Reciprocal Space mapping


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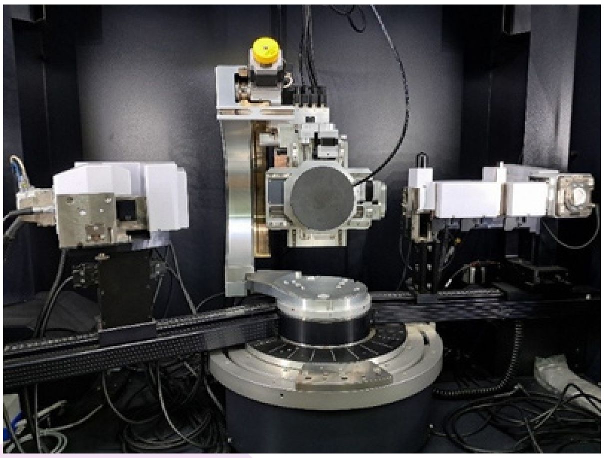
Overview: Reciprocal space mapping is an advanced X-ray diffraction technique essential for characterising epitaxial thin films. It provides insights on lattice spacing, orientation distribution, strain and defects.

This method is especially significant for epitaxial thin films of semiconductors, ferroelectrics and complex oxides where strain-induced effects on crystal structure and properties are widely studied. It enables precise determination of lattice strain and gives more details on the lattice relaxation, composition gradient and dislocation density in the epitaxially grown thin films.



The Technique: The X-ray diffractometer consist of a four circle (2θ, ω, ψ, φ) goniometer that enables precise orientation of the crystal and detector with Cu- Kα source of wavelength 0.154nm with a 4-bounce monochromator using parallel beam optics.

Unlike conventional θ–2θ scans that gives information only along the direction normal to surface of the film, reciprocal space map (RSM) provides a two-dimensional view of diffraction intensity in reciprocal space, offering detailed insights on in-plane and out-of-plane lattice parameters, strain, mosaicity, tilt, presence of multiple domains or defects etc.

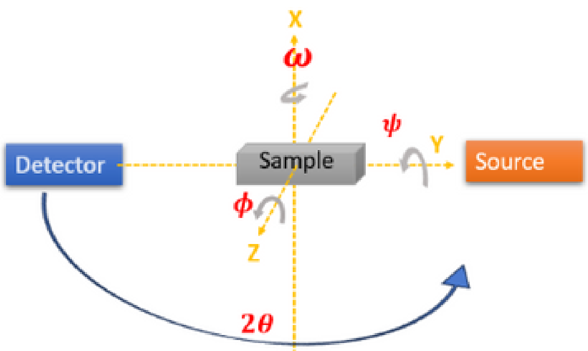


Figure 1. Schematic of four-circle goniometer

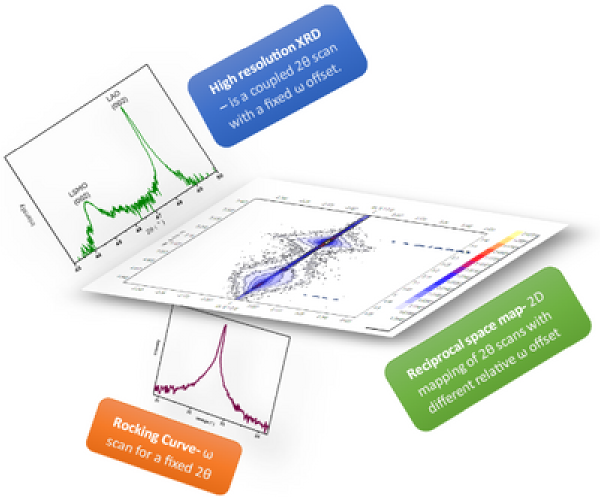


Figure 2. HR-XRD, Rocking curve and Reciprocal Space mapping

In epitaxial films, the arrangement of atoms is in a specific orientation with respect to the substrate, and there would be a substrate-induced strain present in the film. In a typical RSM measurement, the 0D or 1D detector scans both the ω (glancing angle) and 2θ (diffraction angle) axes in small steps around a reflection, allowing one to reconstruct the distribution of diffracted intensity in a plane of reciprocal space.

The scan can be symmetric or asymmetric depending on the alignment. The reconstruction consists of the Y axis corresponding to the out-of-plane (Q_z) component and the X axis to the in-plane (Q_x) component of the scattering vector.

RSM also enables the detection of tilt and mosaic spread present in the film. The elongation or split in certain directions can indicate the presence of tilted domains or dislocations in the film. Rocking curve measurement is performed by fixing the 2θ and sweeping tilting the sample to ± Δω.

The degree of peak broadening is related to mosaicity and defect densities. In high-quality single-domain films, the peaks are typically sharp and well-defined, whereas broad or streaked peaks suggest a less ordered structure. By fixing the 2θ and ω for a particular reflection, an Azimuth scan (φ) gives insights into the crystal symmetry relation of the film with respect to the substrate. Another important application of RSM is in determining lattice distortions and symmetry differences due to substrate-induced strain.

