Characterization of Crystal Structure and Morphology of Ultra-Thin 2D MoS2 Layers Using X-ray Metrology

Lixia Rong, Hao-Ling Tang, Luc Thomas, Hanson Kwok, Michael Phillips, Hongwen Zhou, Qinyi Fu, Lavinia Nistor, Mahendra Pakala

Contact Author Email Address: Lixia_Rong@amat.com

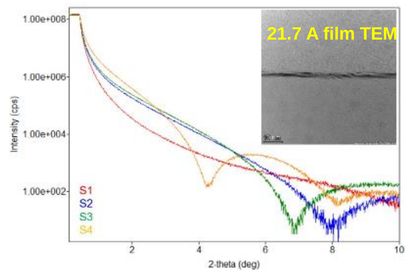
Introduction

- Few-layered two-dimensional transition metal dichalcogenides (2D TMDs) are strong candidates for several applications such as high mobility channel in memory or logic devices, photovoltaics, photodetectors, and optoelectronics
- Since high-quality ultra-thin layers are needed to take full advantage of these 2D materials, precise control of the crystallinity, morphology and thickness of these ultra-thin layers is critical. This requires a set of metrology techniques highly sensitive to these ultra-thin layers and fast enough to provide rapid feedback to help optimize deposition parameters

AMAT Solution

- We use a combination of X-ray scattering and fluorescence metrology techniques, including grazing incidence X-ray diffraction (GI-XRD), in-plane X-ray diffraction (In-plane XRD), X-ray reflectivity (XRR) and wavelength dispersive X-ray fluorescence (WDXRF) to characterize MoS2 samples of various thicknesses
- We show that this fast and non-destructive X-ray based metrology is sensitive to crystal phase, crystallite grain size, thickness, density, composition, and uniformity of the MoS2 films down to sub-nm thickness

XRR Data of MoS2



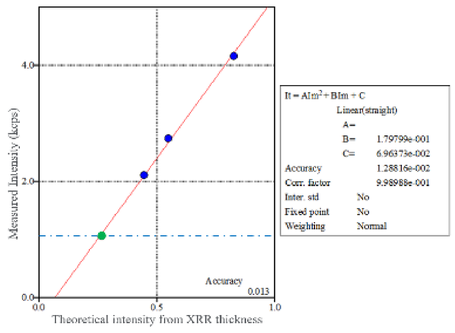
- XRR data of 4 MoS2 films with 21.7 A film TEM image of shown in inset
- S2-4 show clear thickness fringes and can be fitted unambiguously to derive film thickness and density
- XRF can be used to complement XRR when film is thinner than 1nm (S1)

GI and in-plane XRD grain size, XRR fitting thickness, density and WDXRF counts of Mo-La & S-Ka

Sample ID	GI-XRD grain size (A) (002)	In-plane XRD grain size (A) (100)	XRR fitting thickness (A)	XRR fitting density (g/cm3)	WDXRF Mo-Lα (kcps) 2.29 keV	WDXRF S-Kα (kcps) 2.31 keV
Film 1	NA	NA	*7.0*	5.9	1.05	1.19
Film 2	19	92	11.7	6.0	2.10	3.06
Film 3	21	86	14.4	6.2	2.74	4.20
Film 4	30	161	21.7	6.1	4.16	6.87

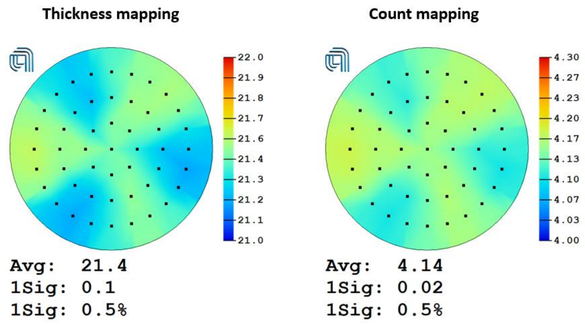
WDXRF FP Method Calibration

- WDXRF is necessary since it has higher energy resolution than energy dispersive XRF (EDXRF)
- WDXRF intensity counts were calibrated with XRR fitting thickness for S2-4
- In FP method, the theoretical intensity calculated based on the XRR fitting thickness is correlated with the measured XRF counts



