Angle Dispersive X-ray Diffraction Beamline (BL-12) for Materials Research

A. K. Sinha (anil@rrcat.gov.in)

Indus Synchrotron Utilisation Division (ISUD)
Raja Ramanna Center for Advanced technology
Indore, India

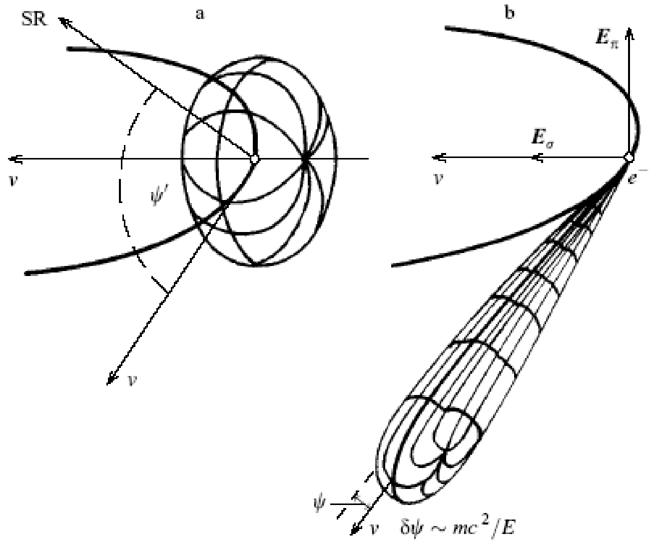
Acknowledgements

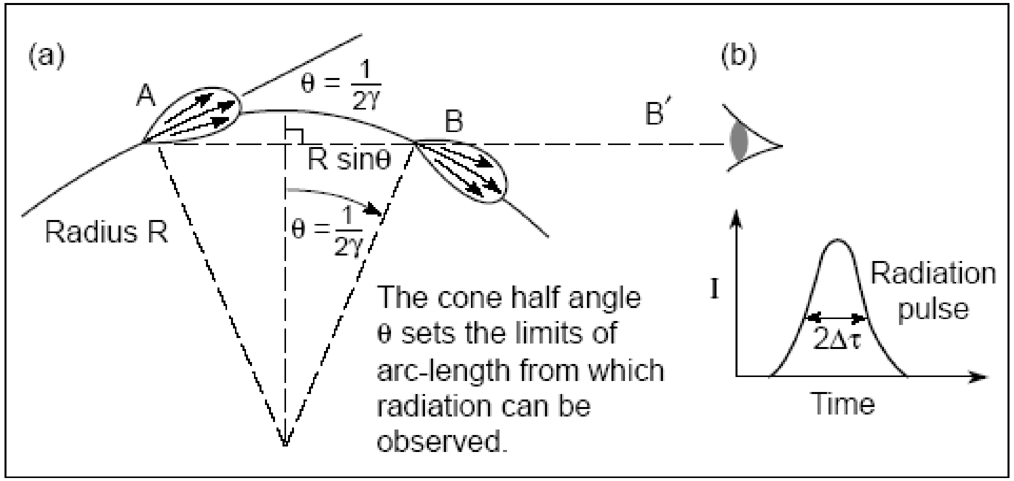
ISUD Colleagues

Plan of Talk

1. Introduction (Creating Synchrotron Radiation)
2. Angle Dispersive X-ray Diffraction beamline
3. Applications of the beamline for Materials Research (Some Results)

Creating Synchrotron Radiation



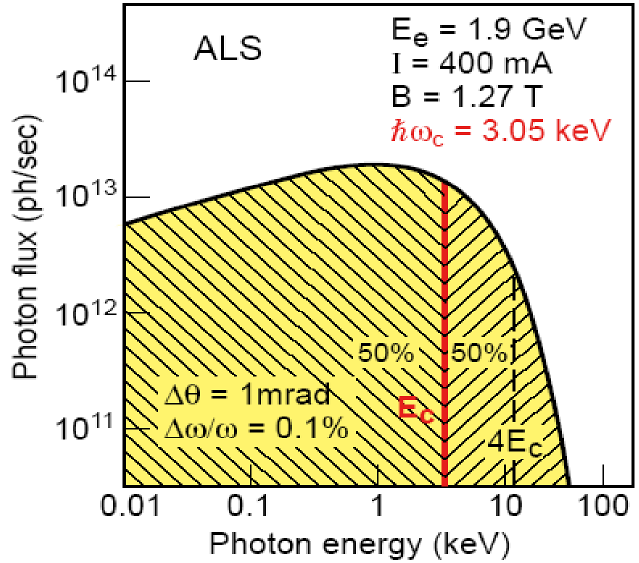


$$2\Delta\tau = \frac{m}{2eB\gamma^2}$$

$$\Delta E \geq \frac{2e\hbar B\gamma^2}{m}$$

$$E_c = \hbar\omega_c = \frac{3e\hbar B\gamma^2}{2m} \qquad \gamma = \frac{E_e}{mc^2} = 1957E_e(\text{GeV})$$

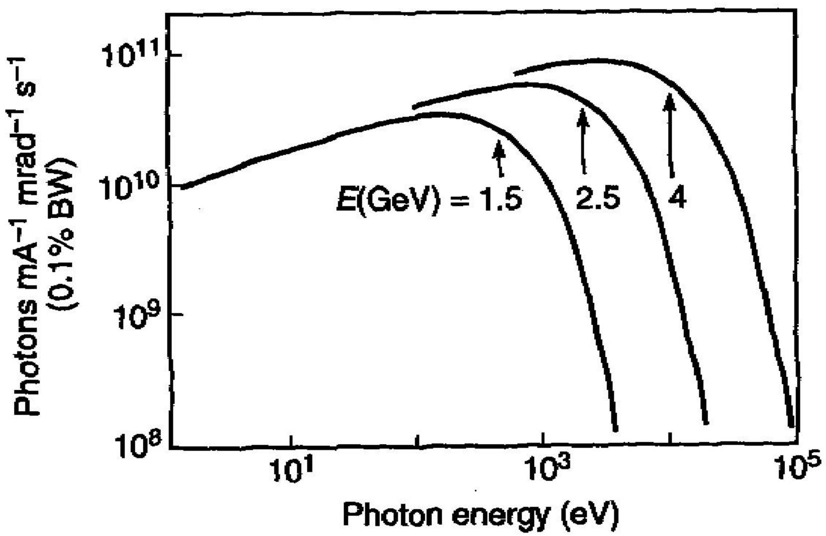
$$E_c(\text{keV}) = 0.6650E_e^2(\text{GeV})B(\text{T})$$



SR Properties

- broad spectral range
  - X-rays-VUV-visible & beyond
- *highly collimated*
  - *~ milli-radian divergence*
- very high flux and brightness
- *linear polarization*
- calculable characteristics
- *Time structure*