Especially in perovskite oxide films, structural distortions like octahedral tilting or ferroelectric displacements can lead to subtle splitting of reflections or asymmetry in reciprocal space.

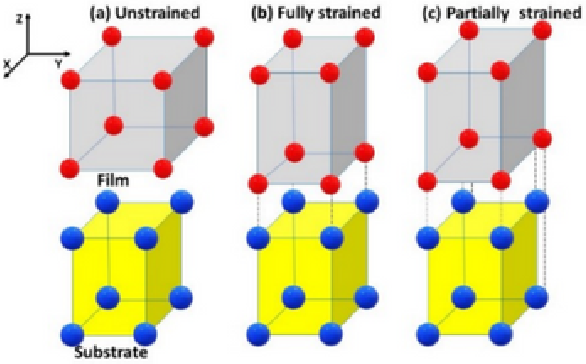


Figure 3. Schematic of (a) unconstrained, (b) fully constrained and (d)partially constrained film on the substrate

Also, in heterostructures and superlattices, where there is stacking of multiple layers of different materials stacked in a certain periodicity and different layer thicknesses, which offers varying lattice mismatch, multiple satellite peaks can appear in the RSM, from which layer thicknesses, periodicity, and interface quality can be inferred.

In conclusion, reciprocal space mapping is a substantial technique for characterizing epitaxial thin films where strain engineering of these films serves as an important strategy in tailoring the properties of materials such as ferroelectricity, ferromagnetism, bandgap in semiconductor industry, etc.

It provides a multidimensional perspective on the crystal structure of the film, enabling precise determination of strain, relaxation, crystallographic tilt, and microstructural defects.

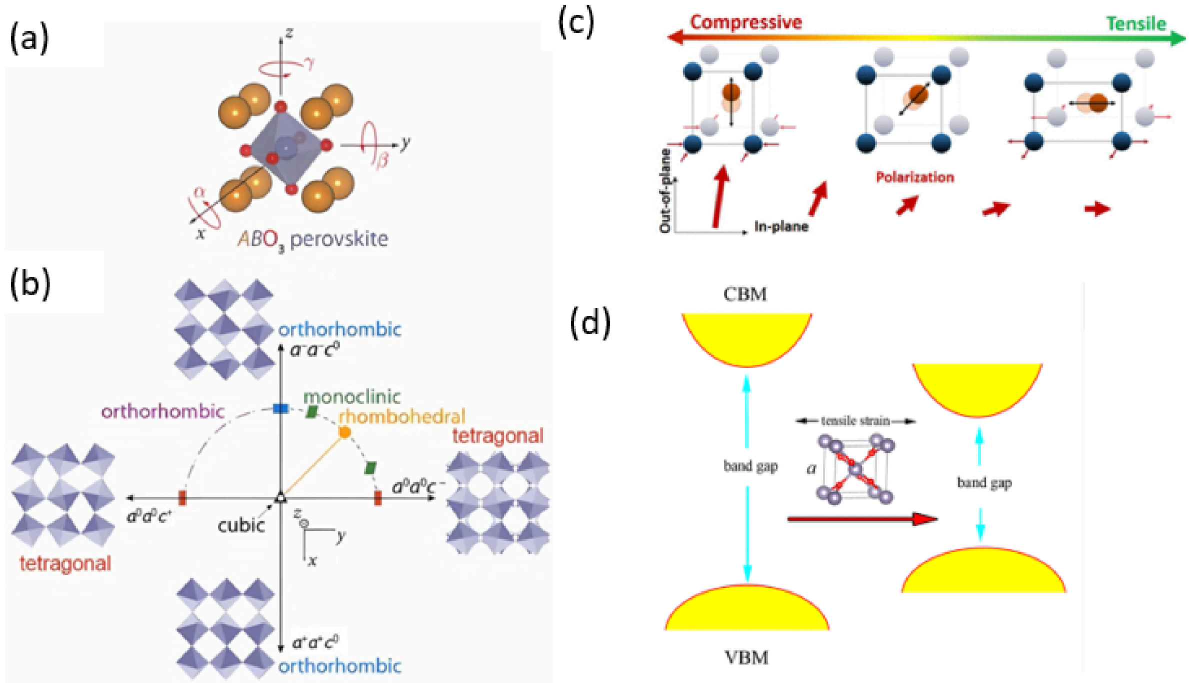


Figure 4. (a) and (b). ABO3 perovskite structure and distortion of BO6 Octahedra forming different crystal structures [1] (c) effect of strain on ferroelectric polarisation [2] (d) tuning of bandgap with strain [3]

It provides information about the subtle unit cell distortions, or strain-induced phase transitions in the epitaxially grown thin films which play a crucial role in understanding structure-dependent properties.

> *Reciprocal space mapping is an advanced X-ray diffraction technique essential for characterising epitaxial thin films. It provides insights on lattice spacing, orientation distribution, strain and defects.*

References
[1] Dhole, S., Chen, A., Nie, W., Park, B., & Jia, Q. (2022). Strain Engineering: A Pathway for Tunable Functionalities of Perovskite Metal Oxide Films. