

# Grazing Incidence X-Ray Diffraction using Synchrotron Light at SLRI

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**Abstract.** This work demonstrates the capability of the grazing incidence X-ray diffraction technique using synchrotron light. The measurement system is set up at the BL7.2W:MX beamline of the Synchrotron Light Research Institute (SLRI). The beamline utilizes hard X-rays from a 6.5-Tesla Superconducting Wavelength Shifter. The photon energy can be chosen between 7 to 18 keV with a photon flux of more than  $10^{10}$  photons/sec at 100mA stored electron beam. The X-ray beam size can be reduced down to 20 microns, allowing XRD measurements in grazing geometry, thus crystal structures of very thin films with a thickness of tens nanometers can be identified. The diffraction patterns are recorded with a 2D CCD detector, allowing more diffraction spots of single crystalline films to be recorded.

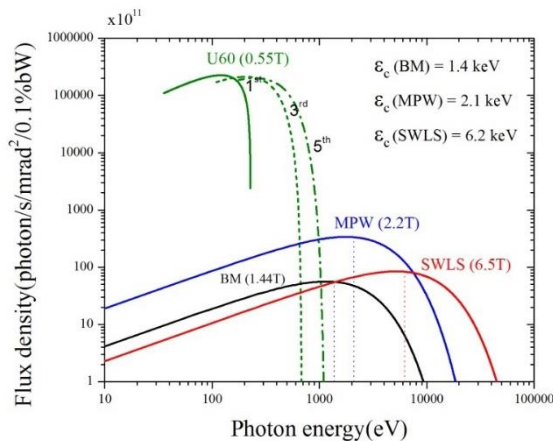
## 1. Introduction

X-ray Diffraction (XRD) is one of well-known techniques for investigating structural properties of materials. When X-ray passed through a crystal, it would scatter off the atom in the sample and then produce constructive interference at specific angle. Conventional X-ray diffractometers (powder diffractometers) typically work on Bragg-Brentano geometry [1], the incident angle is defined between the X-ray source and the sample, the diffraction angle ( $2\theta$ ) is defined between the incident beam and the reflect beam to the detector. In case of very thin films, grazing geometry is required to increase the interaction of X-ray with the materials in the films. The measurement with this geometry is known as grazing incidence XRD (GIXRD). GIXRD technique was first demonstrated by Marra et al. [2] for the study of crystalline surface and interface by varying the incidence angle of X-rays, which is typically very low angle, to get total X-ray external reflection from the sample. With GIXRD, the effective X-ray beam size on the sample surface is increased and the signal from a substrate is decreased [3]. X-ray from a synchrotron light source and from X-ray generator in laboratory may be employed for GIXRD measurements [4]. With the salient properties of synchrotron X-ray, there are several advantages when using synchrotron X-rays. The small size of synchrotron X-ray source allows GIXRD measurements to be performed in a small area on the sample. The intensity of synchrotron X-ray is much higher than rotating anode X-ray and, thus, the data collection time is much shorter when using synchrotron X-ray.

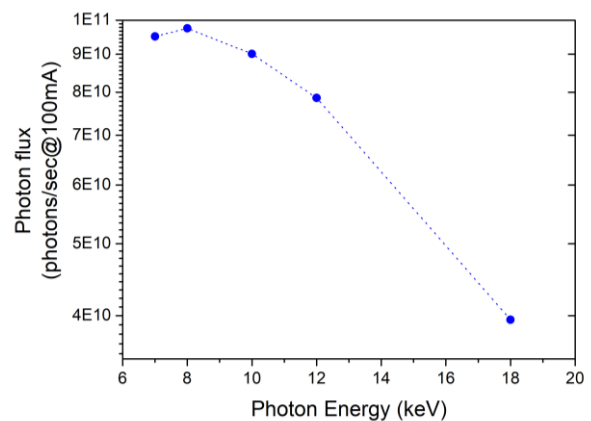
It is well known that synchrotron X-ray has a continuous spectrum, thus, one can choose any X-ray wavelength suitable for the materials being investigated. In this work, a GIXRD technique was developed at the BL7.2W:MX beamline of the Synchrotron Light Research Institute (SLRI). The measurement system was commissioned. The descriptions of the system and test results are given below.