SR Spectrum

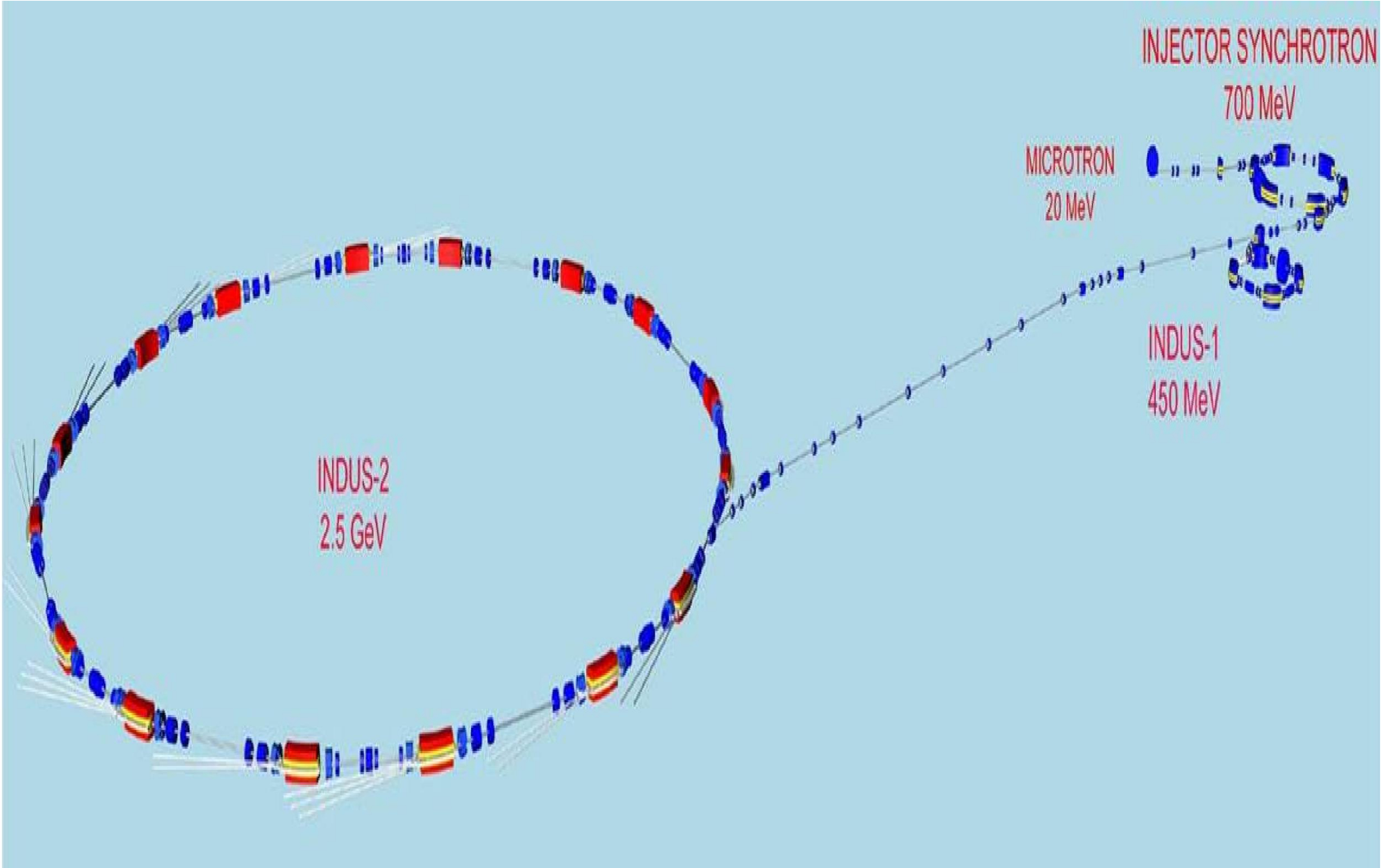


$$E_c(keV) = 0.665BE^2$$

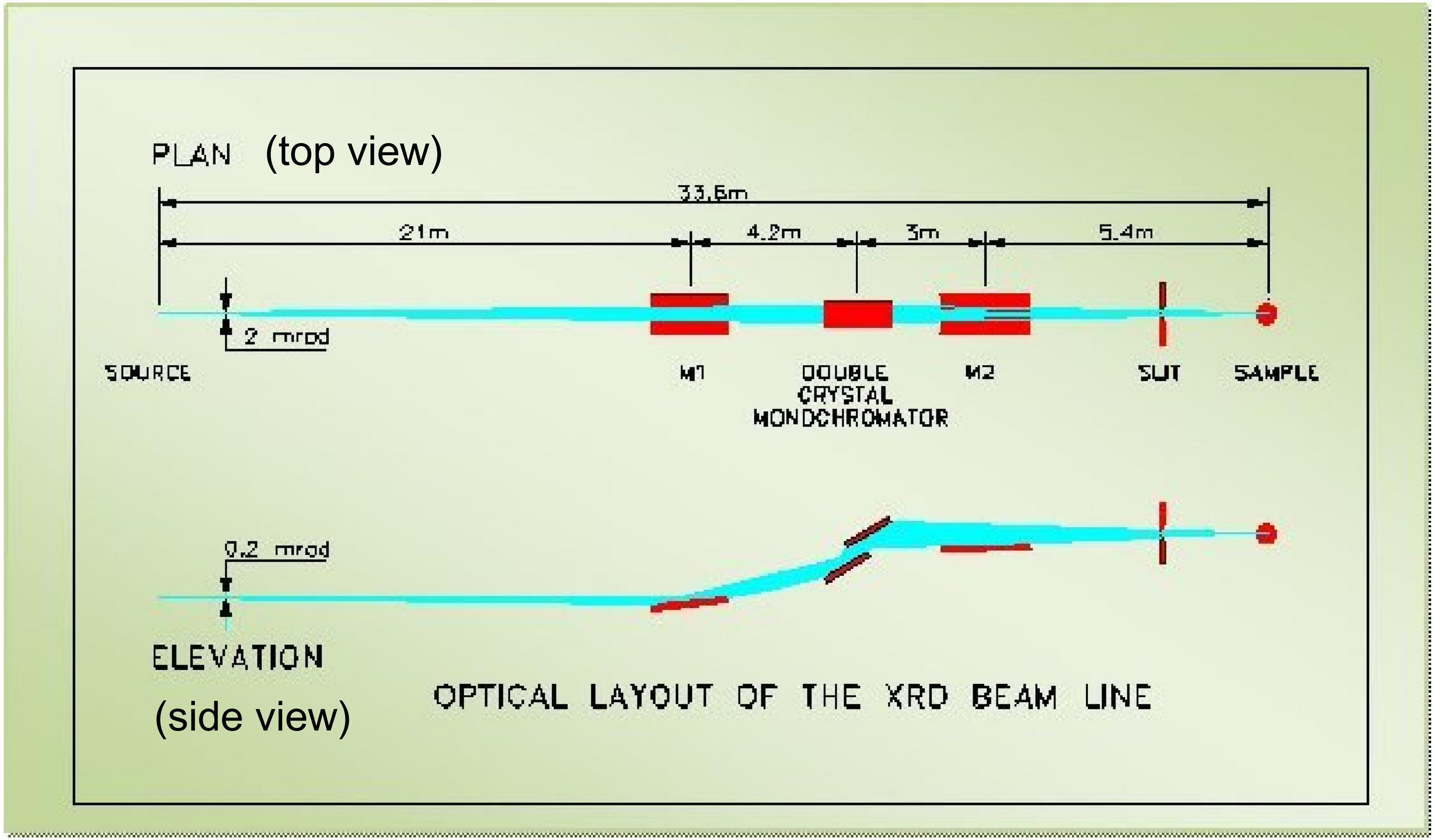
Flux ⇒ photons/sec/mrad/0.1% BW

brightness ⇒ Flux/ mm²/mrad²

Schematic View Of Indus Complex



Optical Layout of ADXRD beamline



Advantages of using adaptive optics

1. Various operation modes of the beamline

1. High energy resolution , low flux mode (collimated mode)

2. High energy resolution, moderate flux mode

2. The photon beam can be focused at desired point making it convenient for use of multiple experimental stations

Measured beamline specifications

➢ Range:	5 – 20 keV
➢ Energy resolution (E/ΔE):	8000 (at 8keV)
➢ Flux: @2.5GeV, 100mA :	3x 10 ^{9} ph/sec (at 11 keV)
➢ Beam size :	0.7mm(h) x 0.5 mm(v)

Applications

- Powder X-ray Diffraction
- Single Crystal Diffraction
- X-ray absorption spectroscopy
- XRD at extreme conditions

Low Temperature XRD (3K – 400K)

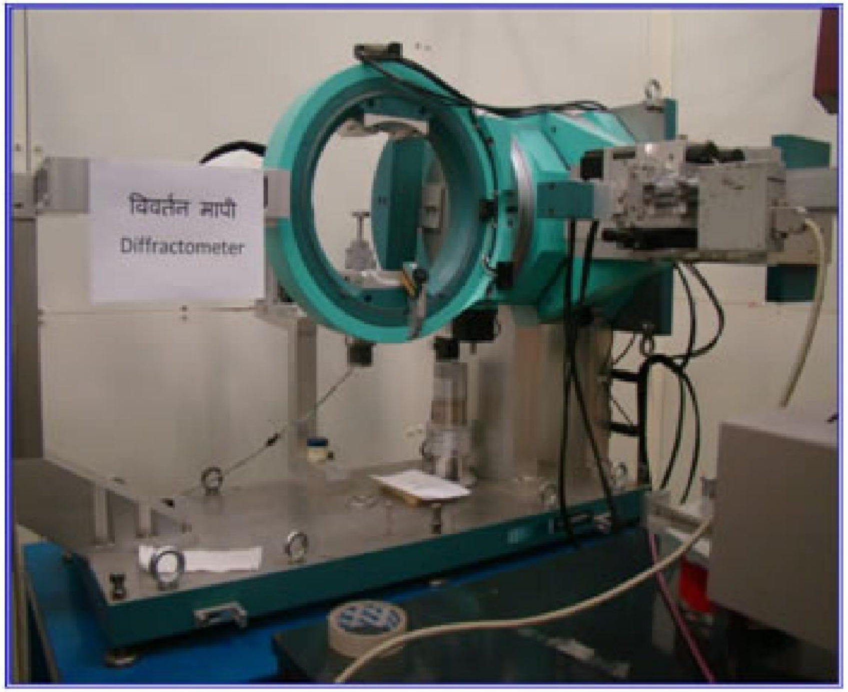
High Temperature XRD (900K)

High pressure XRD

Photograph of the beamline in radiation hutch

EXPERIMENTAL STATIONS

Six circle diffractometer with scintillation detector



Angular resolution:

Powder Sample: 0.015 degree (sigma in 2 theta)

Single Crystal: 14 arc sec (FWHM)

Image plate area detector

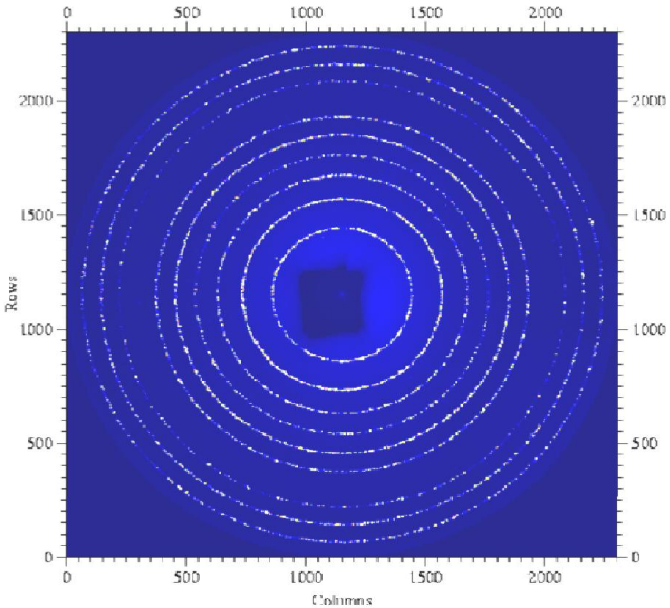
Angular resolution:

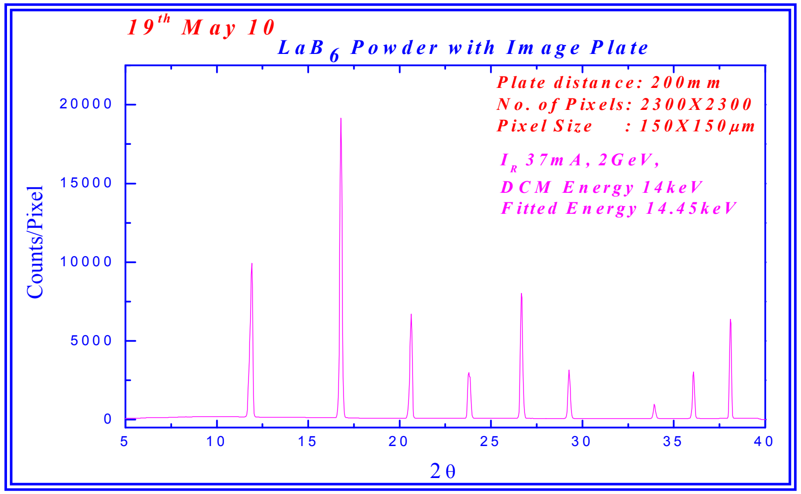
Powder Sample: 0.03 degree (sigma in 2 theta)

Time taken for one pattern: Few Minutes

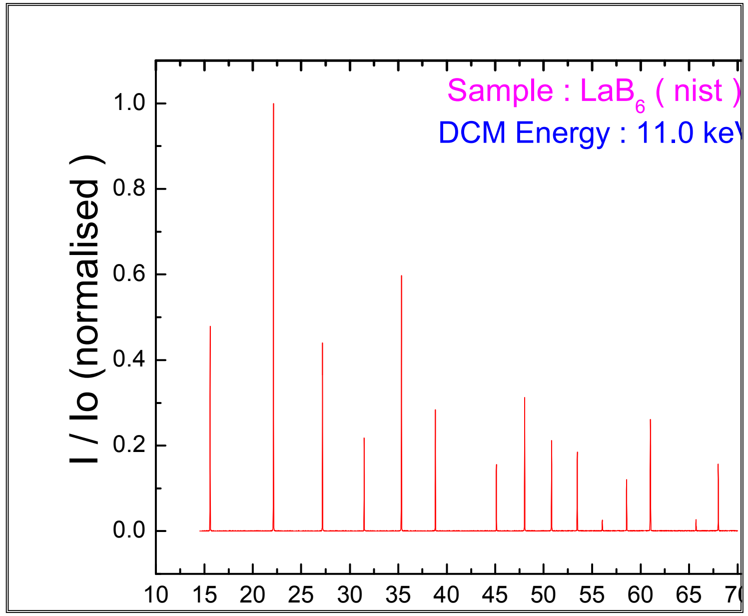
EXPERIMENTAL STATIONS

Powder diffraction at the image plate area detector





Powder diffraction at the Diffractometer



Powder diffraction at the diffractomer with scintillation detector

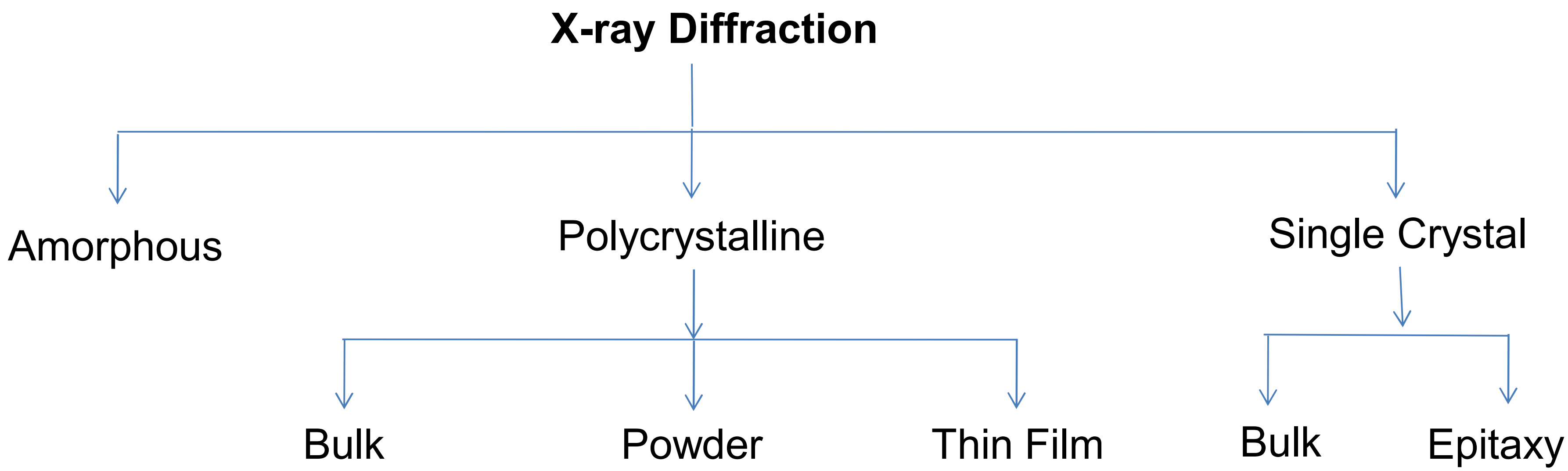
AIP conf proc. 1349 (2011) 503

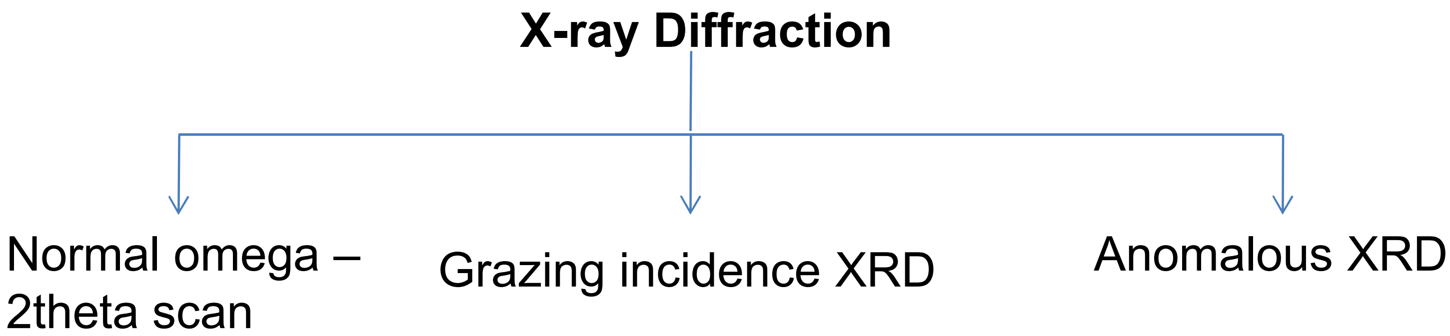
J. Phys.: Conf Series **425**, 072017(2013)

Applications of the beamline

- X ray Diffraction (XRD)
- Anomalous XRD
- X-ray Absorption Near Edge Structure (XANES)
- High Pressure XRD
- Low temperature XRD
- High Temperature XRD

Applications of ADXRD beamline





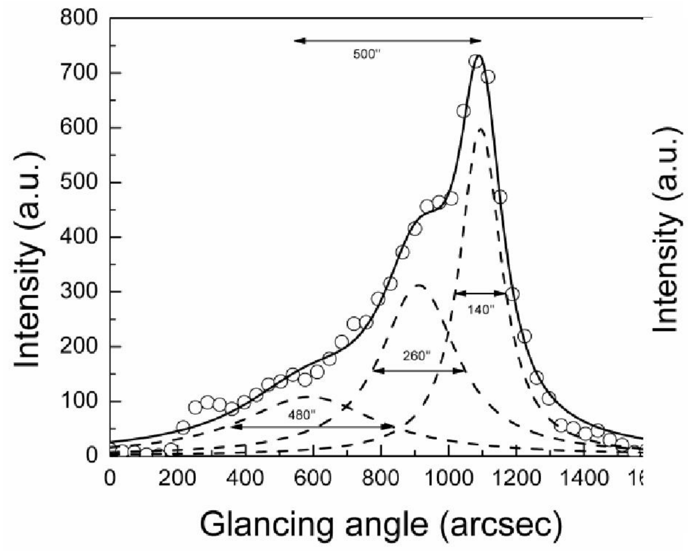
X-ray Diffraction under extreme conditions of temperature and pressure

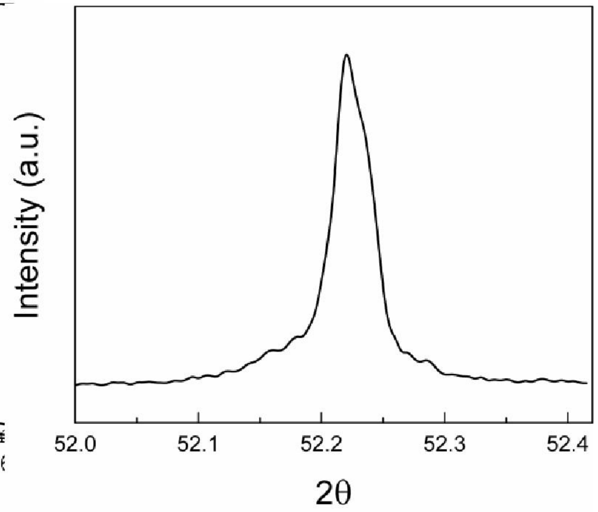
Applications of the beamline

- Single Crystal Diffraction
- Powder diffraction
- Amorphous phase scattering and RDF
- Anomalous XRD
- X-ray Absorption Near Edge Structure (XANES)
- High Pressure XRD
- Low temperature XRD

Applications of the beamline

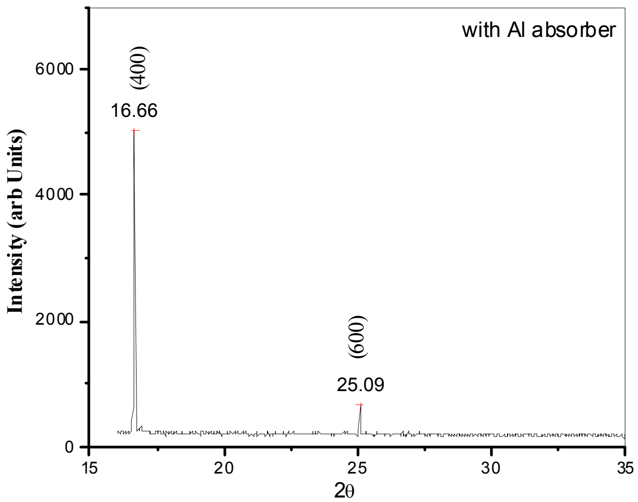
1. Single crystal diffraction

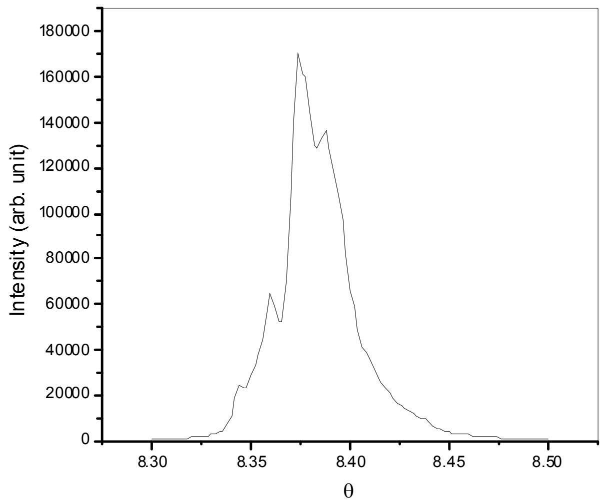




J. Crystal Growth **351** (2012) 88.

(a) Rocking curve for (4 2 2) plane of 0.1 mole% Tl doped CsI and (b) 2Θ position of same plane. (energy- 9750 eV).

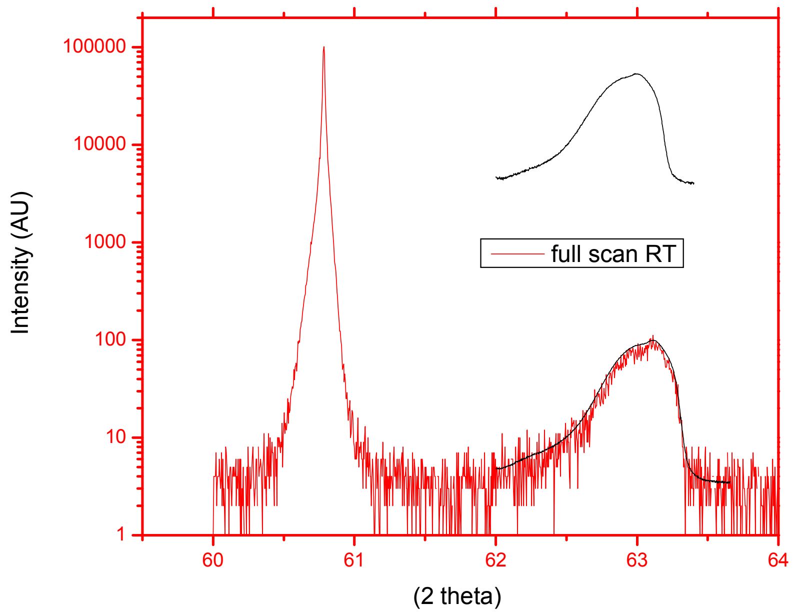


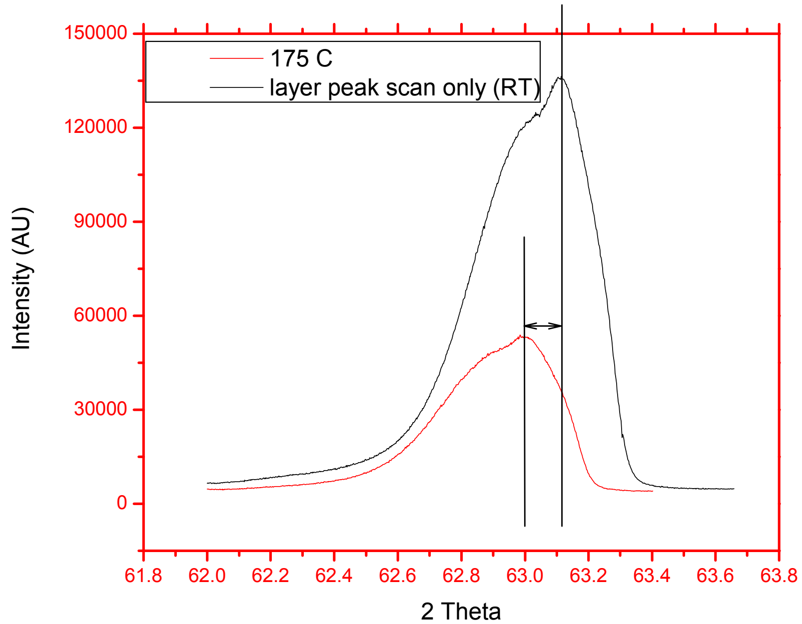


Appl. Optics, **50** (2011) 6006.

(a) Θ-2Θ scan for (100) plane of monoclinic Ga_2O_3 single crystal (b) Rocking curve of the (400) peak of (100) plane

GaP epitaxial layer on Ge (111)



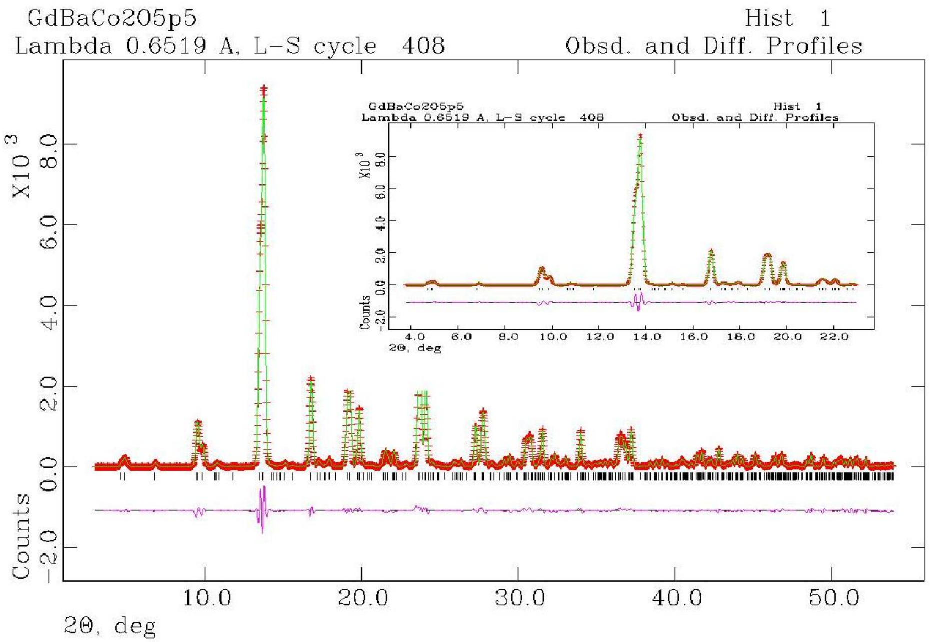


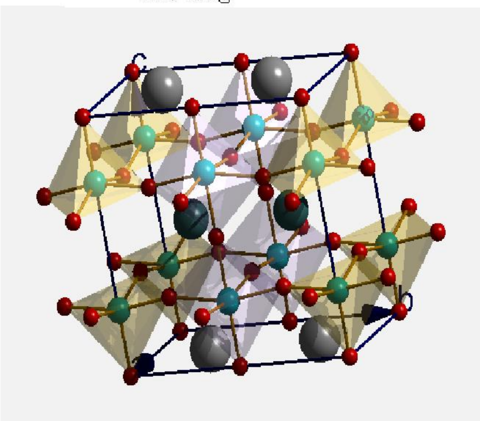
GaP (111) grown on Ge (111) substrate.

Full (444) scan shows Ge and GaP peaks.
Two domains with 60 degree angle between them
High temperature shift in peak may be because of change in lattice parameter and Strain.

