Design, fabrication and testing of elliptical crystal bender for the EXAFS beam-line at INDUS-II synchrotron source

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Abstract. An extended X-ray absorption fine structure (EXAFS) beam-line for X-ray absorption studies using energy dispersive geometry and position sensitive detector is being developed for the INDUS-II synchrotron source. The optical design of the beam-line has been completed based on the working principle that a single crystal bent in the shape of an ellipse by a crystal bender would act as a dispersing as well as focusing element. The heart of the beam-line is the crystal bender which has been designed on the basis of the principle of four-point bending and has been fabricated indigenously. The crystal bender is capable of producing pre-defined elliptical curvature on a crystal surface by applying different couples at the two-ends of the crystal which has variable width along its length. The focusing property of the crystal bender has been tested using a laser source and has been compared with the theoretically simulated results.

Keywords. Synchrotron; EXAFS; elliptical crystal bender.

1. Introduction

The INDUS-II synchrotron source is a 2.5 GeV source which is presently being commissioned at the Centre for Advanced Technology, Indore (<http://www.cat.ernet.in>). An extended X-ray absorption fine structure (EXAFS) beam-line for X-ray absorption studies, using energy dispersive geometry and position sensitive detector, has been proposed to be installed at a bending magnet port of the INDUS-II Synchrotron source (Das *et al* 1999).

The EXAFS technique deals with measurements of fine structures in the X-ray absorption spectra above the absorption edge of different species of atoms in a material (Sayers *et al* 1971). With the advent of the modern bright synchrotron radiation sources, EXAFS measurement has emerged out to be the most powerful structure determination technique that can be applied to any type of material, e.g., amorphous solids, liquids, solution, gases, polymers and surfaces (Lytle 1999; Stern 2001; Tournus *et al* 2002; Hricovini *et al* 2003; Ascone *et al* 2003). The

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Figure 1. Schematic diagram showing the principle of action of the beam-line.

proposed EXAFS beam-line at INDUS-II will carry out the X-ray absorption measurements in energy dispersive mode involving no time-consuming scanning mechanism and thus can be applied to study *in-situ* fast and time-resolved processes (Iwasawa 2003).

A schematic diagram describing the principle of action of the beam-line is shown in figure 1. The basic idea is to use a single crystal (CC) bent in the shape of an ellipse in a particular fashion such that the source (S_0) and the sample positions (S_3) are situated at the two foci of the ellipse (Lee *et al* 1994). White synchrotron radiation from the source (S_0) will be made incident on the crystal and depending on the angle of incidence of the beam and its radius of curvature, the crystal will reflect a particular central energy (E_o) with a certain bandwidth (ΔE) and this spatially dispersed polychromatic radiation will then be focused at the sample position (S_3). The transmitted radiation from the sample will then be detected by a position-sensitive detector (D). Thus the energy dispersed absorption spectra of the sample over the whole band width (ΔE) around the central energy (E_o) will be recorded on the detector simultaneously. The proposed beam-line will cover the photon energy range of 5 keV to 20 keV by a bent Si(111) crystal having $2d$ value equal to $6.2709\text{\AA}$. The beam-line will provide band widths of the order of 0.3 keV, 1 keV and 2 keV at photon energies of 5 keV, 10 keV and 20 keV respectively. The average resolution at the detector has been estimated to be ~ 1 eV per channel (Das *et al* 1999).

The heart of the above beam-line is the crystal bender which would be used to generate the required elliptical curvature on the surface of the Si(111) crystal. Generally two types of mechanisms are used for bending of crystal to achieve sagittal (horizontal) focusing of the beam in Synchrotron beam-line optics. One method is to push the apex of an isosceles triangle-shaped crystal while keeping its base fixed (Batterman & Berman 1983; Flank *et al* 1983; Tolentino *et al* 1990). A perfect triangular crystal would give rise to a cylindrical curvature by this process while an elliptical shape can be approached by using a complicated profiled crystal. However, the more widely-used technique is the four-point bending mechanism where a rectangular crystal is held by two fixed inner rods and are pushed by two external moving

rods (Sparks *et al* 1982; Lee *et al* 1994). The advantage of such a mechanism is that the part between the inner rods would have the exactly desired curvature and it prevents crystal twisting in the other plane which is detrimental to Bragg reflection (Stephens *et al* 1993; Artemev *et al* 2001). An elliptical curvature on the surface of a crystal can be exactly achieved by this process.

