Application Note

X-RAY STRUCTURAL CHARACTERIZATION FACILITY

XRD SML IPF 12-15

NCMN
Nebraska Center for Materials and Nanoscience
University of Nebraska

Orientation distribution of crystallographic texture from pole-figure measurement

Dr. Shah Valloppilly, NCMN Specialist

NCMN X-ray Scattering Facility's Rigaku SmartLab instrument is a multifunctional diffractometer capable of high-resolution diffraction, in-plane pole-figure analysis, reciprocal-space mapping, x-ray reflectivity, grazing Incidence in-plane x-ray diffraction, small angle x-ray scattering etc. [1]. Some unique features of this instrument include SmartLab Guidance software assistance to select an application and set up the appropriate hardware, automatic optics and sample alignment, an additional arm for the in-plane motion of the detector without the need for tilting the sample. This application note demonstrates the in-plane pole figure measurement of a MnBi thin-film sample.

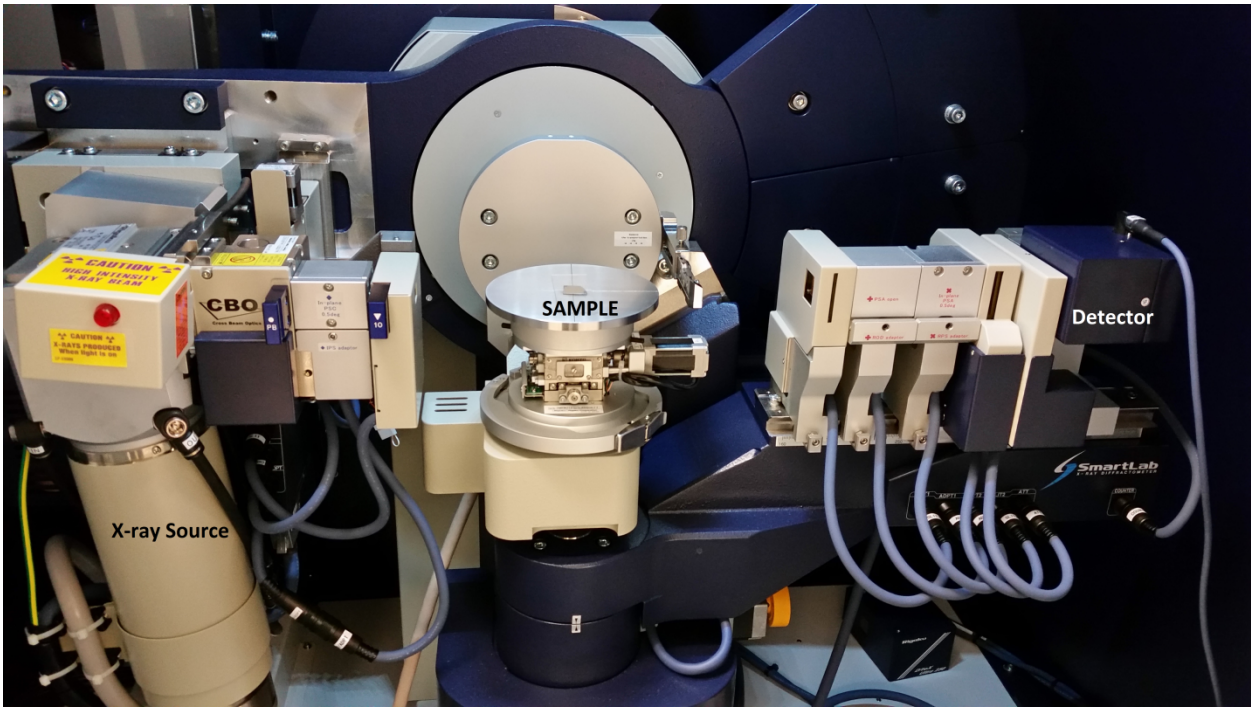

Fig. 1: Photograph of Rigaku SmartLab goniometer set up for in-plane pole-figure measurement. Copper K_α radiation (λ = 1.5418 Å) is used.