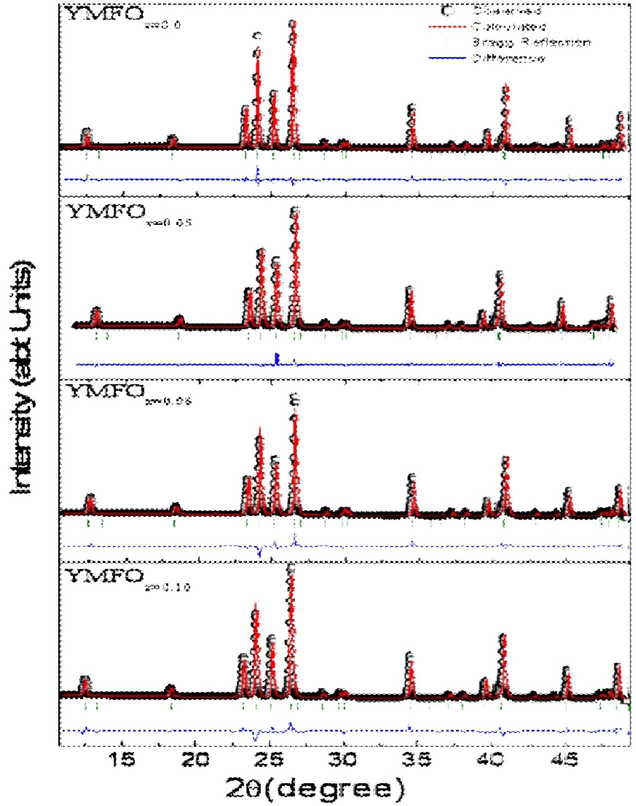
2. Powder Diffraction

X-ray diffraction studies of $Gd_{1-x}Ca_xBaCo_2O_{5.5}$ system ($0 \le x \le 0.30$)





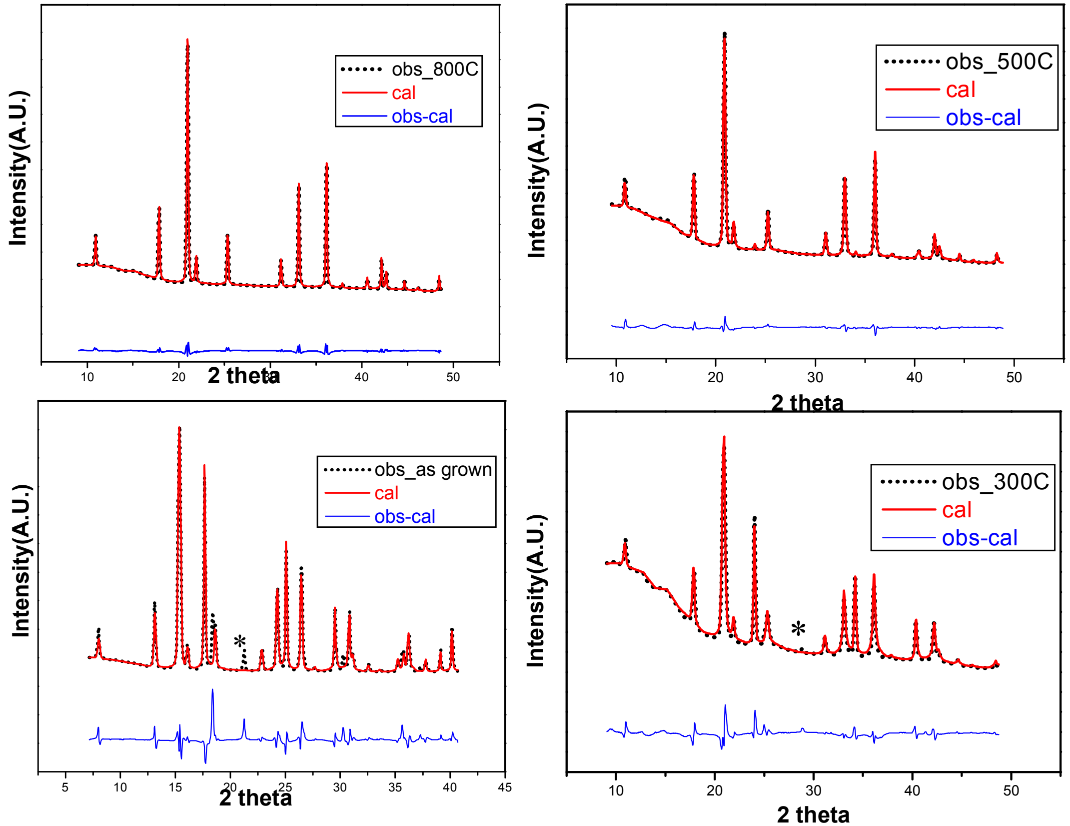
The crystal structure of $GdBaCo_2O_{5.5}$ from the refined values of lattice and structural parameters obtained from the Rietveld refinement of XRD data obtained at ADXRD beamline.



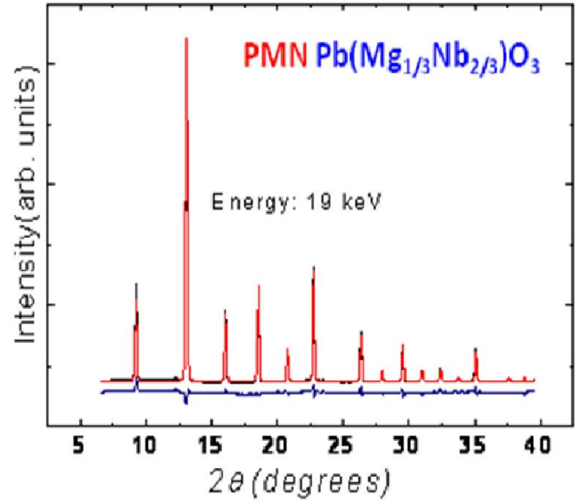
Rietveld-refined XRD pattern of $YMFO_{x=0.0\text{-}0.10}$ samples at 10KeV

J. Appl. Phys. ***113*** *(2013) 104101*

2. Powder Diffraction



Multi-phase Rietveld refinement of various phases of coblat oxide nanoparticles



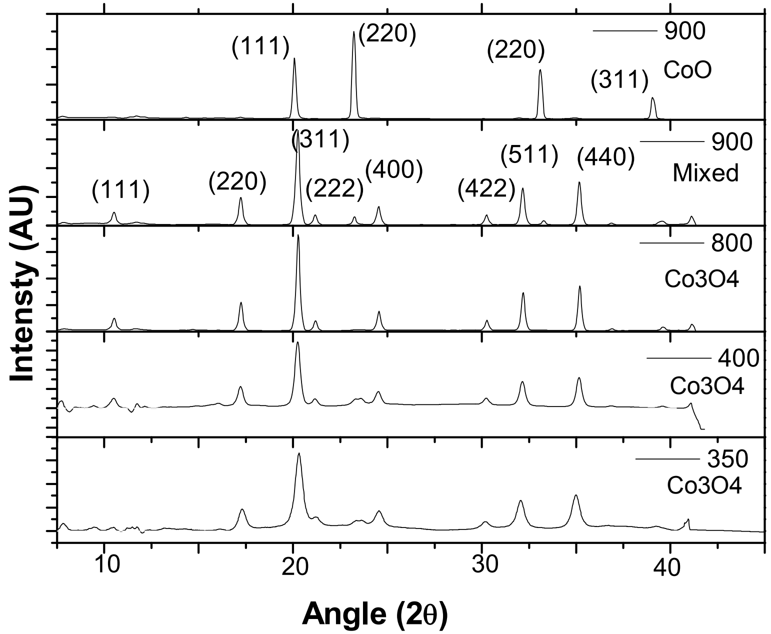
The XRD data along with the Rietveld fitting of relaxor ferroelectric [$Pb(Mg_{1/3}Nb_{2/3})O_3$] obtained on the Angle dispersive XRD beamline (BL-12)

J. Phys. and Chem. of Solids 75 (2014) 397

2. Powder Diffraction (Nano-particles)

Image Plate data

Powder XRD on Cobalt oxide nanoparticles

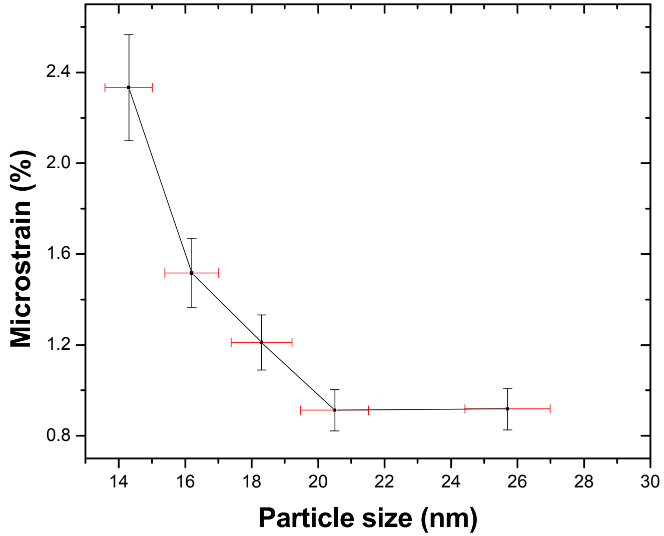


Williamson-Hall Plot

$$(\beta_{measured})^2 = (\beta_{instrumental})^2 + (\beta_{strain})^2 + (\beta_{size})^2$$

