Commissioning experience of X-ray Scattering and Diffraction Beamline for Engineering Applications at Indus-2 Synchrotron Source

Dr. Sanjay Rai
Head,EAL
SUS/RRCAT

Plan of Talk

- Motivation
- Details of the beamline
- Experiments carried out
- Benefits

Why this beamline

- ***Presence of unfavourable residual stresses and texture can adversely affect the performance of components. Residual stresses can reduce fatigue life, enhance stress corrosion cracking and enhance creep deformation rate in bulk materials. Stresses can also influence the performance of thin film based devices e.g. MEMS, mulitlayer mirror structures etc.***
- ***For successful management of residual stresses, It is important to quantify stresses and understand it influence on the small smples as well as on large component/device.***
- ***Testing large size optics for performance assessment***

Why this beamline ...

- ***This beamline will use powder diffraction not for solving crystal structure but for understanding micro structure***

- ***Diffraction will be used for measuring macro and micro stresses and also for measuring texture of materials for solving applied egineering problems***

- ***Testing large size (1m length) synchrtron optics***

To meet the mentioned objective a new beamline was designed

- *This beamline is capable to operate in various beam modes viz; white beam, pink beam and monochromatic beam*

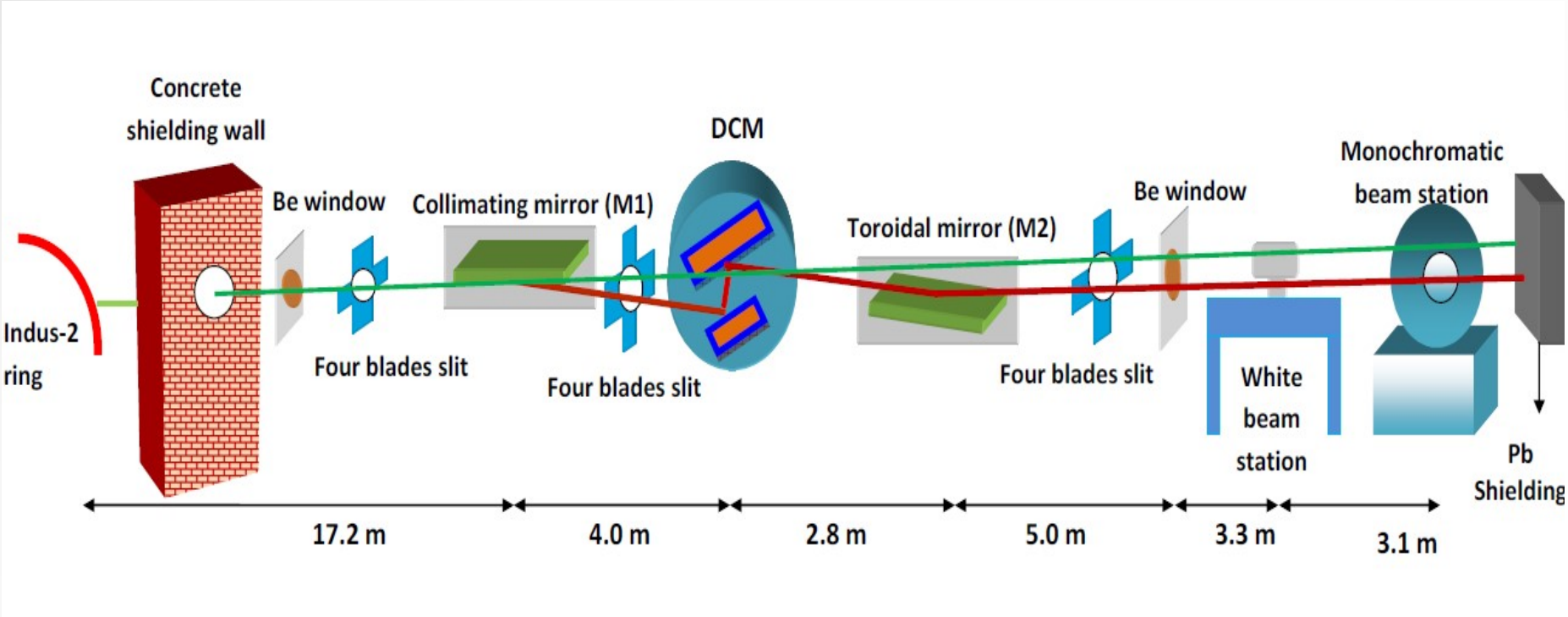
- *Energy range in monochromatic beam mode is~ 5-25 keV*

- *Energy range in white beam mode is ~ 5-45 keV*

- *Monochromatic beam experimental station has capability to handle large size samples and 1m long synchrotron optics*

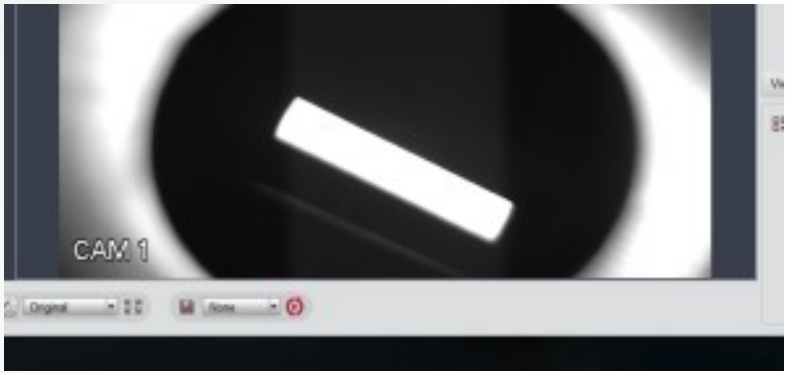
- *Its flexible design also allows a wide range of scattering experiments such as phase identification, kinetic studies, anomalous scattering, grazing angle diffraction and x-ray reflectivity*