In the present communication, the design, development and testing of an indigenous crystal bender has been described, which has been developed based on the four-point bending principle discussed above and will be used for sagittal focusing of the synchrotron beam in the EXAFS beam-line at INDUS-II.

2. Optical design of the beam-line

The detailed optical layout of the beam-line is shown in figure 2. The beam coming from the synchrotron source is first collimated by two copper blocks (K,K) kept at an angle of 15° with the beam path. The collimated beam with horizontal divergence of 1.5 mrad then falls on a Be window (B) of suitable thickness which help to cut-off the unwanted low-energy part from the continuum. The transmitted beam through the Be window falls on the Ta slit (S_1) which defines the final divergence of the synchrotron beam. The beam emerging from slit with required vertical and horizontal divergences falls on the crystal (CC). After reflection from the crystal the dispersed beam passes through the second slit (S_2). The slit (S_2) helps in shielding the scattered radiation so that the beam gets sharply focused on the sample (S_3). From the sample, the beam diverges further and finally gets detected at the CCD-type position sensitive detector (D). A plane mirror (M) is then used before the detector to cut-off the higher harmonics from the radiation diffracted by the Si crystal. W is the shielding wall that isolates the front-end of the beam-line from the rest portion.

Figure 2. Optical layout of the beam-line.

Table 1. EXAFS beam-line fixed parameters.

Parameter	Value
Crystal type	Si(111)
$2d$ value	6.2709 Å
Source to crystal distance (p)	20,000 mm
Horizontal beam divergence (U_m)	1.5 mrad

Some of the parameters of the beam-line which have been decided based on practical considerations, are listed in table 1. An Si single crystal with (111) orientation ($2d = 6.2709\text{Å}$) has been chosen as the polychromator since it has a very narrow Darwin width, low thermal expansion coefficient and high thermal conductivity. The source to Si crystal distance (p) has been fixed at 20,000 mm on practical considerations. A reasonable detector size of 25×25 mm has been chosen considering the resolution, cost and availability of the CCD type position-sensitive detectors and horizontal beam divergence (U_m) has been chosen to be 1.5 mrad taking into considerations the detector and crystal sizes and heat flux falling on the different components of the beam-line.

The parameters of the beam-line which are functions of the central energy of interest (E_o) are shown in table 2. The energy band widths (ΔE) at different central energies have been estimated considering practical values of the radius of curvature (R) of the crystal. The values of the different other parameters shown in table 2 are determined as follows.

The Bragg angle θ_0 for the central ray at the pole of the crystal is obtained from the central photon energy E_o or wavelength λ_0 from the well-known Bragg relation (James 1962):

$$\sin \theta_0 = \lambda_0/2d = 12.398/2dE_o(\text{keV}). \quad (1)$$

Since the source to crystal distance (p) and horizontal divergence (U_m) are fixed, the length of the illuminated portion of the crystal (l) corresponding to the central energy E_o is then determined by the simple geometrical relation:

$$U_m = (l/p) \sin \theta_0. \quad (2)$$

For simplicity in calculations, initially we have assumed the elliptical crystal to a spherical cylinder having a uniform radius of curvature (R_0). Since l is known, the radius of curvature (R_0) can be determined by the following relation for pre-specified value of the energy band width (ΔE) for all values of E_o (Lemonmer *et al* 1978):

$$\Delta E = E_o l \cot \theta_0 [(1/R_0 - \sin \theta_0/p)] \quad (3)$$

Table 2. EXAFS beam-line variable parameters.

Parameters	Values		
Photon energy (E_o)	5000 eV	10,000 eV	20,000 eV
Band pass (ΔE)	297 eV	1123 eV	2000 eV
Bragg angle (θ_0)	23.28°	11.40°	5.67°
Crystal length (l)	75.9 mm	151.8 mm	303.5 mm
Crystal radius (R_0)	2803 mm	6287 mm	26,550 mm
Crystal to sample distance (q)	570 mm	641 mm	1404 mm

Table 3. Parameters of the elliptical cylinder shaped crystal.