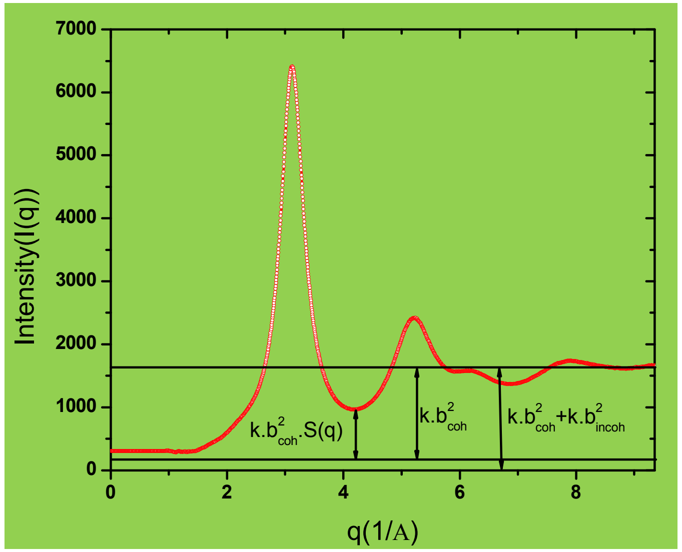
$$B\ (\text{strain}) = 2.\ \varepsilon.\ \tan\theta$$

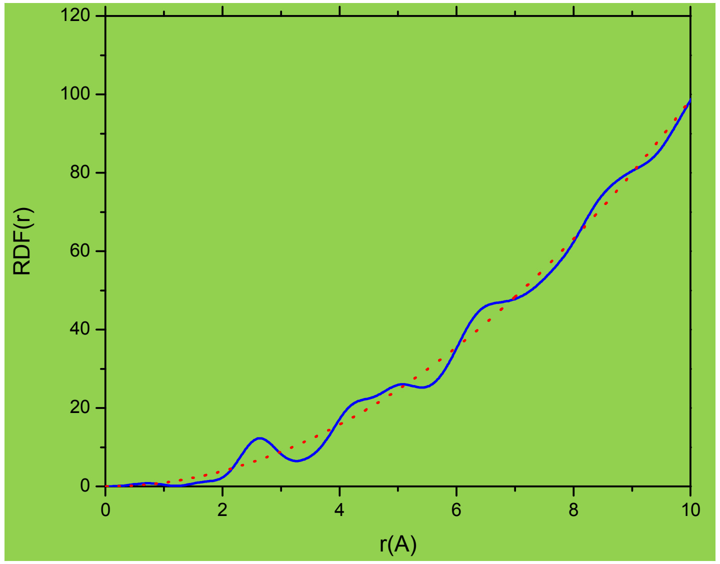
$$B\ (\text{size}) = (0.9 \times \lambda)/(D \cos\theta)$$



Appl. Phys. A 108 (2012) 607

3. Amorphous phase scattering and RDF

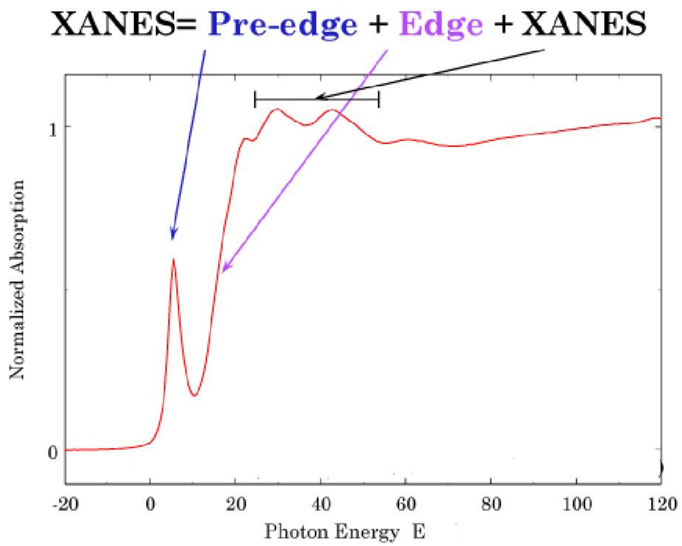




1. Take XDR data with highest possible q range
2. Correct the data for absorption correction, correction for sample holder etc
3. Normalize the such that I(q) = 1, at q tending to infinity and I(q) = S (0) at q = 0
4. S(0) may be calculated by thermodynamic limit
5. This gives static structure factor (S(q)).
6. FT of S(q) gives Radial distribution function (RDF), which is the atomic distribution of atoms in real space

J. Appl. Phys. 111 (2012) 113518.
J. Alloys and Compounds (2014), In Press
Measurement (2014), In Press

4. X-ray absorption spectroscopy (XANES)

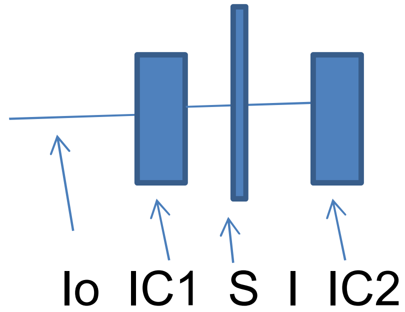


Information

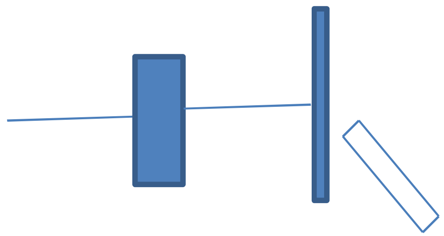
1. Oxidation state of selected atom in a compound
2. Electronic energy levels
3. Geometry of at atom in the lattice

Measurement Modes

1. Transmission
2. Fluorescence
3. Photoelectron yield

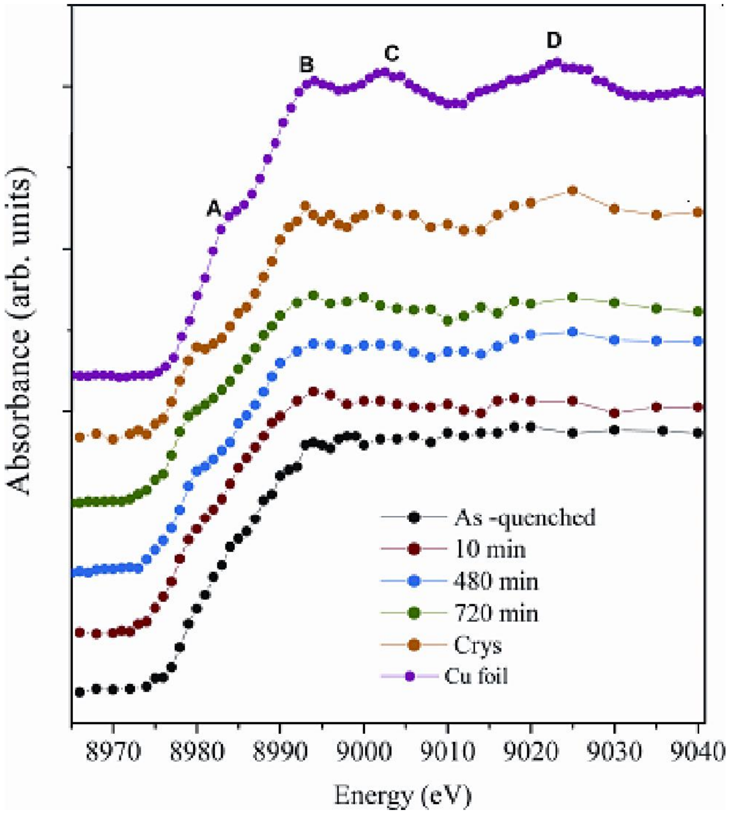


I = Io x exp (- abs.t)



4. XANES

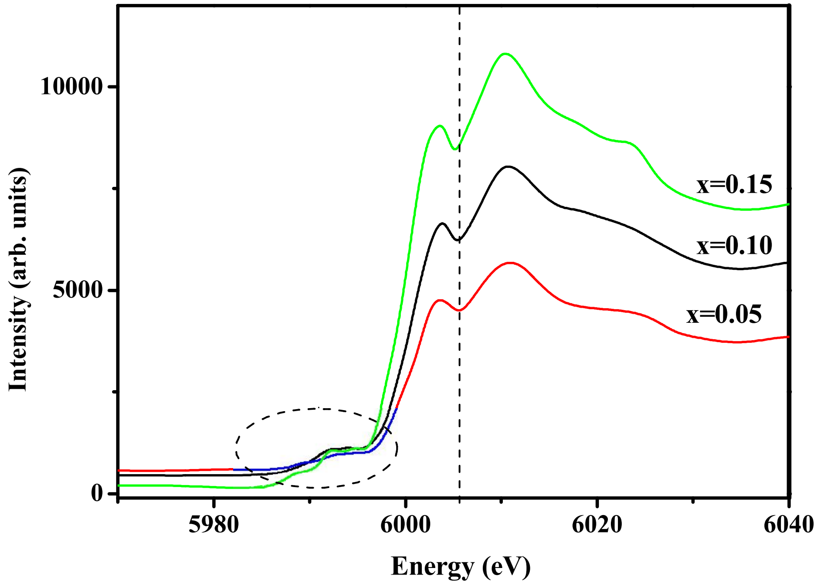
XANES at Cu edge



$Fe_{72.9}Cu_{0.9}Nb_{3.1}Si_{13.1}B_{8.9}$ (Finemet)