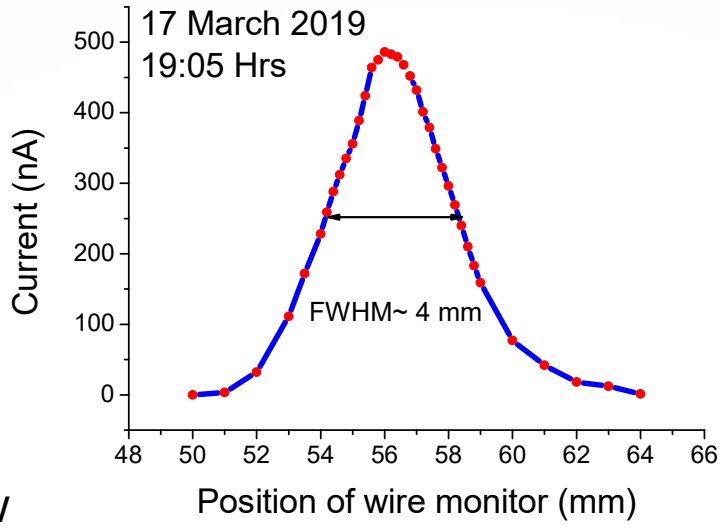
Bemaline layout and optics and other details



- ***Beam line has collimating and toroidal mirrors to achieve a spot size of ~300 micron***

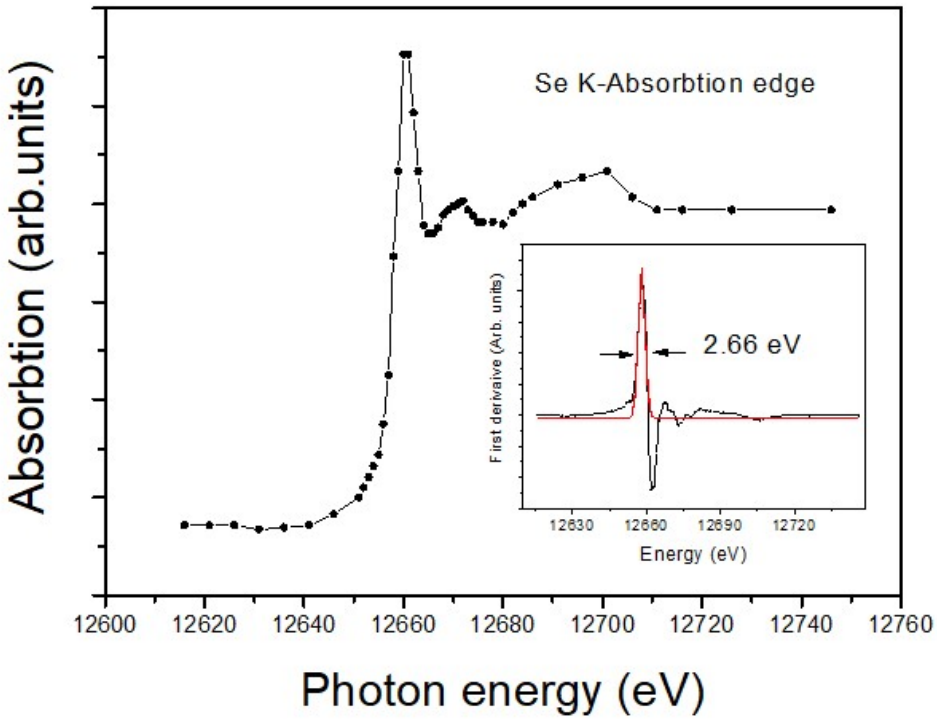
- ***Beamline uses Si 111 based double crystal monochromator and has resolution of ~5000 at 12 keV***