## 2. Descriptions of BL 7.2W:MX Beamline

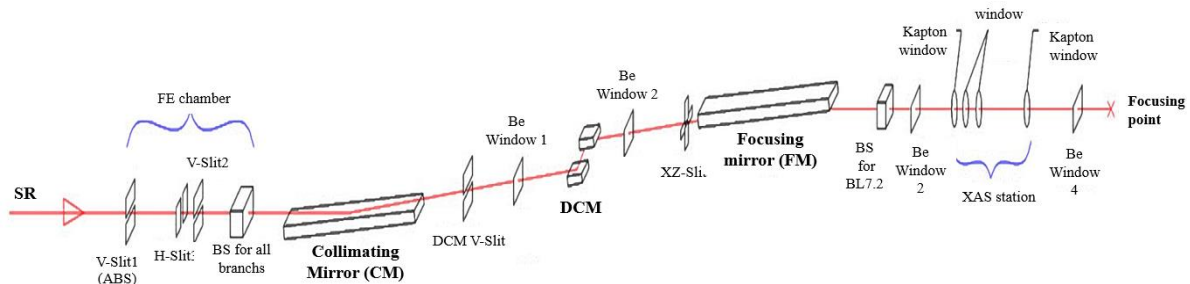
The beamline BL7.2W:MX utilizes synchrotron light produced from a 6.5 Tesla superconducting wavelength shifter (SWLS) installed in the 1.2-GeV storage ring of the Siam Photon Source (SPS) of SLRI. The calculated synchrotron spectra generated from SWLS, as well as from other devices, i.e. a linear undulator with a period of 60mm (U60), Multi-Pole Wiggler (MPW) and Bending Magnet (BM), are shown in figure 1 [5]. The actual photon flux measured before entering the beam shaping slits of the XRD set up is shown in figure 2. It is clearly shown that the photon flux from 7 to 18keV range is in the order of  $10^{10}$  photons/s, which is more than sufficient for general XRD measurements. The photon flux is varied with photon energy and maximum at about 8 keV. For GIXRD measurements, the beam size is reduced down to 20 microns by using the beam shaping slits in order to obtain small beam area on the sample



**Figure 1.** The SPS spectral flux densities [5]

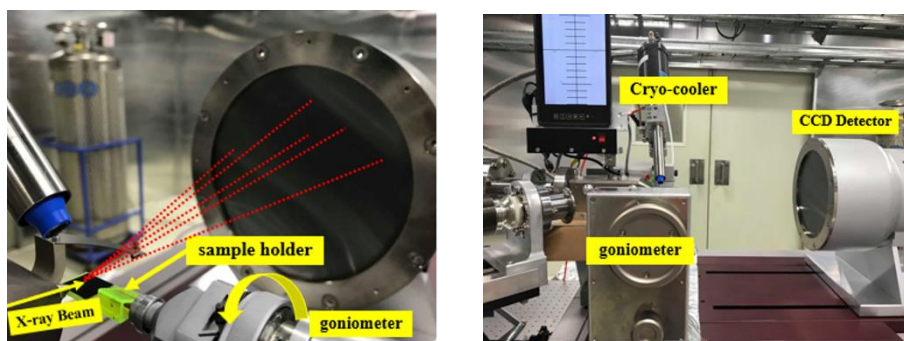


**Figure 2.** Measured photon flux before entering the beam shaping slits of the XRD measurement system.



**Figure 3.** Optical layout of the BL7.2W: MX beamline

The optical layout of the BL7.2W:MX beamline is shown in figure 3. Major optical elements of the BL7.2W:MX beamline such as a cylindrical collimating mirror (CM), a commercial double-crystal monochromator (DCM) and a toroidal focusing mirror (FM) are used to obtain a focused hard X-ray beam with required characteristics for diffraction experiments of crystalline samples.



