Thin Film / GIXRD / Reflectometry
GeniX CU High Convergence

Xenocs® Brighter solutions

Application Note n° AN-G11

Abstract

The GeniX Cu High Convergence system was tested at the off-line x-ray characterization laboratory in CEA, LETI, MINATEC on a diffractometer made of a 4 circle diffractometer for thin film XRD and XRR applications.

We present results in Grazing Incidence X-ray Diffraction applications coupling the GeniX Cu High Convergence with a Position Sensitive Detector 1D detector. The results were compared to those obtainable using a standard diffractometer equipped with a standard sealed tube (1,6 kW) and using a 0D detector in a scanning mode. The same GeniX can be used for reflectometry measurements and we provide results in comparison with a standard reflectometer. Comparative results reveal good data quality for the GeniX configuration with additional small spot capability.

Grazing Incidence X-ray diffraction and Reflectometry

Data courtesy of Dr Patrice Gergaud, CEA-LETI,France.

Introduction

Microfocus sealed tube systems are now increasingly used in single crystal X-ray diffraction and small angle X-ray scattering applications benefiting from high brightness X-ray beam, low maintenance and high reliability. The use in thin film applications is however still limited, in particular due to the high efficiency of standard 1D X-ray source set-ups (high power sealed tube coupled to 1D Goebbel mirrors) for large area analysis but as well due to the use of standard analyzing configurations based on 0D detectors in scanning mode.

To demonstrate the capability of the GeniX beam delivery system for XRD and XRR thin film measurements, XENOCS installed a GeniX Cu High Convergence on a diffractometer of CEA, LETI. We present the results of Grazing Incidence-XRD measurements carried out with a Position Sensitive Detector (INEL detector) which illustrate the capability to do fast measurements with good accuracy as well as with high spatial resolution providing mapping capability. The same system can be used for powder diffraction (phase analysis), or X-ray reflectometry measurements.

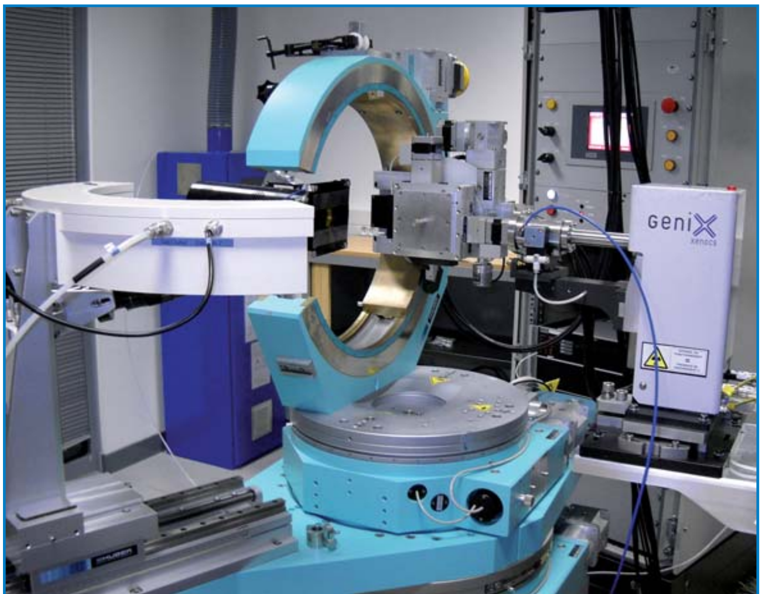

Fig. 1 : 4 circle diffractometer in GIXRD configuration at CEA, LETI, MINATEC.

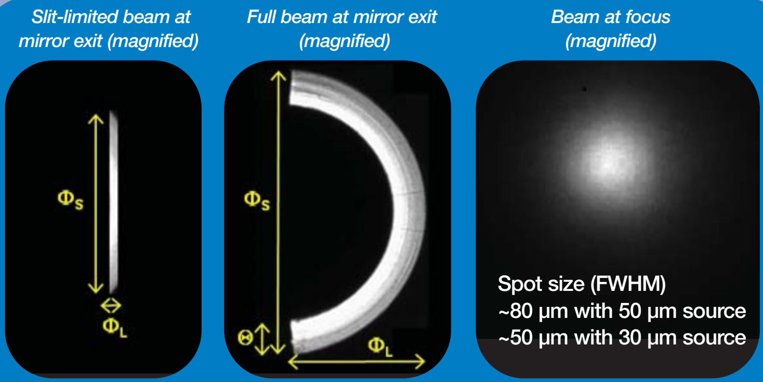

Fig. 2 : CCD images of the X-ray beam of GeniX Cu HC. The GeniX for GIXRD measurements uses a linear slit (left image) to limit divergence compared to full beam (right image)

Table I *Beam Properties of the Genix Cu HC for the full beam configuration and the silt limited configuration which is the one used in our GIXRD experiments.*

Wavelength	1.54 Å / 8 keV (Cu Ka)	
Kb contamination	< 0.3%	
Beam configuration (customizable)	Full beam	Slit-limited Beam
Typical flux (vacuum, 50W/50µm source)	> 600 x 10^6 phs/s	> 30 x 10^6 phs/s
Beam Convergence (Φ_L, Φ_S and Θ are defined in Fig.3)	$\Phi_S \geq 4°$ $\Phi_L \geq 2°$ $\Theta = 0.54°$	$\Phi_S = 2.6°$ $\Phi_L = 0.05°$
Spot size at focus (FWHM, 50µm source)	~80 µm	~80 µm
Spot size at focus (FWHM, 30µm source)	~50 µm	~50 µm

Table II Description of both GIXRD set-ups compared

	Configuration A GIXRD scanning detection mode	Configuration B GIXRD Static detection mode
Source	Long Fine Focus Sealed Tube (1,6 kW)	GeniX microfocus source (50W)
Illumination optics	Goebel 1D mirrors	FOX3D CU 21_21HC
Incident flux*	550 Mph/s 7 by 10 mm² (sample projection)	40 Mph/s on 80 µm by 300 µm (sample projection)
Data acquisition	Scanning 0D detector (0,03°/s)	Static Mode with Inel Gas Detector (120° collection)

* Incident flux for total beam path in air.

Experiment

The diffractometer of CEA-LETI that was used for the experiments is a multi-application set-up (XRD/XRF) made of a 4 circle goniometer from HUBER and providing the capability to adapt different XRD detectors (0D, 1D, 2D detectors). Moreover this diffractometer is particularly adapted for thin film analysis in semiconductor applications with a high precision mechanics (very small sphere of confusion) and an adapted sample stage for sample mapping enabling thin film measurements on small test structures (lower than 100 microns).

The GeniX Cu HC was mounted on the diffractometer equipped with a Position Sensitive Detector (INEL CPS 120 Curved gas detector) placed at a distance of 250 mm from the sample stage (Fig.1). The GeniX Cu HC is equipped with a unique FOX3D 21_21 HC focusing multilayer optic with an ellipsoidal shape of half-revolution providing high solid angle of collection in sagittal plane. The optic is mounted with a linear slit producing a highly collimated beam in one plane while keeping high convergence in the other plane. When coupled to microfocus sources, the optic is particularly adapted for small spot one-dimensional XRD applications such as GIXRD, powder diffraction or X-ray reflectometry. Figure 2 illustrates the beam properties of the FOX3D Cu 21_21HC optic with linear slit used in the GeniX and Table I gives the typical beam parameters (flux and divergence of the optic).

Requirements on incidence X-ray beam for GIXRD depend on the detection mode. When a static detection mode with a PSD detector is used, it is convenient to have a small X-ray beam in the equatorial plane (parallel to the surface normal direction). Indeed a small X-ray beam in this direction will reduce the beam broadening and consequently maintain a sufficient angular resolution for the detection with a PSD detector having a wide angle of detection.