First Image of the beam after Be window

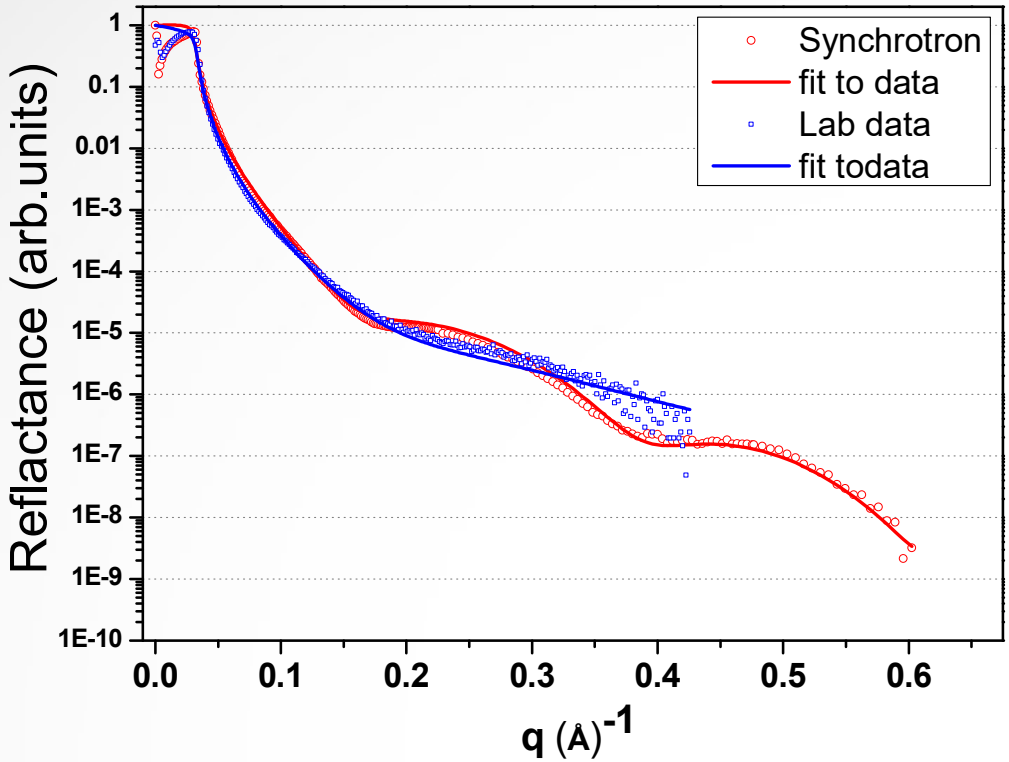
Beamline Energy Resolution



Energy resolution is obtained as ΔE = 1.5.eV at Se absorption edge

The maximum photon flux was found to be ~1.12 x10^9 photons/s/mm^2/100 mA at 12 keV

X-ray reflectivity measurements

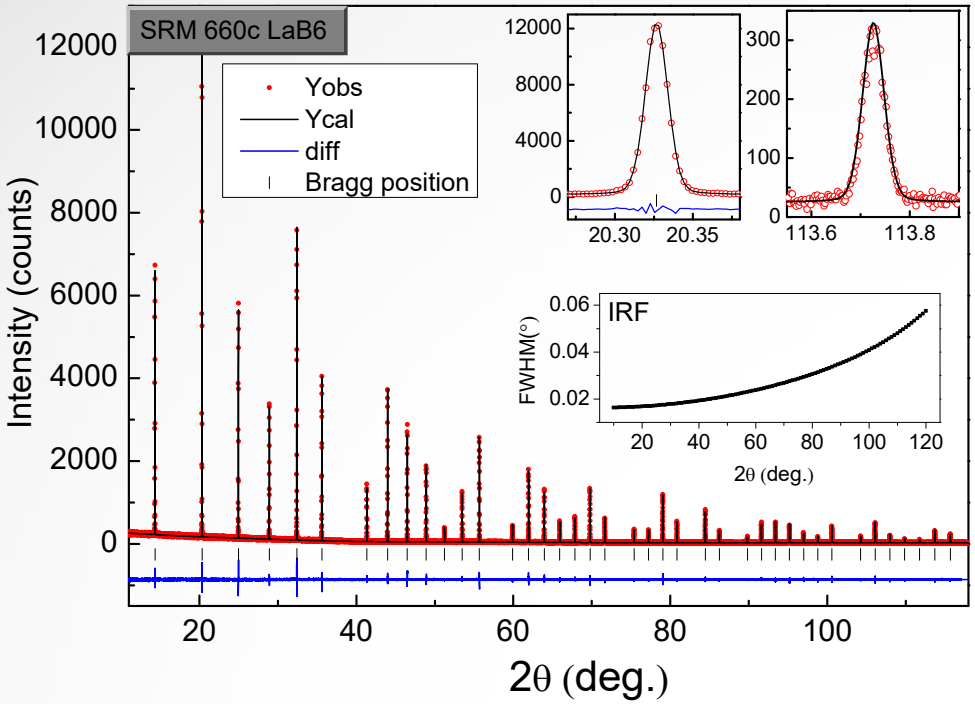


	Synchrotron beamline data		Lab data	
Layer	Thickness	Roughness	Thickness	Roughness
Top layer	27 Å	3.2 Å	12 Å	1.3 Å
Substrate	-	3.1 Å	-	7.0 Å

X-ray reflectivity (XRR) measurements on polished float glass substrate carried out at the beamline. For comparison lab source data is also included in the figure.

Highest dynamic range recorded at Indus-2 Large q range availability provides correct estimation of substrate roughness as ~3.1 Å

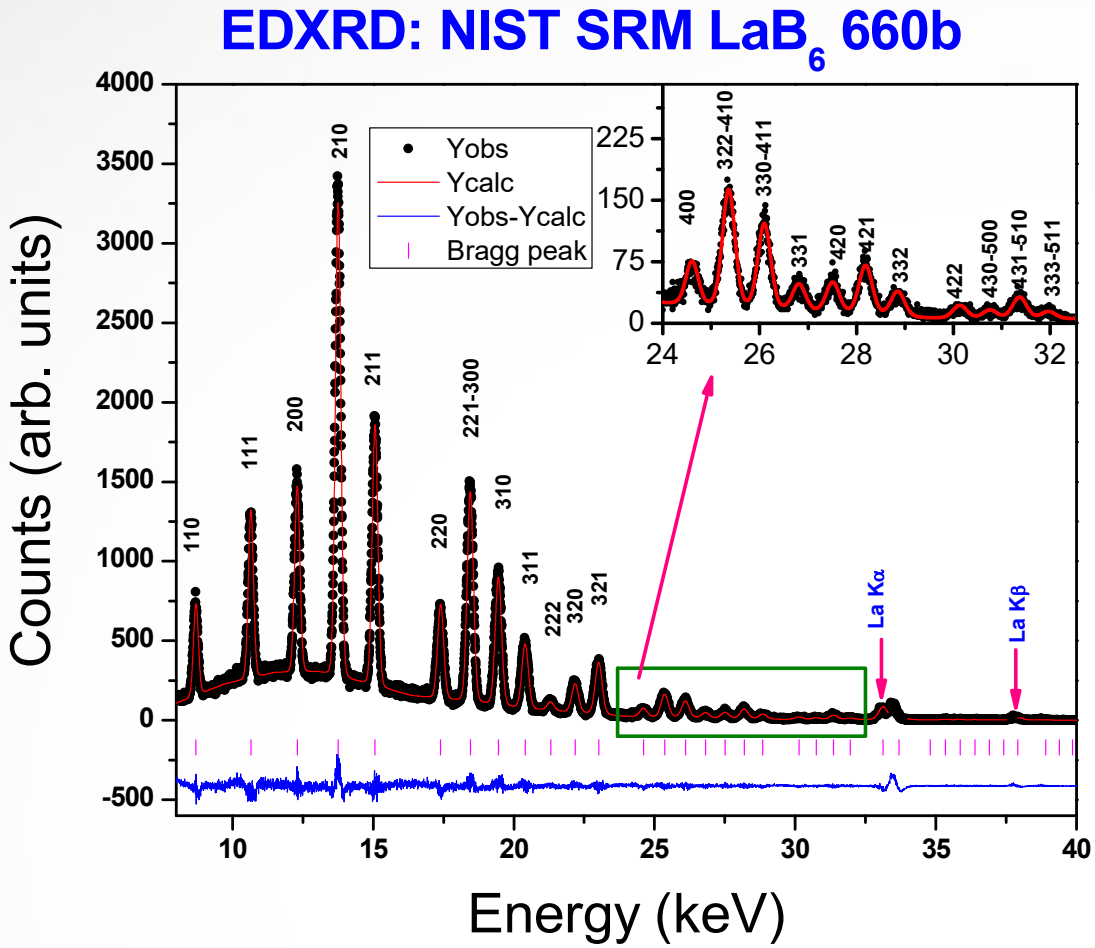
X-ray diffraction pattern of NIST SRM 660C LaB6



The Lebail fitting of NIST SRM 660C LaB_6 powder pattern measured at 12 keV: experimental data (red circles) and fitted (blue line), with their difference (residual, line below). Insets show fitting profiles of low and high angle peaks and IRF.

The high resolution achieved with standard powder would be helpful to generate high quality data for obating reliable micro-structural details using line profile

White beam EDXRD measurements of NIST SRM 660C LaB6



This operation mode is very useful for dynamic studies on materials as whole pattern was recroded in 50 seconds

Stress Measurement Using Diffraction Technique

Special Resolution

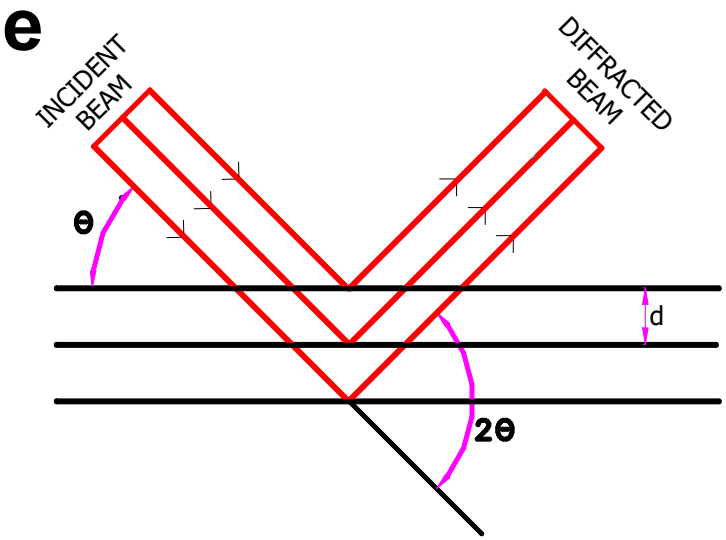
With Lab Source : ~2 mm

With Synchrotron : 200 micron

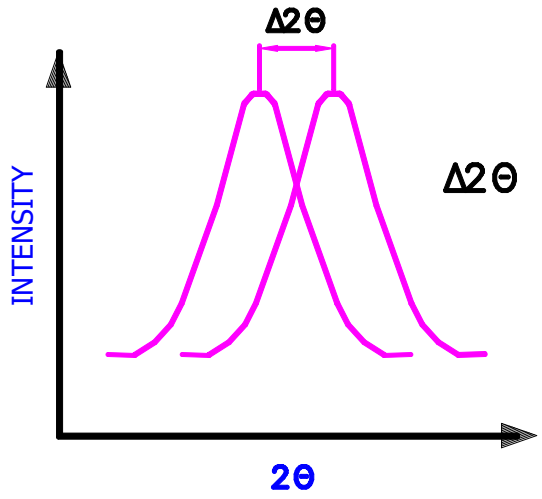
Penetration Depth in steel

With Lab Source : 1-5 micron

With Synchrotron : 5- 100 micron



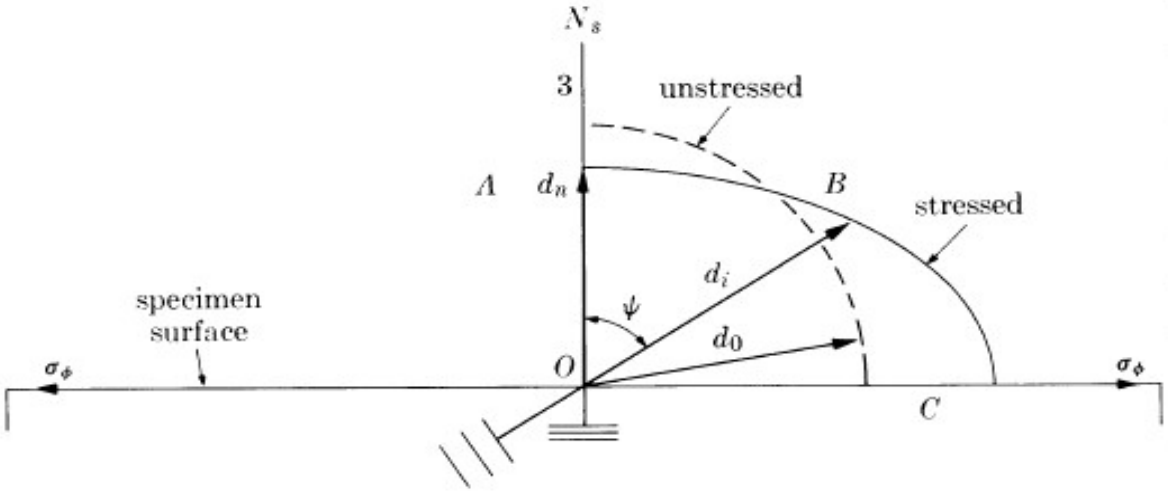
BRAGG'S LAW $2d \sin\theta = \lambda$



$\Delta 2\theta \equiv \Delta d$

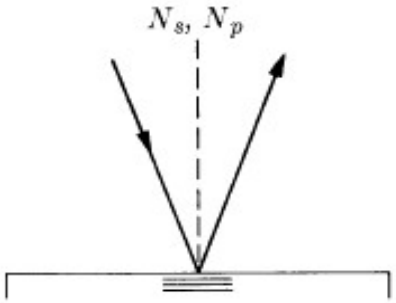
$\frac{\Delta d}{d}$ = STRAIN

The $\sin^2\psi$ Method

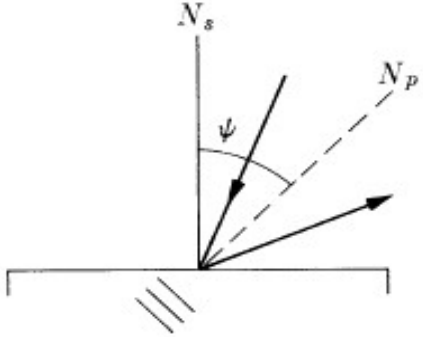


(a) Vector diagram of plane spacings d for a tensile stress σ_ϕ

$$\frac{d_{\phi\psi} - d_n}{d_n} = \frac{(1+\nu)\sigma_\phi}{E}\sin^2\psi$$



(b) Measurement of d_n



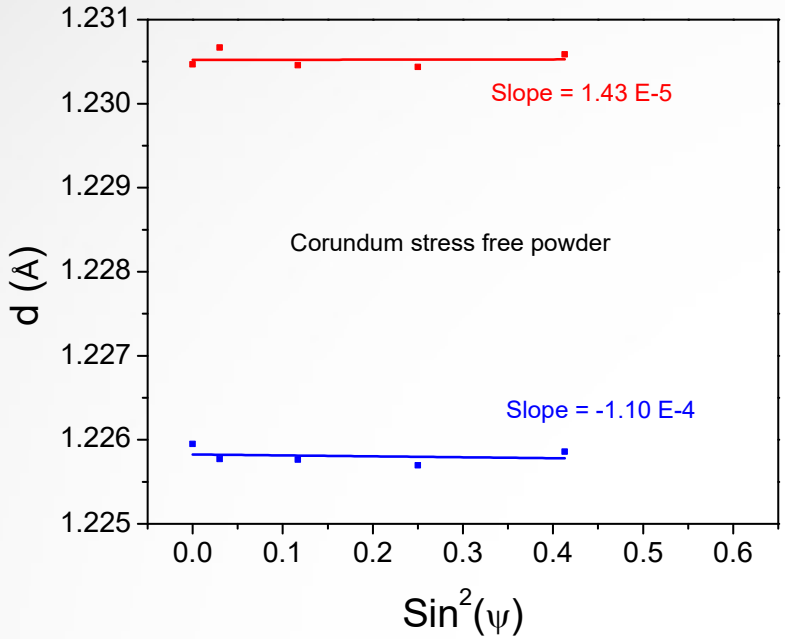
(c) Measurement of d_i

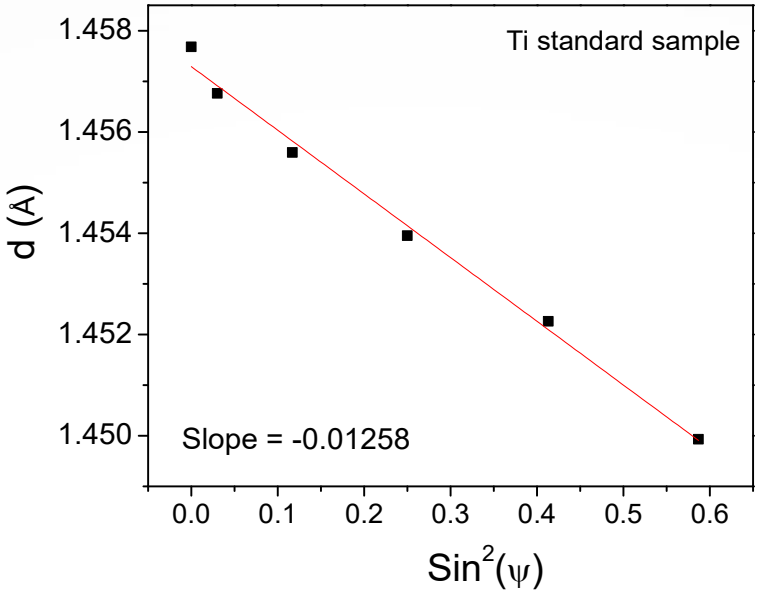
Requirements for stress measurements

The goniometer should be aligned such that the x-ray beam should pass through the rotation axis of the goniometer within sphere of confusion for compound motion of all four axes i.e. within 20 micron.

Stress value measured should have minmum influence due to sample loading varations.

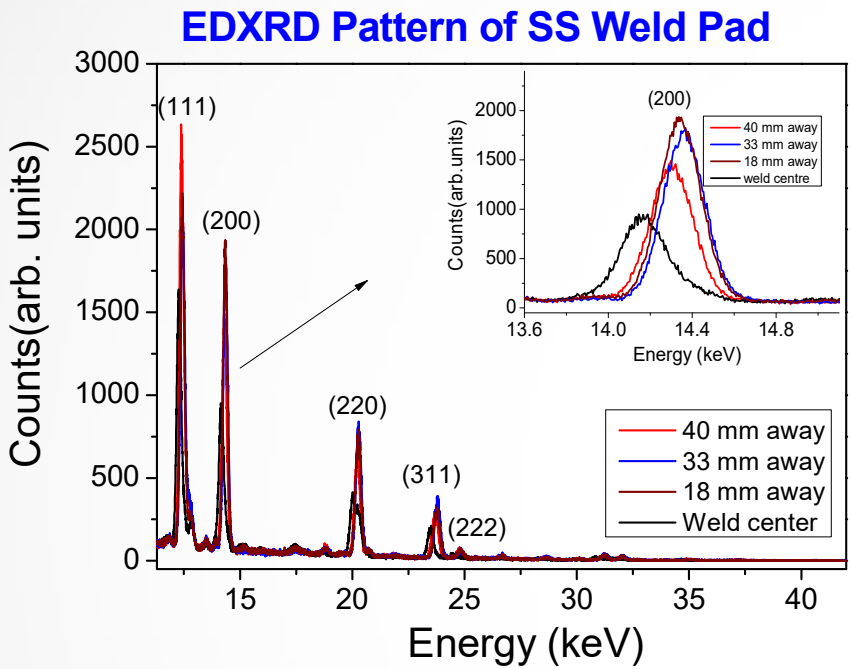
Calibartion of stress measurements

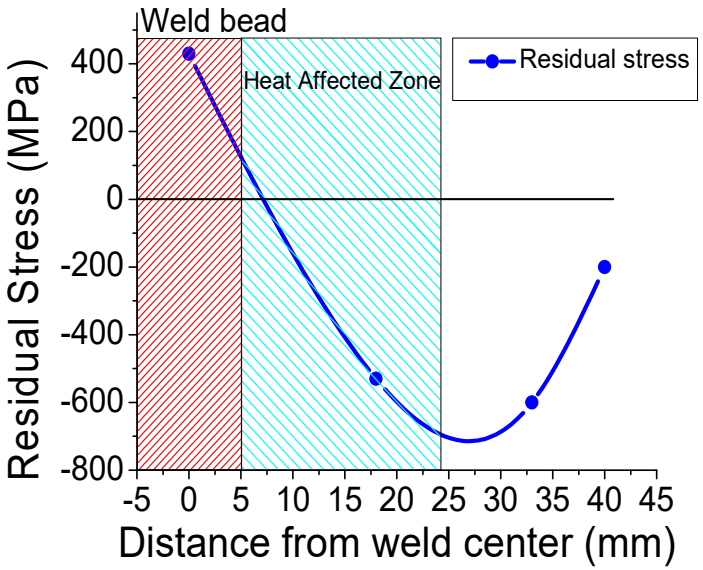




Plot of d vs $\sin^2(\Psi)$ curve for (a) stress free corundum (Al_2O_3) powder (b) Stresses Ti standard sample. Stress measurements were done in side inclination mode using beam energy of 15 keV. mean stress value of 710 ± 20 MPa was estimated.