**Figure 4.** Photo of the experimental station of the BL7.2W:MX beamline.

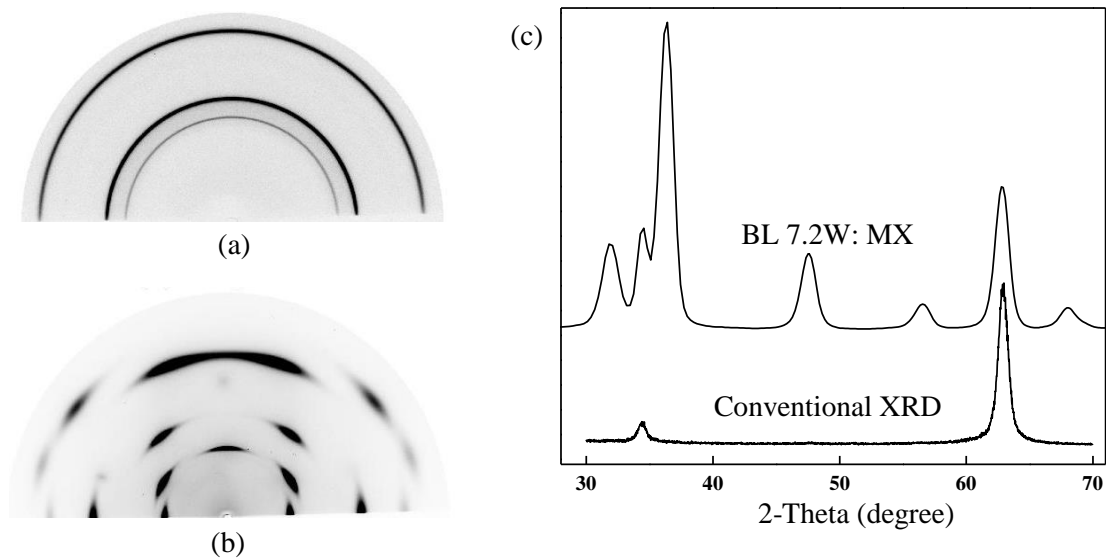
Figure 4 shows the experimental station of BL7.2W: MX beamline which consists of cryo-cooler, sample holder and Mar165 CCD detector system (Rayonix LLC, Evanston, Illinois, USA). The sample holder and the detector are mounted on a MarDTB Goniometer system (Marresearch GmbH, Norderstedt, Germany). Taking the advantage of accurate controlling of a high precision phi-axis of MarDTB goniometer system, the sample can be tilted/rotated to adjust the angle of incidence between  $0^\circ$  to  $360^\circ$  with accuracy  $0.002^\circ$ . Thus, diffraction measurements with geometry is doable without any difficulties. The distance between the sample and detector can be varied between 45 to 390 mm. In addition, the detector can be rotated around the sample with the maximum angle of 30 degrees. The obtained 2D diffraction images may be processed with various 2D image processing computer programs, e.g. SAXSIT which was developed for small angle X-ray scattering data analyses [6].

### 3. Commissioning results and Discussion

The commissioning of the GIXRD set up was carried out on germanium antimony tellurium (GST) and aluminium doped zinc oxide (AZO) thin films prepared by using pulsed DC magnetron sputtering technique. The 4-bromo benzoic acid was used as a calibration standard to determine the accurate distance between the sample and detector. The diffraction images were collected using a monochromatic X-ray beam with a wavelength of  $1.291 \text{ \AA}$  (9,600 eV) with an appropriate exposure time varying depending on the sample materials and sample-to-detector distance.

Figure 5(a) and (b) show the diffraction image taken from GST and AZO thin films. Grazing incidence angle was fixed at  $1.0^\circ$  for GST thin film and  $0.6^\circ$  for AZO thin film, respectively. The distance of sample to the detector was fixed at 70 mm. The collection time for both samples was 2 min. The full Debye ring in figure 5(a) indicates that the X-ray beam falls on a sample of tiny crystals in all orientations, implying that the sample has a perfect polycrystalline texture [7]. Whereas the diffraction image taken from the AZO thin film appears as a partial ring, as in figure 5(b). Thus, this AZO film is not polycrystalline. The film was grown with a preferred orientation. With a 2D detector used at the beamline, one can collect diffracted beams in 2 dimensions. It is clearly shown in figure 5(c) by comparing data from the beamline and from a conventional system that some diffracted beams cannot be collected by a conventional laboratory GIXRD system using an X-ray tube.

The high intensity of synchrotron X-ray requires a short data collection time. The diffraction images/patterns in figure 5 were taken with collection time of 2 and 45 min at the synchrotron beamline and conventional GIXRD laboratory system, respectively. In addition to the advantage of a short data taking time, the synchrotron GIXRD can be carried with any X-ray wavelength from 7 to 18 keV, thus depending on specific requirements suitable for different sample materials.



**Figure 5.** 2D GIXRD diffraction images of (a) GTS, (b) AZO thin films and (c) GIXRD patterns of AZO film measured at the BL7.2W:MX beamline (top) and conventional GIXRD system (bottom).

#### 4. Conclusion

The advantages of synchrotron GIXRD at SLRI over conventional XRD systems have been demonstrated. Higher photon intensity and tunability of incident X-ray of synchrotron GIXRD are useful. In addition, a 2D detector available at the synchrotron beamline allows more types of materials to be investigated.

#### 5. References

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