The GeniX Cu HC with a linear slit fulfills these requirements in the vertical plane whereas the focused beam in the other dimension enables surface mapping with high spatial resolution while maintaining a high flux due to the high solid angle of the optic.

Results on GIXRD measurements

GIXRD tests were carried on a thin Pt layer of 100 nm with a grazing incidence angle of 15° (Figure 2). Results are compared to GIXRD measurements carried on with a standard diffractometer equipped with a standard sealed tube configuration (1,6 kW line focus sealed tube coupled to Goebbel Mirror) and with a 0D detector coupled to a parallel plate collimator (0.27 degree of acceptance). Table II compares both set-ups measurement conditions.

Figure 3 shows comparative results achieved with the two set-ups on the same sample. The collection time has been adapted to get the [100] peak at 2θ=39.7° of same intensity for both configurations. In the configuration A (scanning), each channel is accommodated for 1s whereas for the configuration B (static), all channels are accumulated simultaneously. Whereas statistics (counts /seconds and per channel of 0.03°) are better with the GIXRD scanning mode, measurements were still of very good quality due to the high flux density of the GeniX and low noise of the gas detector. It is to be noted that the flux level achievable with the GeniX Cu HC can be modified with a linear slit of increased aperture at the expense of angular resolution. A flux increase by a factor of 2-3 is possible while maintaining an angular resolution comparable with the standard GIXRD set-up.

Angular Resolution is even better with the GeniX as illustrated in Figure 3 (0,39° with the scanning GIXRD diffractometer of configuration A, compared to 0,26° with the GIXRD set-up including the GeniX of the configuration B). Of course, in the static mode, decreasing the incident angle will decrease also the angular resolution. For instance at 5° of incidence (footprint of about 1 mm length), the peak width is broadened of 0.3°, which is still reasonable. Moreover total measurement time was significantly lower (16 X) for the GeniX configuration illustrating the advantage of using a Position Sensitive Detector for wide angle measurements such as in typical powder diffraction measurements. Data acquired with the GeniX is really comparable to data acquired with the standard diffractometer used in a scanning mode. ***Therefore, the asset of the Genix setup is to perform blanket film analysis with the advantage of avoiding mechanical movement of the detector, reduced experimental time and with the benefit of having a small beam footprint.*** This feature allows avoiding spurious signal arising from undesired areas, and simply avoids the sample preparation. As a bonus, footprint is also small enough to offer mapping capability. With a 15° angle of incidence the spot size on the sample plane with the GeniX was 310 µm by 80 µm compared to 10 mm by 7 mm beam with the sealed tube system. This is illustrated on Figure 4 by representing the data obtained and normalized by the illuminated area. This emphasizes the advantage of using the configuration B for mapping.