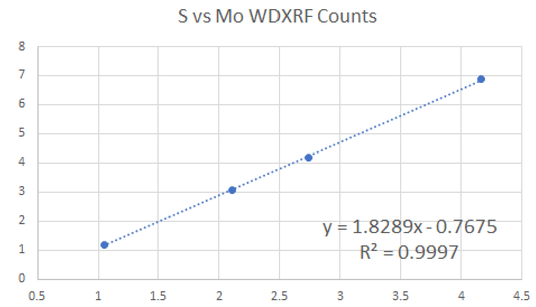
Fundamental parameter method calibration between calculated intensities and WDXRF measured counts.

WDXRF Mapping & Uniformity Data



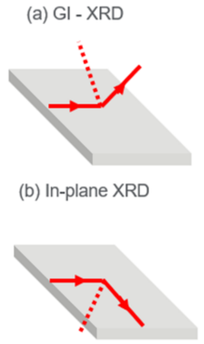
49-point WDXRF thickness & count mapping of sample S4 (thickness 21.7 A) indicates good film uniformity

WDXRF Consistent Composition Data



WDXRF S-Ka & Mo-La counts good correlation shows constant composition for all 4 films

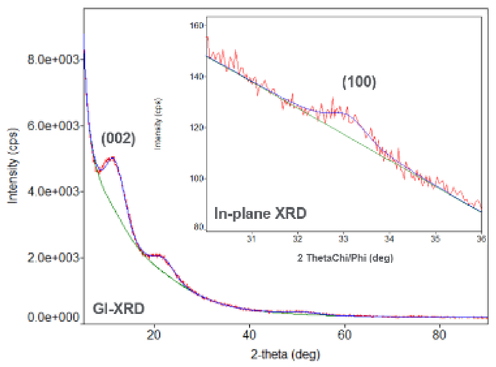
XRD Configuration of Thin Film



- GI & In-plane techniques are preferred for ultra-thin films because they provide higher film intensity relative to Si substrate background noise than the conventional θ-2θ configuration
- GI-XRD provides information of any existing randomly oriented lattice planes of the film
- For in-plane XRD configuration, the measured diffraction intensities are from the lattice planes normal to the sample surface, so the technique is more sensitive to in-plane crystallinity and grain size

Schematics illustrate of the XRD measurement geometries of the MoS2 films. XRD data usually measure scatted X-ray intensity as a function of ω and/or 2θ.

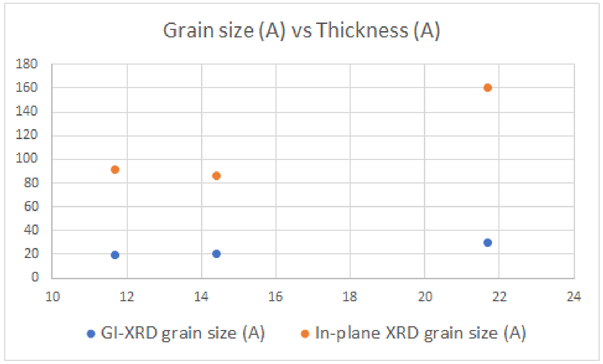
XRD Data of MoS2



- Polycrystalline hexagonal beta phase MoS2
- GI-XRD 002 grain size 19A
- In-plane XRD 100 grain size 92A
- Large in-plane grains are critical for MoS2 application in memory or logic device channels

GI-XRD 2θ scan with incident angle 0.5° for S2 (thickness 11.7A). In-plane 2θχ/φ scan with incident angle 0.35° is shown in inset.

XRD Grain Size & Thickness of MoS2



GI & In-plane XRD grain size analysis shows that in-plane grain size is larger and increases significantly with 2D MoS2 film thickness

Conclusion

- XRR combined with WDXRF are suitable for characterization of 2D MoS2 films in a wider range of thicknesses
- In-plane XRD is sensitive to MoS2 lateral grain size which is a key performance driver for 2D MoS2 based devices
- This suite of X-ray based metrology techniques offers a fast, non-destructive, and sensitive methodology for in-depth characterization and optimization of ultra-thin layer 2D materials