First Results from a Dispersive EXAFS beamline at Indus-2 SR facility

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Outline

• Introduction to Indus SR
• Beamline description
  – Basic working principle
  – Optical layout
  – Design procedure
  – Mechanical Layout
  – Experimental station
• Commissioning results
Schematic view of Indus complex
Indus-2 Parameter

Energy : 2.5 GeV
Current : 300mA
Field : 1.5 T (BM)
Circumference: 172.5m
Lifetime: 15 Hrs
$\lambda_c = 1.98 \, \text{Å} \ (6.23 \, \text{KeV})$
Beam size
$\sigma_x : 0.234 \, \text{mm}$
$\sigma_y : 0.237 \, \text{mm}$
RF frequency : 505.812 MHz

Indus-2 synchrotron spectrum

Beam Energy : 2.5 GeV
a: Bending Magnet
b: Wave Length Shifter

Photon Flux vs. Photon Energy (eV)
Status of Indus-2

Indus-2 operation at 2.5GeV

Beam Current [mA]

Beam Energy [MeV]

Time [Hrs]
Beamline Layout view of Indus-2

02: MI
04: PRC
07: XRL
13: GIS
09: EXAFS-scanning
14: PES
16: XRFμ
18: SAXS
37 BLs

♣ BL Under construction
Characteristics of ED XAFS BL at Indus-2

- Source: Bending magnet
- Energy range: 5-20keV
- Resolution: $10^{-4}$
- Band pass: 300ev to 2000eV
- Flux: $10^{12}$ photons/sec/1000eV
- Polychromator: Si(111)
- Detector: CCD(2k x 2k ; pixel: 13.5µ)
Principle of Action:

• Synchrotron Radiation is diffracted and focused on sample by a perfect crystal, bent elliptically such that the sample and the source are at focii of the ellipse. Transmitted intensity is detected by a position sensitive CCD detector.

<table>
<thead>
<tr>
<th>Energy (eV)</th>
<th>$\theta_o$ (deg)</th>
<th>a (mm)</th>
<th>b (mm)</th>
<th>c (mm)</th>
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<tbody>
<tr>
<td>5,000</td>
<td>23.28</td>
<td>10285</td>
<td>1335.5</td>
<td>10198</td>
</tr>
<tr>
<td>10,000</td>
<td>11.40</td>
<td>10320</td>
<td>708.1</td>
<td>10296</td>
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<tr>
<td>20,000</td>
<td>5.67</td>
<td>10702</td>
<td>524</td>
<td>10689</td>
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Optical Layout of EXAFS Beamline at INDUS-2

Top view:
- Shielding Wall
- Beam Aperture
- Slit System
- Pre-Mirror

Side view:
- Bent Crystal Polychromator
- Slit System
- Sample

Used for horizontal focusing –cum-dispersion

Used for vertical focusing –cum-higher harmonic rejection
Design Procedure

- Fixed parameters
- Variable geometrical parameters
- Ray tracing: check the performance
- Fixing the specs of optical components
- Simulation of heat load on each optical component
- Mechanical specs of different subsystem
  - Mirror mount, crystal bender et al
- Simulation of radiation dose
- Fixing specs of Hutch

# Fixed Parameters

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
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<tbody>
<tr>
<td>Crystal type</td>
<td>Si (111)</td>
</tr>
<tr>
<td>2d Value</td>
<td>6.2709 Å</td>
</tr>
<tr>
<td>Source to Crystal Distance (p)</td>
<td>20,000 mm</td>
</tr>
<tr>
<td>Detector length (L)</td>
<td>25 mm</td>
</tr>
<tr>
<td>Horizontal Beam Divergence (Uₘ)</td>
<td>1.5 mrad</td>
</tr>
<tr>
<td>Vertical Beam Divergence (Uₘ')</td>
<td>0.2 mrad</td>
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</table>

\[ \sin \theta_o = \frac{\lambda}{2d} = \frac{12.398}{2dE_o(keV)} \]

\[ U_m = \left( \frac{l}{p} \right) \sin \theta_o \]

\[ \Delta E = E_o l \cot \theta_o \left( \frac{1}{R} - \frac{\sin \theta_o}{p} \right) \]

\[ \frac{1}{\sin \theta_o} = R \left( \frac{1}{p} + \frac{1}{q} \right) \]

\[ U_{m'} = \left( \frac{l}{q} \right) \sin \theta_o \]

\[ r = \frac{L}{U_{m'}} \]