J. Appl. Phys. 110 (2011) 933537

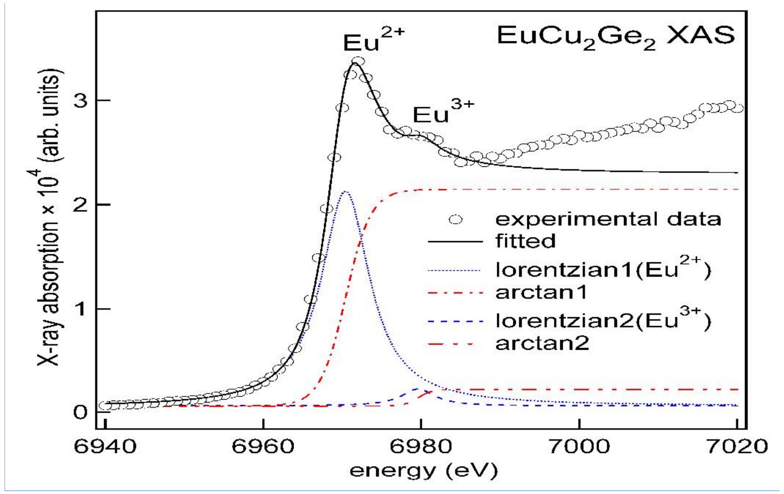
XANES at Cr edge

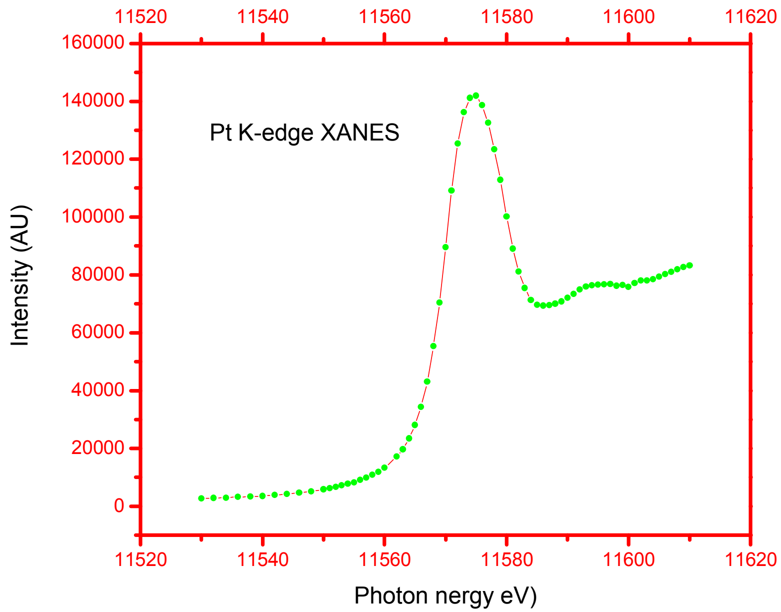


$(Na_{0.5}Bi_{0.5}TiO_3)_{(1-x)}(BiCrO_3)_x$

J. Mat. Sci. **47** (2011) 2011

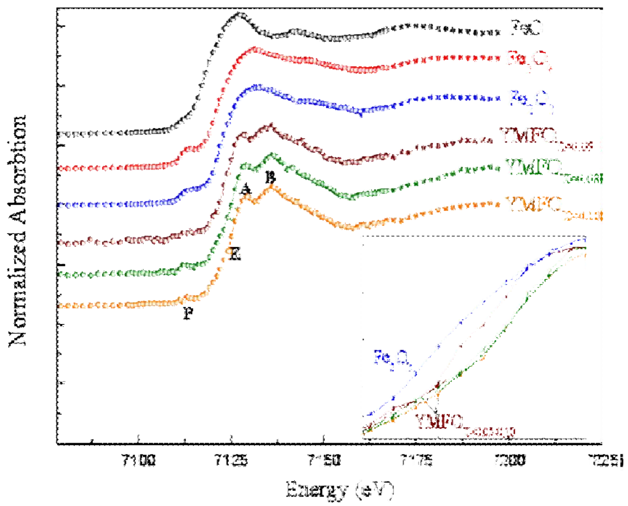
4. XANES





The relative intensities and position of Eu^{2+} and Eu^{3+} states were determined by fitting XANES spectra with a two component model consisting of an arctangent step function and a Lorentzian peak for each valence state. From fitting, the valence state population for Eu^{2+} is about 82%

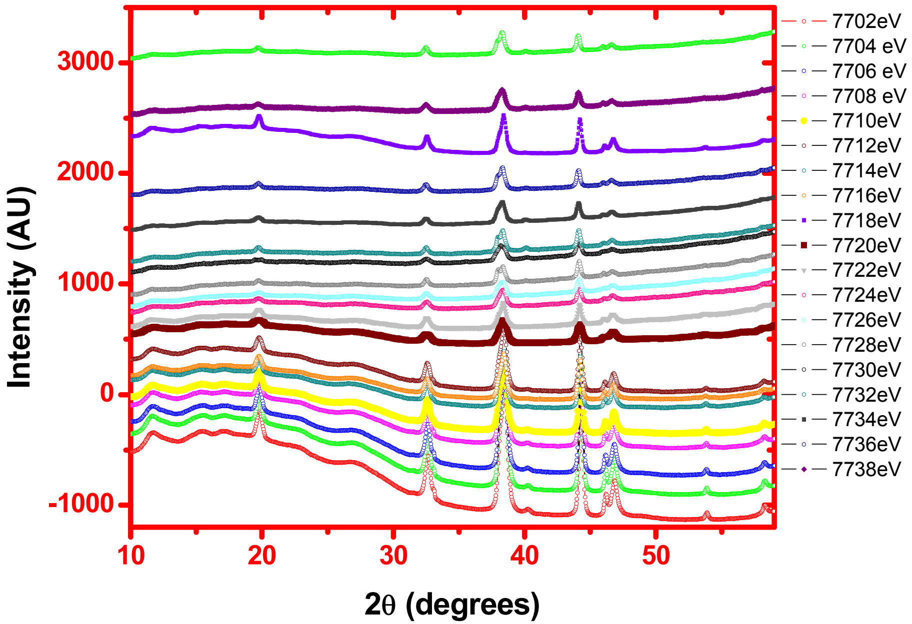
Phys. Rev. B. (2012) 00510

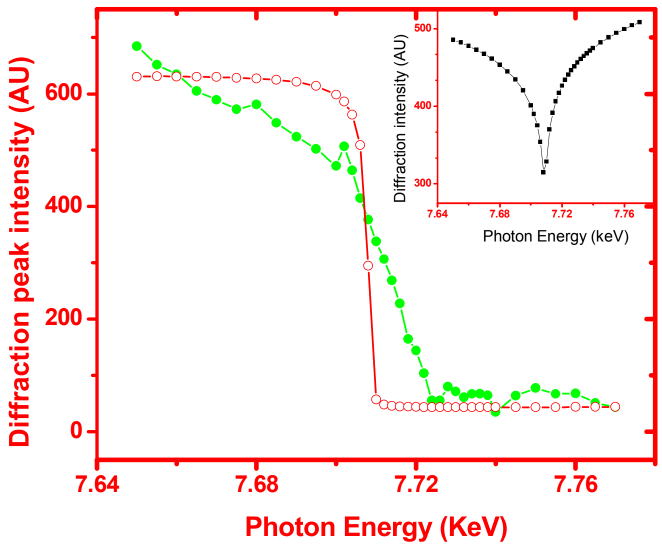


Normalized Fe-K edge XANES spectra.