E_o (eV)	p (mm)	q (mm)	θ_0 (deg)	α (deg)	a (mm)	b (mm)	c (mm)
5,000	20,000	570	23.28	66.72	10285	1335.5	10198
10,000	20,000	641	11.40	78.60	10320	708.1	10296
20,000	20,000	1404	5.67	84.33	10702	524	10689

Since the values of p , θ_0 and R_0 are known, the crystal to sample distance (q) is then determined from (4) given below (Lemonmer *et al* 1978):

$$1/\sin \theta_o = R_0/2[(1/p + 1/q)]. \quad (4)$$

The above calculations have been done for various photon energies and the computed data corresponding to two extreme photon energies of 5 keV and 20 keV and at an intermediate photon energy of 10 keV are shown in table 2. The data presented in table 1 and table 2 have been used in the ray-tracing programme “SHADOW” (SHADOW GUI 1997) to check the focusing of the diffracted beam at the sample position and the available resolution at the detector position. The detailed results of this exercise have been reported elsewhere (Das *et al* 1999, 2002, 2003). The above calculations have been done first assuming the crystal to be bent in the shape of a spherical cylinder. However, as has been discussed earlier, for perfectly focussing the polychromatic beam at the sample position, the crystal has to be bent in the shape of an elliptical cylinder as shown in figure 1. The ray-tracing work has also been carried out assuming an elliptically bent crystal and the geometrical parameters of the ellipse, viz., the semi-major (a) and semi-minor (b) axes, as defined in figure 1, have been obtained by the ray-tracing analysis using the values of θ_0 , p and q for different central photon energies (E_o). The parameters of the ellipse thus obtained are shown in the table 3 for three different photon energies of 5 keV, 10 keV and 20 keV. Design of the crystal bender has been made in such a way that the above elliptical parameters can be generated on the surface of the crystal at different Bragg angles.

3. Mechanical design of the crystal bender

As has been mentioned earlier, the crystal bender that has been described in the present communication, works on the principle of four-point bending. The schematic mechanical design of this bender is shown in figure 3. In this bender the crystal is held in proper position, pivoted at two points (P,P) and is pushed from the back at two other points (Q,Q) by a worm-shaft and worm-wheel arrangement. The mechanism gives rise to two couples at the two ends of the crystal. The worm wheels are driven by stepper motors to produce precision torques on the crystal.

For a crystal of length L and applied couples C_1 and C_2 at two ends, the bending of the crystal is given by the following Bernoulli–Euler equation (Padmore 1996):

$$EI \frac{d^2 y}{dx^2} = \frac{C_1 + C_2}{2} - \frac{C_1 - C_2}{L} x, \quad (5)$$

$$I = bh^3/12, \quad (6)$$

Figure 3. Mechanical design of the crystal bender.

where y is the displacement at any point x along the length of the crystal, E is Young's modulus, I is the section modulus or moment of inertia, b is the mechanical width of the crystal, and h is the thickness of the crystal.

The second derivative in (5) is the curvature of the ellipse and it is a function of the distance (x) of any point along the crystal from its centre ($x = 0$). Since C_1 and C_2 are independent of x , the quantity I has to be varied, either by changing the width b or thickness h to get the required elliptical shape of the bent crystal. In the present arrangement the crystal width b has been chosen to vary. The length of the crystal (L) has been estimated to be 410 mm and its thickness (h) and width at $x = 0$ (b_0) have been taken to be 3 mm and 30 mm respectively.

Assuming first that the crystal is bent in the shape of a spherical cylinder with constant radius of curvature R_0 , the values of the two couples can be found out from (5) as (Padmore 1996):

$$C_1 + C_2 = 2EI_0/R_0, \quad (7)$$

$$C_1 - C_2 = (3EI_0L \sin \alpha/2R_0)[(1/p) - (1/q)], \quad (8)$$

where R_0 is the radius of curvature, I_0 is the value of section modulus at $x = 0$, and $\alpha (= 90 - \theta_0)$ is the angle of incidence while the parameters p and q have been defined earlier in figure 1 as well as in tables 1 and 2.