Stress measurement on SS weld pad using EDXRD





Microstructural characterization of iron power using XRD at EABL, Indus-2

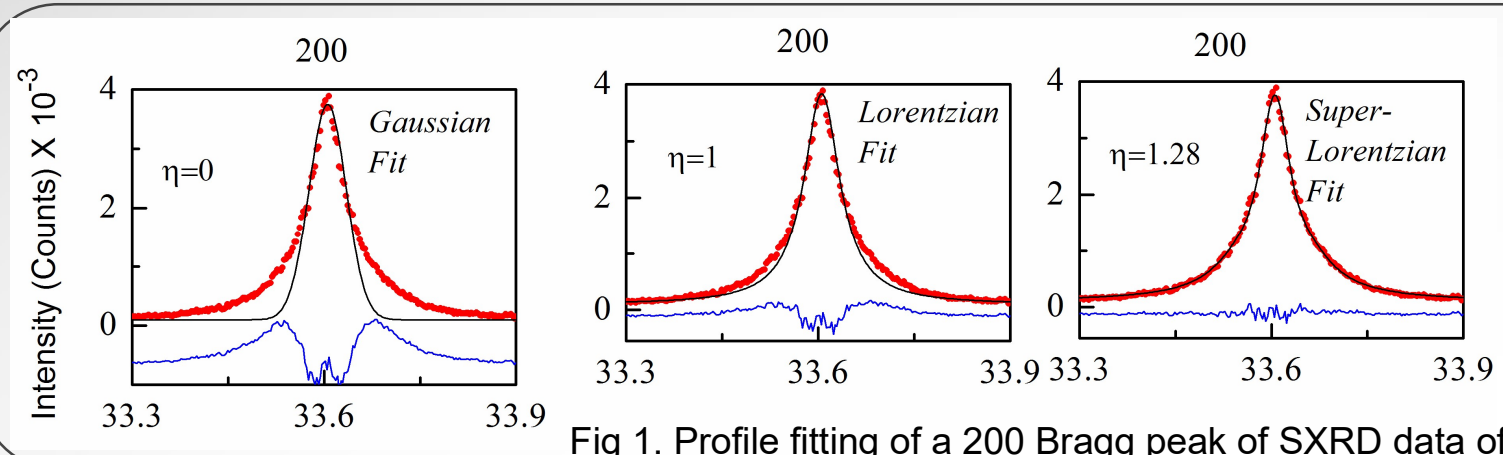


Fig 1. Profile fitting of a 200 Bragg peak of SXRD data of Fe powder.

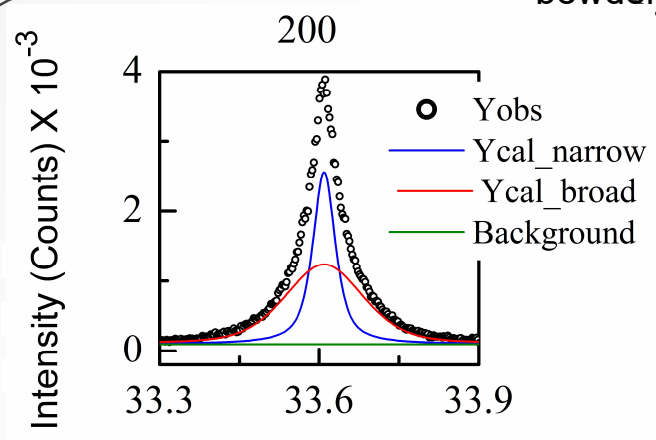


Fig 2. Profile fitting of a 200 Bragg peak of SXRD data of Fe powder using convolution of bimodal microstructure.

