TLS 24A1 XPS, UPS, XAS, APXPS

- XPS, UPS
- Materials Science, Batteries, Catalysts

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Hold the Sun in the Palm of Your Hand

Microbeam GIWAXS is used in the study of solar cells.

A thin film is a layer of material deposited or coated on substrates with a thickness in the range of nanometers to a few micrometers. X-ray scattering methods are frequently used for thin film analysis. Due to the thickness of thin films and the existence of substrates, the transmission geometry of small-angle X-ray scattering (SAXS) and wide-angle X-ray scattering (WAXS) is limited. Therefore, grazing-incidence small-angle X-ray scattering (GISAXS) and grazing-incidence wide-angle X-ray scattering (GIWAXS) are extensively applied in thin film structural analysis. In the geometry of grazing incidence, an X-ray beam impinges on the surface of a thin film with an angle close to the total reflection angle, thereby increasing the penetration path in the sample. In-plane and out-of-plane structures of thin film are extracted from X-ray scattering patterns that are collected by two-dimensional X-ray detectors.

TPS 25A is capable of performing transmission and grazing-incident SAXS/WAXS. The total X-ray photon flux at the sample position is approximately 10^{12} photons/second and the vertical and horizontal beam sizes at the sample position are approximately 3 and 5–10 μm, respectively. With the typical incident angle (*i.e.*, 0.05°), the footprint on the thin film is about 5 mm. The sample stage is composed of a hexapod and a stand for thin films. The six-axis positioning system provides with submicron precision and rigid stability. For scattering pattern collection, the instrument is equipped with two area X-ray detectors, a Dectris EIGER X 1M and 16M. The 16-megapixel detector is for SAXS/GISAXS and the smaller, 1-megapixel detector is for WAXS/GIWAXS. With the incident X-ray photon energies in the range of 5.4 to 20 keV, the q-range with the small-angle detector covers 0.0007 to 0.7 $Å^{-1}$. An order-of-magnitude of two in the q-range can be achieved in one acquisition. For wide-angle experiments, the q value is up to 5 $Å^{-1}$.

For an isotropic SAXS sample, the magnitude of the momentum transfer can be expressed as:

$$q = \frac{4\pi}{\lambda} sin\left(\frac{2\theta}{2}\right)$$

where 2θ is the scattering angle and λ is the wavelength ofthe incident X-ray. In grazing-incidence geometry, the vector components must be taken into account:

$$q_x = \frac{2\pi}{\lambda}\left[cos\,cos\,(\alpha_f)\,cos\,cos\,(2\theta_f) - cos\,cos\,(\alpha_i)\right]$$

$$q_y = \frac{2\pi}{\lambda} cos\,cos\,(\alpha_f)\,sin\,sin\,(2\theta_f)$$

$$q_z = \frac{2\pi}{\lambda}\left[sin\,sin\,(\alpha_f)\,sin\,sin\,(\alpha_i)\right]$$

where x, y, and z are the in-plane direction, beam direction, and out-of-plane direction, respectively, α_i is the incident angle, α_f is the out-of-plane scattering angle, and $2\theta_f$ is the in-plane scattering angle.

Recently, Wei-Guang Diau's group at National Yang Ming Chiao Tung University utilized the GIWAXS technique in their perovskite solar cell (PSC) studies at **TPS 25A**.[1,2] PSCs are solar cells that utilize a perovskite-structured compound as a light adsorber. Originally, the $CaTiO_3$ crystalline structure was used; however, more recently the general perovskite structure ABX_3 is utilized, which comprises either a hybrid organic–inorganic or tin halide-based material. In comparison with silicon-based solar cells, the fabrication processes of PSCs use a relatively simple coating method and baking temperatures are generally under 150°C.

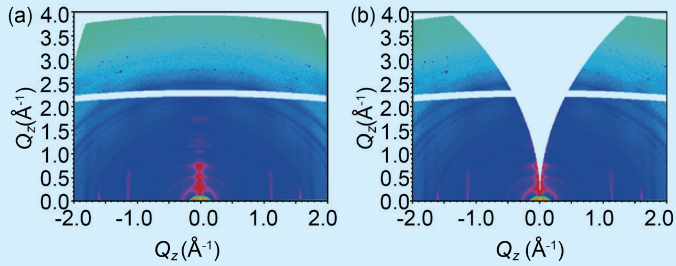

Fig. 1: (a) Raw GIWAXS pattern; (b) GIWAXS pattern with pole figure correction. [Reproduced from Ref. 1]

The new series of pyrrolopyrrole-based polymers were synthesized as hole-transporting materials for Sn-based PSCs by Diau's group. The GIWAXS patterns of the thin film samples showed good consistency with their X-ray diffraction data. The orientations of the tin-perovskite deposited on different hole-transporting polymers was almost isotropic. The research group also investigated the addition of different co-cations in the two-step fabrication of tin-based PSCs. An additional δ-phase (the tetragonal lattice) was observed from the normal cubic perovskite structure in GIWAXS patterns of specific co-cationic perovskite thin films. The GIWAXS technique at **TPS 25A** can directly assist the users in measuring crystalline properties of thin film devices. (Reported by Jhih-Min Lin)

This report features the work of Wei-Guang Diau and his coworkers published in Adv. Mater. ***35****, 2300681 (2023) and ACS Energ. Lett.* ***8****, 2423 (2023).*

TPS 25A Coherent X-ray Scattering
- GISAXS, SAXS, WAXS
- Materials Science, Batteries

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Revealing the Key Elements of Energy Storing

The charge/discharge mechanism in electrode material of batteries is discussed from the perspectives of X-ray absorption fine structure.

X-ray absorption fine structure (XAFS) spectroscopy is a powerful technique to investigate the physical and chemical characteristics of materials. XAFS can be divided into X-ray absorption near edge structure (XANES) and extended X-ray absorption fine structure (EXAFS). The spectral energy region nearing the absorption edge of an absorbing atom is referred to as XANES and the oscillation features which extend for 1,000 eV or more above the absorption edge, are referred to as EXAFS. The XANES spectrum exhibits high sensitivity to oxidation and geometry, while the EXAFS region depends on radial distribution of electron density surrounding the absorbing atom. This allows XAFS spectroscopy to be used as a quantitative method for determining bond length and coordination number. So far, this technique has been widely applied to several scientific research fields, including battery development and catalysis.

The development of battery technology has become increasingly innovative alongside the rapidly expanding market for rechargeable electric vehicles. Creating novel battery types or improving existing ones remain a challenge. For instance, batteries which work through redox reactions of the electrodes may be accompanied