***J. Appl. Phys.* 113** *(2013) 104101*

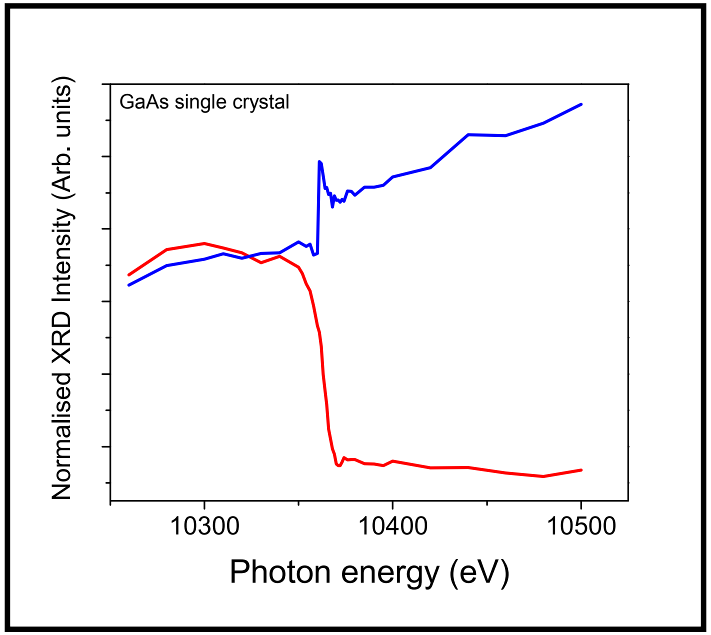
5. Anomalous XRD



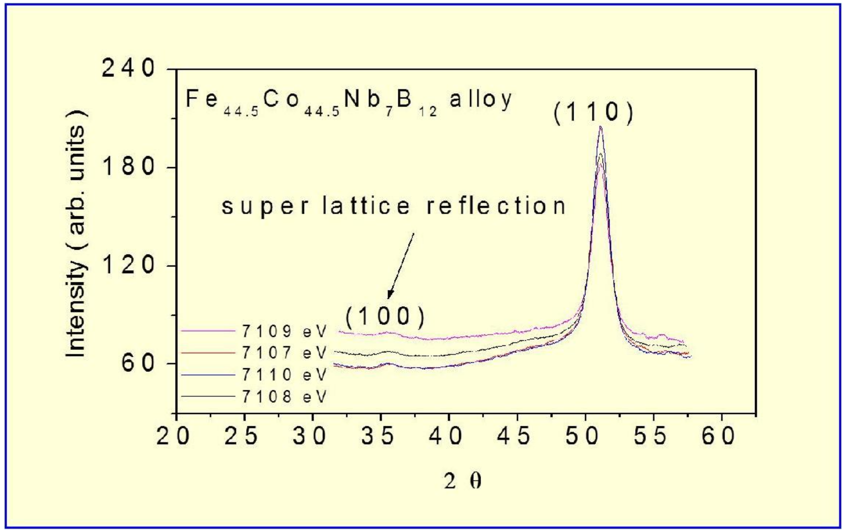


DANES in Cobalt oxide nanoparticles

5. Anomalous XRD



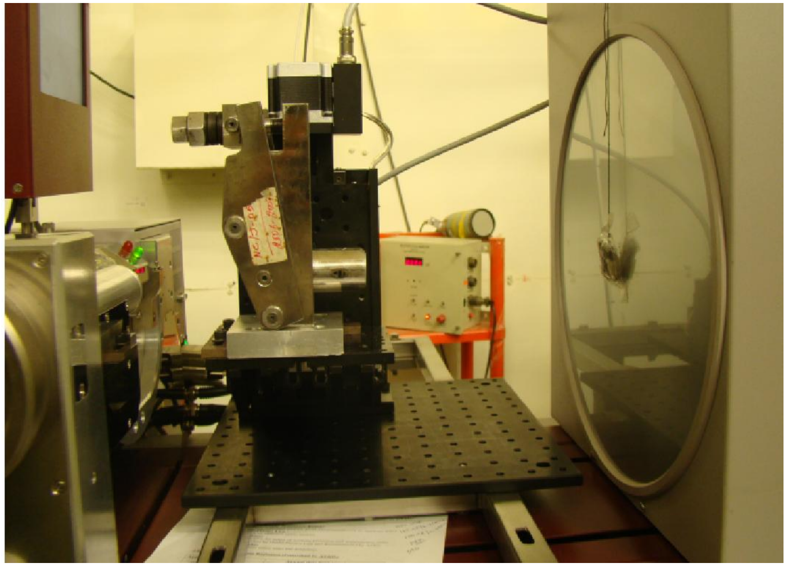
Normalized XRD intensity for Ga ended (blue) and As ended (red) faces of GaAs single crystal.

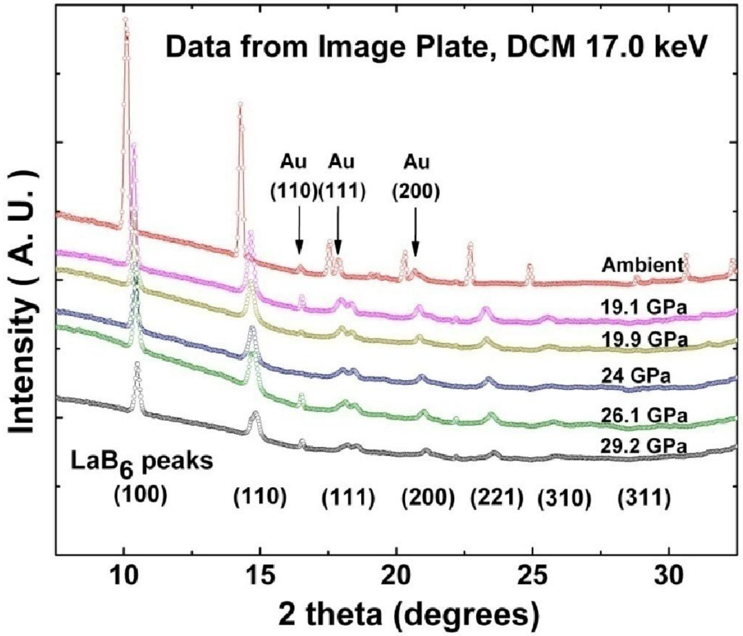


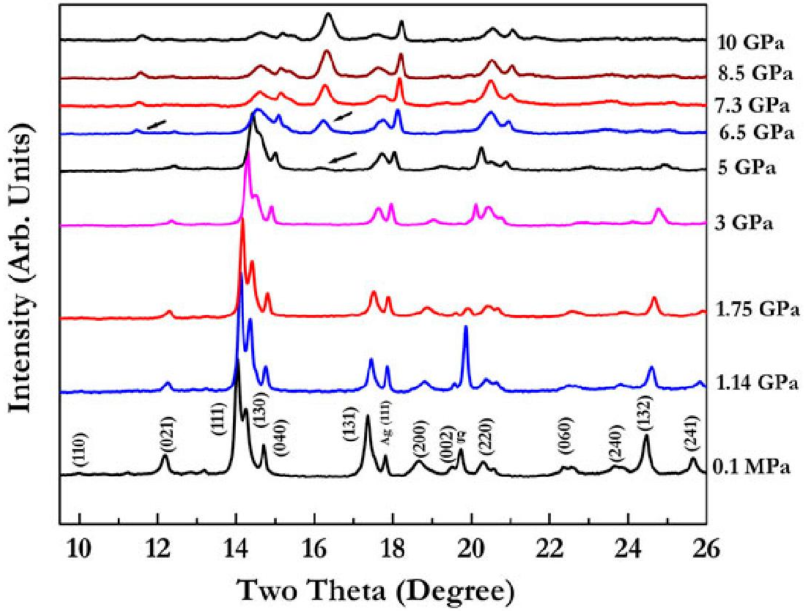
The (100) superlattice reflection, obtained using anomalous XRD at 7112 eV (Fe K-edge), is indicative of ordered structure.

J. Appl. Phys. 111 (2012) 113518

6. High pressure XRD







HPXRD pattern of LaGa with Ag as an internal pressure calibrant.

Phil. Mag. (2013)
http://dx.doi.org/10.1080//4786435.2013.826880

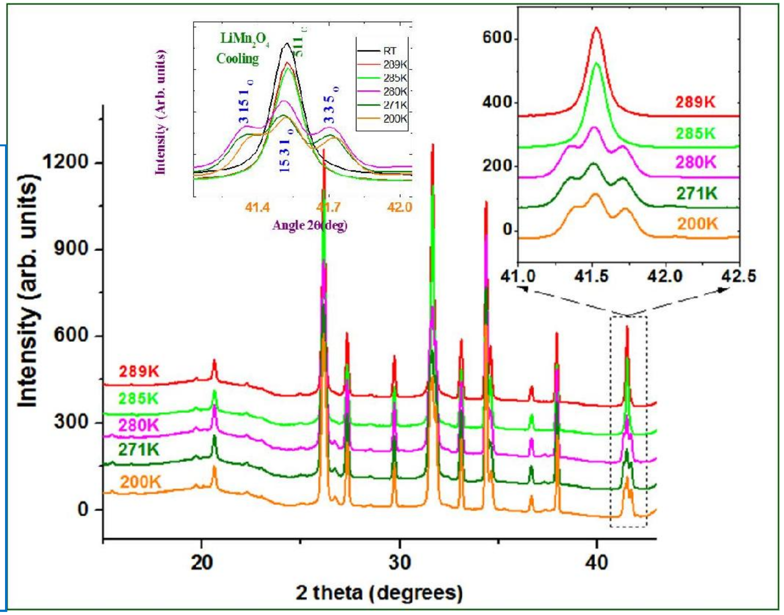
7. Low Temperature XRD Measurements

➢Liquid He based flow type cryostat

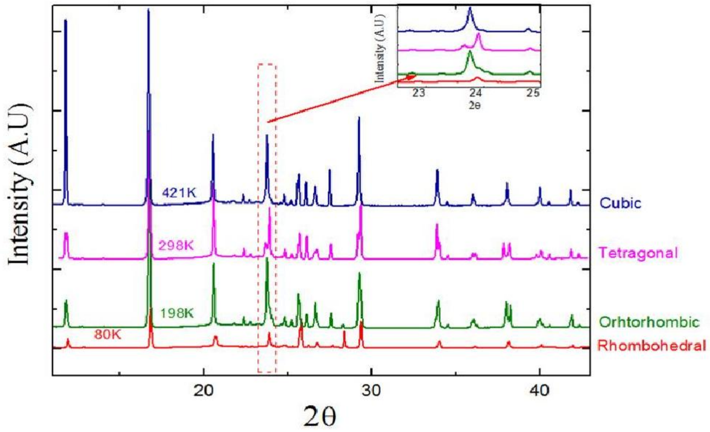
➢ Temperature Range - 3K – 450K

➢ PID Temperature Controller (Lakeshore 331)

Temperature stability ≈ 0.15K

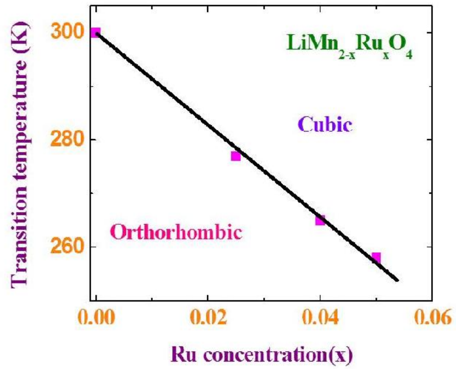


XRD pattern for Ru doped $LiMn_2O_4$ at various temperatures between 289K and 200K showing phase transition from Cubic to orthorhombic phase.



XRD pattern for $BaTiO_3$

Structural transition temperature as a function of increasing Ru concentration



Thank You for your kind attention