## Derived Parameters

<table>
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<th>5000</th>
<th>10,000</th>
<th>20,000</th>
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<tbody>
<tr>
<td>Photon Energy $E_o$ (eV)</td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>Band Pass $\Delta E$ (eV)</td>
<td>297</td>
<td>1123</td>
<td>2000</td>
</tr>
<tr>
<td>Bragg Angle ($\theta_o$)</td>
<td>23.28°</td>
<td>11.40°</td>
<td>5.67°</td>
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<tr>
<td>Crystal length $l$ (mm)</td>
<td>75.9</td>
<td>151.8</td>
<td>303.5</td>
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<tr>
<td>Crystal Radius $R$ (mm)</td>
<td>2803</td>
<td>6287</td>
<td>26,550</td>
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<tr>
<td>Crystal to Sample distance $q$ (mm)</td>
<td>570</td>
<td>641</td>
<td>1404</td>
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<tr>
<td>Sample to Detector Distance $r$ (mm)</td>
<td>475</td>
<td>534</td>
<td>1170</td>
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Ray tracing result

<table>
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<tr>
<th></th>
<th>5 keV</th>
<th>10 keV</th>
<th>20 keV</th>
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</thead>
<tbody>
<tr>
<td>Focal spot</td>
<td>20</td>
<td>40</td>
<td>50</td>
</tr>
<tr>
<td>Spatial resolution</td>
<td>0.3eV/pixel</td>
<td>1.1eV/pixel</td>
<td>2.1eV/pixel</td>
</tr>
<tr>
<td>at detector</td>
<td></td>
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</table>
Optical components

• Polychromator

• Pre-mirror:
Polychromator

**Crystal Bender**
Generates elliptical curvature on a profiled crystal of ½ m length

Mean Radius of Curvature:
2 m to 20 m (within +/-0.5%)

Indigenously built

Stepper motor driven shafts
Stroke: 0-12 mm (required 3-4 mm)
Resolution: 0.5 μm

Actual bender inside a vacuum chamber with Ga/In based cooling arrangement

460 mm long profiled Si (111) Crystal; Darwin width: 0.6 arc sec
Harmonic Rejection Mirror:

Substrate: Si
Coating: Rh
Radius: 1319 m
Size: 1 m × 30 mm
Surface Roughness: 3 Å r.m.s.

Cylindrical Mirror
Side cooled, gravity compensated

Reflectivity vs. photon energy (eV)

A: 0.2°
B: 0.4°
C: 0.6°
Indigenously built Mirror chamber with mirror mount

Movement resolution of mount
Shafts inside UHV : 1µm
Mechanical layout of EXAFS BL at Indus-2

- Front End
- Beam Aperture Chamber
- 1st Beam View Chamber
- Precision Slit System
- Mirror Chamber
- 2nd Beam View Chamber
- Bender Chamber
- Sample Mount
- Beam-1
- Beam-2
- Beam-3
- Shielding wall
- Experimental station
- Sample stage
Commissioning stages

• Alignment of Front-end with SR
• Integrating the front end with beamline and achieving vacuum in subsystems
• Optical Alignment of with SR using BV systems and CCD detector
• Checking the SR foot prints at different locations of the beamline
• Checking the performance of the bender
• Recording absorption of standard samples to check the spatial resolution and band pass energy
Illumination on crystal for 20 keV setting

Beam reflected from mirror which is used

Direct Beam which is blocked

275 mm
Beam spot at sample position

200µm

500µm
Absorption Spectra of Mo Foil **
20 keV Setting
12.03.08@ 6 ma/2 GeV

**Complete Spectrum Taken In 1 Second
Mo-Nb

Mo & Nb absorption edge obtained at 20 keV setting

Nb Absorption edge
18987 eV
Channel No: 1577

Mo Absorption Edge
20000 eV
Channel No: 1067

Calibration: 1.98 eV/channel
MoMoO$_3$

Reported Difference in $E_0$: $\sim$9 eV

Chemical shift

Reported Value: 5 eV


$E_0$(Athena half Edge step) = 18999 eV

$E_0$(Athena Half edge step) = 19005 eV

$\mu$

Enerav (eV)

18950 18975 19000 19025 19050 19075 19100

$\text{Nb}$

$\text{Nb}_2\text{O}_5$
EXAFS Spectra of Bi$_2$O$_3$ taken at L$_{\text{III}}$ edge

- 13keV setting
- 0.6eV/pixel (Pb-L$_{\text{III}}$ and Bi-L$_{\text{III}}$)

After Data Reduction & Fourier Transform

Bi-O nearest distance: 2.23Å
Agrees well with reported value

Energy (eV)

$\mu t$

$r (\text{Å})$

$\chi(r)^2$
EXAFS MEASUREMENTS

- U(VI) sorbed on alumina and kolinite
- Crystalline Pb$_5$Ge$_3$O$_{11}$
Sorption of U(VI) by alumina and kaolinite at varying pH has been studied by X-ray absorption Spectroscopy. The absorption intensity was found to increase with increasing pH of the suspension.
X-ray absorption spectrum of U(VI) sorbed onto Alumina

Fourier transform of X-ray absorption spectrum of U(VI) sorbed onto Alumina-3 (pH=7)
Conclusion:

Results from newly developed EXAFS beamline at Indus-2 are found to be in good agreement with those reported in literature which implies the satisfactory performance of the beamline.

Further efforts are being made to improve the signal to noise ratio.
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