The parallel beam produced by the Cross-Beam Optics (CBO) device is delivered by the parallel-beam (PB) slit. An in-plane parallel-slit collimator of 0.5° is used to control the axial divergence of the incident beam. The sample is placed on a 12 cm diameter wafer sample plate attached on top of a sample stage capable of tilt corrections and height adjustment. The tilt stage (R_x-R_y stage) aligns the sample surface parallel to the x-ray beam and the Z-translation positions the sample at the right height accommodating samples of up to 3 mm thickness.

In this application note, pole-figure analysis of two prominent crystallographic textures of $Mn_{50}Bi_{50}$ (Fig. 2a: hexagonal structure, $P6_3/mmc$, a =4.29Å, c = 6.12Å) thin film is presented. In a conventional x-ray diffraction (Fig. 2b) probing inter-planar distances along the surface normal, the films exhibits a predominant (001) texture. Presence of only (00ℓ) reflections may indicate single-crystalline or a highly textured film.

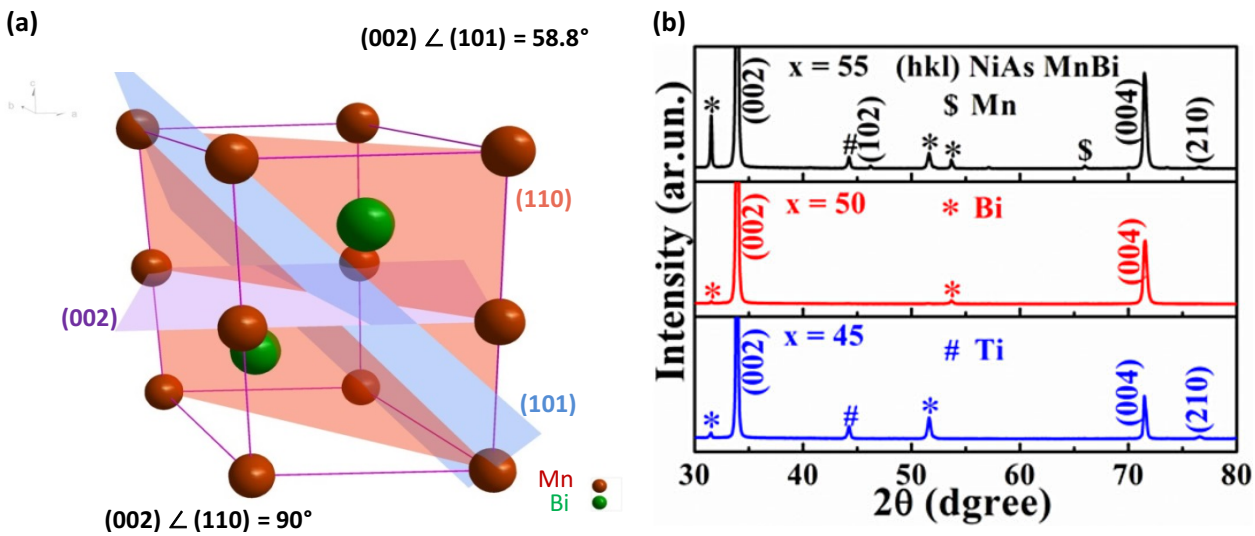

Fig. 2: (a) Hexagonal unit cell of MnBi illustrating (110), (101), (002) planes and the angle between them (b) x-ray diffractograms of Mn_xBi_{100-x} (x = 45, 50, 55) thin films indicating strong (001) texture. Pole-figure was measured on x = 0 sample for planes (101) and (002) [2].