Now, in our case (Ygural and Senster 1995),

$$E = 7000 \text{ kg/mm}^2 \text{ (for Si) and}$$

$$I_0 (= b_0 h^3 / 12) = 67.5 \text{ mm}^4.$$

For a mean energy (say of order 10 keV), as shown in table 2, radius of curvature at $x = 0$, i.e., $R_0 = 6287 \text{ mm}$.

Substituting all values, in (7 and 8) we have

$$C_1 + C_2 = 150.66,$$

$$C_1 - C_2 = -68.58.$$

Solving these two equations, we get

$$C_1 = 41.03 \text{ kg mm}, C_2 = 109.62 \text{ kg mm}$$

Since for an ellipse, radius of curvature varies at various points, the elliptical surface required corresponding to a certain energy (given in table 3) has been divided into a number of small segments so that each segment remains small enough to assume constant radius of curvature within that segment. Normals are drawn on the tangents at the two end sections of each segment and the intersection of these two normals gives the centre of curvature of that segment. The radial distance from this centre of curvature to the mid point of the segment gives the radius of curvature for that segment. Radius of curvature of each segment corresponding to the mean energy of 10 keV is given in table 4, where the 410 mm long crystal has been divided into several small segments, each of length 5 mm.

Putting in the values of C_1 and C_2 in Bernoulli's equation (5) and considering that

$$d^2y/dx^2 = 1/R(x), \text{ we have } b(x)/R(x) = 0.00478285 + 0.00001062x. \quad (9)$$

In the above equation, substituting for x and the corresponding $R(x)$ which has already been determined, variation in width $b(x)$ of the crystal is calculated. The $b(x)$ values for the 10 keV energy are also shown in table 4. The same calculations have been done for various other energies also and the $b(x)$ values have been determined. The typical shape of the Si crystal required for 10 keV energy is shown in figure 4. Taking into considerations the mechanical requirement of clamping the total crystal length has been taken to be 460 mm.

Software simulation of crystal bending has been done by finite element modelling and analysis using SDRC's I-DEAS Master Service Version-8 code. A finite element model of the Si crystal between the two points (Q,Q), as shown in figure 3, is created using thin shell elements of 3 mm thickness. Physical properties in the form of thickness and material properties such as modulus of elasticity and Poisson's ratio are supplied from the literature (Ygural & Senster 1995). To simulate the actual bending with application of couples, the appropriate boundary condition is applied. The nodes at the two edges are allowed to rotate about their own axes which are parallel to the plane of the crystal. To arrest the rigid body mode, movements of all nodes at the left edge are restrained in the X , Y and Z directions. The co-ordinate system convention assumed here is shown in figure 5. As the bending occurs, the right edge of the crystal moves towards the left edge, hence all the nodes of the right edge are allowed to move in the X -direction only. The results obtained for the deflection of crystal corresponding to 10 keV photon energy is shown in figure 5. Values of deflection at each node along the length of the crystal have been found and compared with the actual

Table 4. Radius of curvature and variation of width of crystal along length for 10 keV energy.

