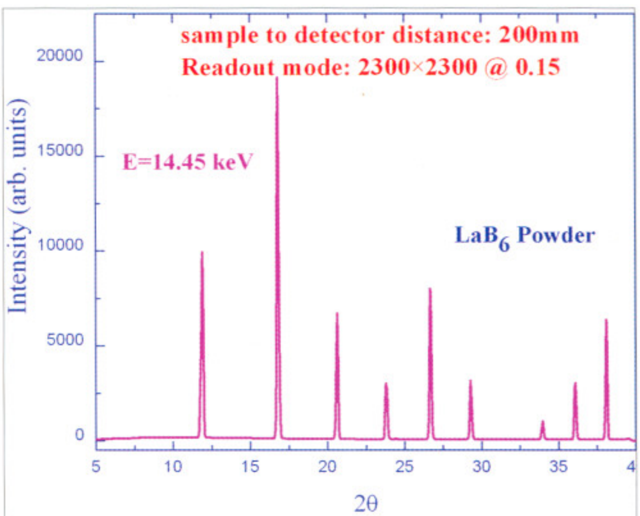


*Fig. T.1.9b: The diffraction pattern of $LaB_6$ powder (image plate data) using Fit2d software*

**Summary and Conclusions**

A high resolution X-ray diffraction beamline has been designed, developed and commissioned on Indus-2 Synchrotron Source by Indus Synchrotron Utilization division (ISUD), RRCAT. The beamline has been characterized with respect to stability of photon beam, photon flux, photon energy resolution, angular resolution etc. The measured parameters compares favorably with the designed parameters. The two experimental stations namely a) diffractometer and b) Image plate have been installed and aligned. The beamline is well equipped with detector and sample manipulation facilities. The beamline has been used for x-ray diffraction on single crystal and powder (polycrystalline) samples and hard x-ray reflectivity measurements. The beamline is a national facility and is available for scientists working in academic institutions, laboratories and industries for carrying out research. In this newsletter, we have reported typical results to demonstrate capabilities of the beamline.

## Acknowledgements:

Authours are thankful to G. M. Bhalerao, S. R. Kane and Soma Banik for their support.

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