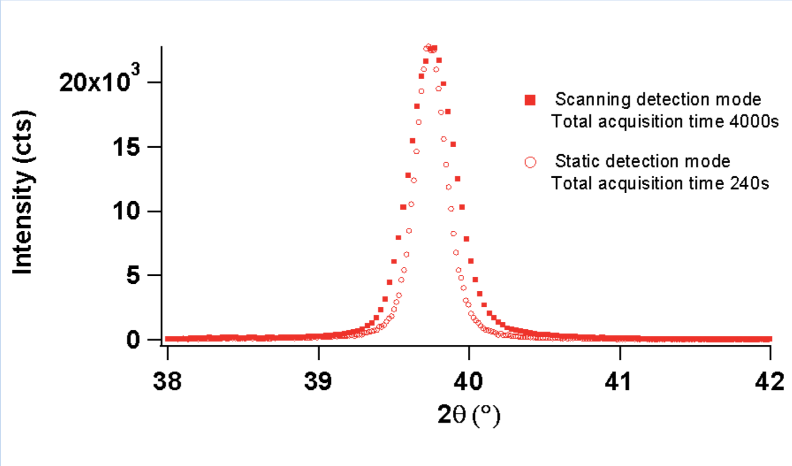

Fig. 3 : Comparison of both methods in term of resolution. It shows a peak width of 0.39° and 0.26° for the scanning and static mode respectively. Intensity is expressed in total counts (cts). Scanning mode, configuration A: (■) Sealed tube Cu source + 1D mirror, thin film collimator, detector 0D, step by step mode and total acquisition time 4000s. Static mode, configuration B: (○) Genix Cu source + FOX3D 21-21, collimating slit 0.05°, curved detector 1D, fixed and total acquisition time 240s.

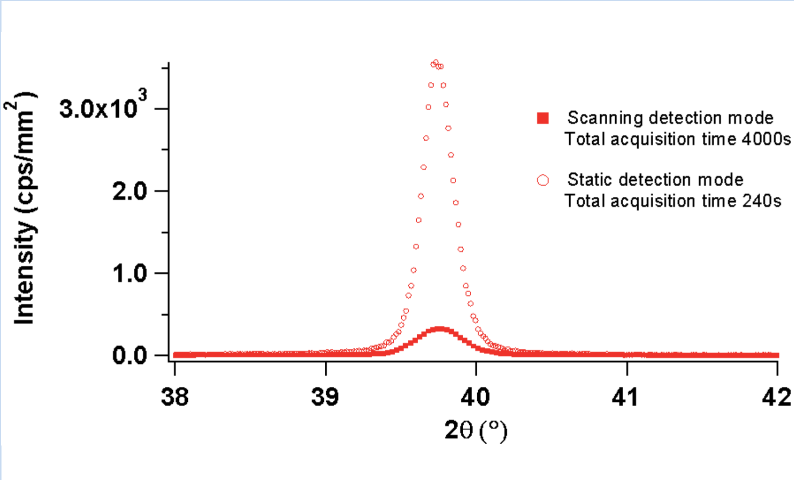

Fig. 4 : Comparison of GIXRD measurements on a Pt film with representation of data normalized by the illuminated area and exposure time of 1s per channel of 0.03°. Intensity is expressed in counts per sec. and per mm².
Scanning mode, configuration A: (■) Sealed tube Cu source + 1D mirror, thin film collimator, detector 0D, step by step mode and total acquisition time 4000s.
Static mode, configuration B: (○) Genix Cu source + FOX3D 21_21HC, collimating slit 0.05°, curved detector 1D, fixed and total acquisition time 240s.

Results on reflectometry measurements

Table III Summary of the results obtained with various test multilayer samples

Sample	Multicouche xN layer	d (Å)	Reflectometer	d_{RAVET} (Å)	d_{HENKE} (Å)
A (flat)	$(Mo/B_4C)_{x50}$	50	SEIFERT XRD 3003 TT	49,97	49,97
			GeniX 21-21-HC	50,00	50,00
B (flat)	$(Cr/B_4C)_{x30}$	35	SEIFERT XRD 3003 TT	35,56	35,56
			GeniX 21-21-HC	35,45	35,45
C (curved)	$(Mo/Si)_{x32}$	73	SEIFERT XRD 3003 TT	ND	ND
			GeniX 21-21-HC	71,23	71,41