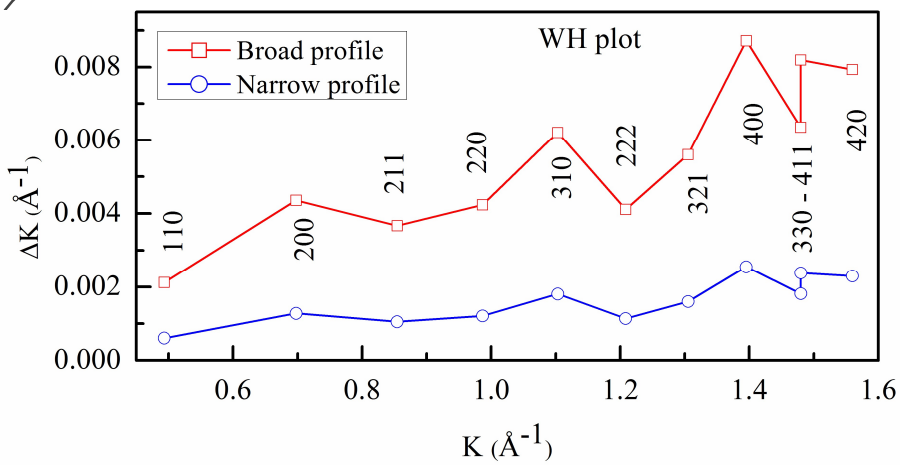
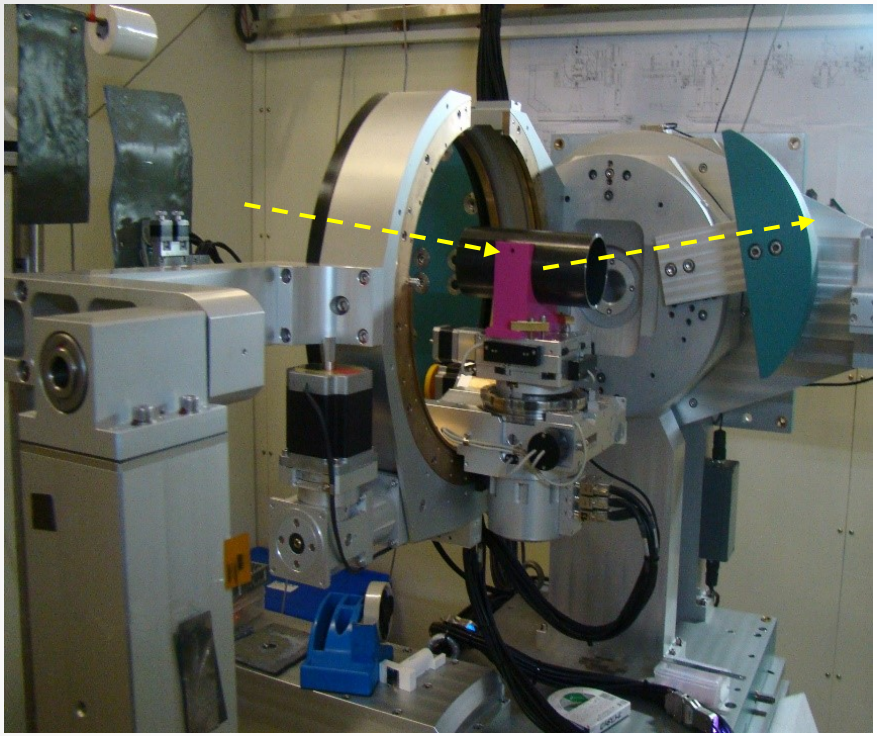


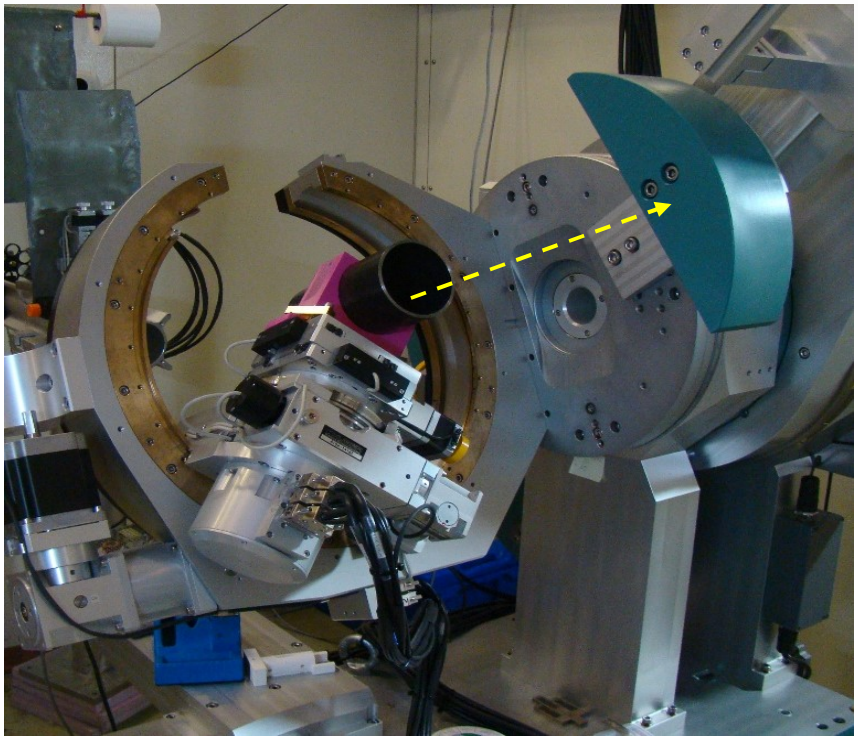
Fig 3. Comparison of deconvoluted narrow and broad profiles of Bragg peaks of Fe powder using WH plot.

Summary: The observed super-Lorentzian peak shapes of XRD data of Fe was modelled using convolution of bimodal microstructural parameters. The dislocation densities estimated for narrow and broad profile were about 2×10^{14} and 2×10^{15} m^{-2},

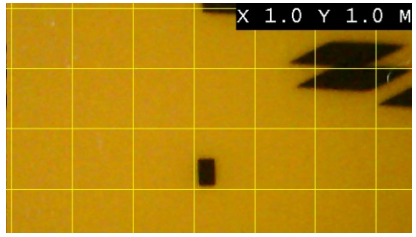
- J. Appl. Cryst. 54, https://doi.org/10.1107/S1600576721000601.

Residual stress measurement on inside surface of a tube





By reducing the spot size to 500 micron and tilting tube in chi orientation (side inclination) Stress measurement on inside surface in hoop stress direction could be performed



How it helps

This beamline will have unique facility for in-situ mechanical deformation measurments of metallic samples.

Recording X-ray diffraction patterns during deforming metals is a useful tool to get a deeper understanding of the coupling between microstructure and mechanical behavior and also determining X-ray elastic constants of unknown materials.

Observed mechanical behavior in loaded conditions would provide useful input for developing computational simulation tools used by engineers to predict performance of metallic components.

Thank you for attention