x (mm)	$R(x)$ (mm)	$b(x)$ (mm)	x (mm)	$R(x)$ (mm)	$b(x)$ (mm)
-205	9310	24.260	5	6202	29.992
-200	9233	24.549	10	6133	29.984
-195	9155	24.827	15	6063	29.964
-190	9077	25.098	20	5995	29.946
-185	9000	25.363	25	5926	29.916
-180	8927	25.617	30	5857	29.879
-175	8845	25.865	35	5789	29.839
-170	8768	26.106	40	5721	29.792
-165	8691	26.338	45	5654	29.744
-160	8615	26.565	50	5586	29.683
-155	8538	26.781	55	5519	29.620
-150	8462	26.992	60	5452	29.550
-145	8386	27.195	65	5386	29.478
-140	8310	27.390	70	5319	29.394
-135	8234	27.576	75	5253	29.308
-130	8159	27.758	80	5187	29.215
-125	8084	27.933	85	5121	29.115
-120	8009	28.099	90	5056	29.014
-115	7934	28.257	95	4991	28.906
-110	7859	28.407	100	4926	28.791
-105	7789	28.553	105	4861	28.669
-100	7711	28.691	110	4797	28.547
-95	7637	28.821	115	4733	28.417
-90	7563	28.943	120	4669	28.281
-85	7489	29.058	125	4606	28.144
-80	7416	29.168	130	4542	27.994
-75	7343	29.271	135	4479	27.843
-70	7270	29.366	140	4417	27.693
-65	7197	29.454	145	4354	27.529
-60	7124	29.533	150	4292	27.365
-55	7052	29.609	155	4230	27.194
-50	6980	29.677	160	4169	27.023
-45	6908	29.738	165	4107	26.839
-40	6836	29.791	170	4046	26.656
-35	6765	29.841	175	3986	26.472
-30	6694	29.883	180	3925	26.275
-25	6623	29.918	185	3865	26.079
-20	6552	29.945	190	3805	25.876
-15	6482	29.969	195	3746	25.674
-10	6411	29.982	200	3687	25.465
-5	6341	29.994	205	3626	25.235
0	6272	30.000

Figure 4. Shape of the crystal required to achieve the elliptic bending for 10 keV photon energy.

deflection required to achieve the elliptical shape. The results are found to agree well within acceptable limits. Figure 6 shows the stress distribution in the crystal in the bent condition corresponding to 10 keV photon energy. Maximum principal stress induced in the crystal is found to be $0.758 \text{ kgf mm}^{-2}$ which is less than the maximum allowable stress.

4. Testing of the crystal bender

Figure 7 shows the photograph of the crystal bender fabricated indigenously based on the above design. Two PC-controlled stepper motors are used to push the warm shafts for providing the required torques at the two ends of the crystals. The crystal bender has been tested both mechanically and optically. The set-up for optical testing of the crystal bender is shown

Figure 5. Deflection analysis of crystal (mm) for bending profile required for 10 keV photon energy. (The values of the deflection in mm are represented by the gray codes shown in the right-most portion of the diagram.)

Figure 6. Stress analysis of crystal (kgf mm^{-2}) for the bending profile required for 10 keV photon energy. (The values of the stress generated in the different portion of the crystal in kgf mm^{-2} , are represented by the gray codes shown in the right-most portion of the diagram.)

Figure 7. Photograph of the indigenously developed crystal bender.

Figure 8. Schematic diagram showing the arrangement for testing of crystal bender.

schematically in figure 8. Light from an He–Ne laser source has been focussed by a microscope objective on the pin hole of a spatial filter to simulate a synchrotron source. The light diverging from the spatial filter is then made incident upon the crystal mounted on the crystal bender which is kept at a distance of 20 m from the source. The crystal is elliptically bent to different radius of curvatures using different torques at the two ends of the crystal bender and the focus spot has been monitored on a ground glass plate as a function of grazing angle of incidence and the mean radius of curvature of the ellipse. It has been observed that the width of the best focus spot obtained is $300\text{ }\mu\text{m}$ at a distance of 641 mm from the centre of the crystal and at an grazing angle of incidence of 11.40° . This is equivalent to the configuration for Bragg reflection of 10 keV as can be found from table 2. Figure 9 shows the photograph of

Figure 9. Image spot at the focus position of the crystal: (a) Theoretical; (b) experimental.

the focus spot at the above condition along with the focus spot simulated theoretically (using the ray-tracing programme "SHADOW") under the same condition with a synchrotron beam. The difference between the widths of the simulated and experimental focal spots arises due to the fact that the latter spot is formed by specular reflection of the optical beam from a real surface while the former is generated assuming Bragg reflection of X-rays from crystal planes.

5. Conclusion

A crystal bender has been designed and fabricated indigenously for using in a dispersive EXAFS beam-line to be set up at INDUS-II synchrotron source. The bender will be used to bend Si(111) crystals in elliptical shape for sagittal focusing of the synchrotron beam on the sample. The function of the bender has been tested optically using a divergent laser beam. The focus spots obtained at the sample position were recorded on photographic plate and have been compared with the theoretically generated focus spots.

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