Experiment of reflectometry can be conducted using similar illumination setup as the configuration B here above described (Table II). The data collection was in this latter case collected in scanning mode using a 0D detector, as described below. The low divergence of the X-ray beam defined by the slit at the exit of the mirror allows a good compromise between intensity and angular resolution. Results of experiments conducted on two flat multilayer samples and one concave multilayer mirror and investigated in scanning mode are presented. The beam used was similar to previous GIXRD experiment, with a spot size of 80µm FWHM and a slit-defined divergence of 0.05°. Scanning has been performed in omega (sample) - 2theta (detector) configuration with a point detector from Oxford Instrument and a 2-theta mechanical step of 0.02°. Figure 5 represents the typical reflectivity curve obtained on a flat CrB4C and a MoB4C with respectively 50Å and 35Å of period. The data were analysed either with Ravet or Henke refinement [1] and results are summarized in Table III and are compared with data obtained on classical scanning XRR setup based on Cu radiation long fine focus source, a collimated channel cut and point detector (Seifert XRD 3003 TT). This classical setup provides approximately 12mm wide footprint and useful flux on the sample in the range of 1 Mph/s. Typical measurement times for the Seifert system is in the range of 1,5 hour. Those values are to be compared with the GeniX system detailed in Table I : 80 µm wide footprint, 26 Mph/s and typical measurement times in the range of 10min.

As seen on the figure, the Kiessing fringes shown in the insert of Figure 5 are present but not clearly observed due to the choice of the detector slit, driving a divergence of 0.05° and of the choice of the mechanical scanning step size of 0.01°. However, higher resolution can be readily obtained with a thinner slit and a finer step scan resolution. Finally, it has been possible to determine accurately the periodicity of a multilayer with a *measurement of the XRR curve in a concave spherical cap sample*, with typical radius of 6m. *Such a concave sample is absolutely impossible to be investigated in standard reflectometer due to long fine focus source where compromises on flux are impractical. The value of the period as obtained is to be compared with the target value measured on the secondary sample flat deposited.*

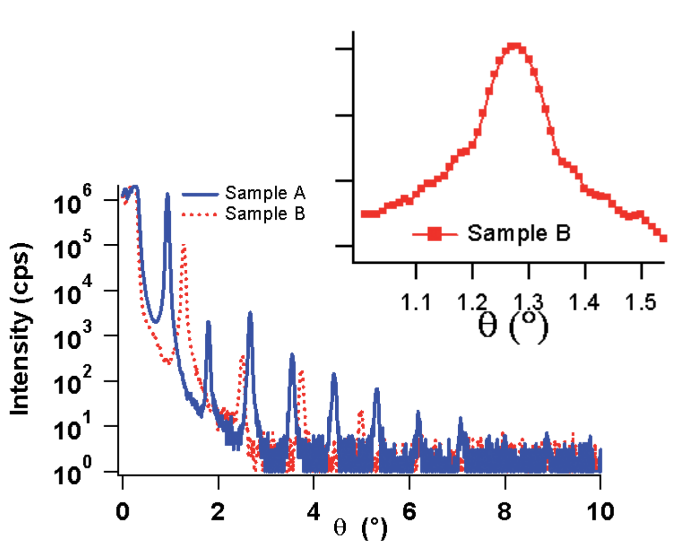

Fig. 5 : Reflectometry curve obtained in omega-2theta scan. Exposure time is of 1s per step. Insert is a zoom on the [1.0-1.55]° range of the sample B curve. Insert depicts a close view of sample B curve around theta=1.25°

Conclusion

The GeniX, with its high efficiency and well controlled X-ray beam, enables an efficient coupling with Position Sensitive Detector for GIXRD measurements with good signal to noise measurements, possibly very high angular resolution and additional small spot mapping capability. Besides classical blanket analysis, applications such as phase analysis, grain size study can be done efficiently with such set-up. The same GeniX Cu 21_21HC can also be used for fast reflectometry measurements whereas 2D diffraction applications (texture) on small spot or high resolution XRR can be done with modified optical slits. The main asset of this setup lies also in the unique capability for reflectometry measurement on non-flat substrates. Additionally, the low power tube consumption (50 Watts) offers ease of integration, low cost of ownership, a reduced carbon footprint and low maintenance.

[1] Henke B.L. *et al.,* J. of X-ray Sci. and Technol. 2(1), 17-80 (1990)

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