In-plane pole figure measurements are performed by collecting diffracted beam intensity of a Bragg reflection as function of sample rotation (β= 0 - 360°) angle at various tilt angles of the sample surface normal (α = 0 – 90°). The intensity $I(\alpha,\beta)$ plotted as a 2-dimensional map is known as pole-figure and is a representation of the distribution of a crystallographic orientation in space. In pole-figure representation, the tilt angle α varies radially and the rotation angle β varies circularly as shown in Figs 3a & b, respectively, of {001} and {101} set of planes. A sharp, narrow distribution of {001} pole intensity indicates the high degree of orientation of this direction parallel to the surface normal. It is also remarkable to note that the {101} pole plot shows a narrow uniform distribution of intensity, rotationally symmetric to the surface normal as in a fiber texture. A line profile analysis of {001} and

{101} pole plots in Fig 3(c) shows the intensity distribution as a function of tilt angle with respect to the film surface normal. The tilt angle of ~ 60° estimated from pole figure analysis is very close to the calculation of inter-planar angle between (002) and (101) planes i.e., 58.8°. The results indicate that there is a very high coherence of atomic order perpendicular to the film plane whereas, in the lateral direction, the planes are randomly oriented. In this example, texture analysis helps to understand the growth mechanism of thin films that has direct relationship to its magnetic properties.

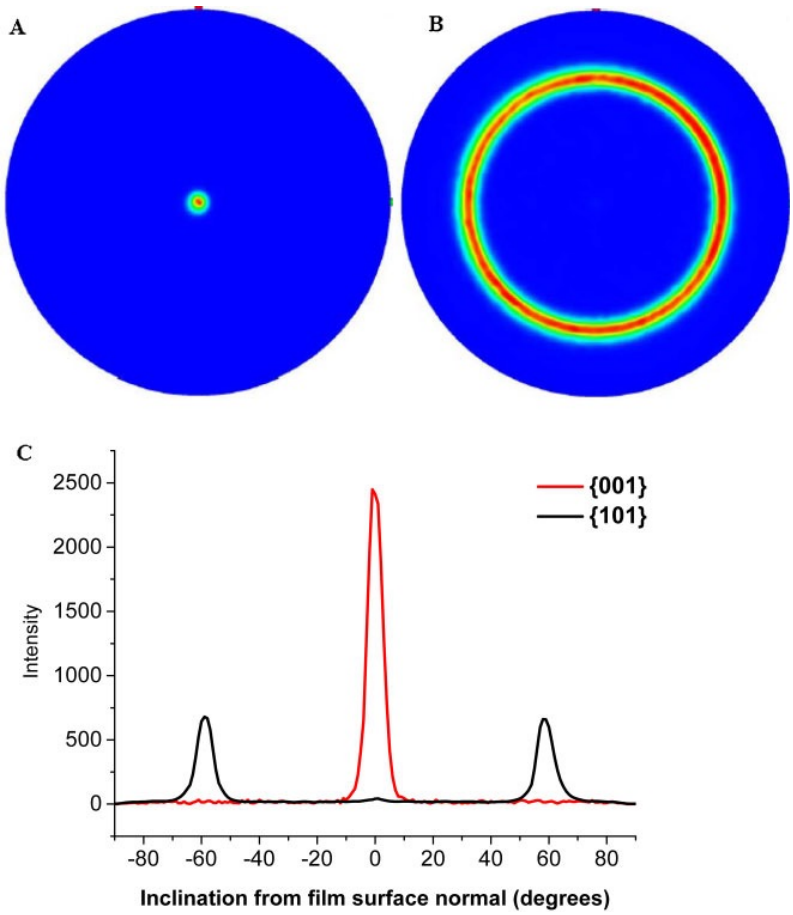

Fig. 3: Pole-figure plots of (A) {001}, and (B) {101} set of planes in MnBi thin film. Line profiles (C) along the radial direction represents the tilt angle vs. intensity for both orientations.

Pole figure analysis provides valuable information on the crystallographic texture that may be related to physical, chemical and mechanical properties of materials. The SmartLab diffractometer provides the in-plane pole figure option to study texture properties of films, fibers and bulk samples. This option is a unique method to obtain full coverage of pole figure without the need for tilting the sample.

References

1. The Rigaku Journal **27** (2), p 6 (2011)
2. High-energy-product MnBi films with controllable anisotropy, Wenyong Zhang et. al., Phys. Status Solidi B **252**, p 1934 (2015)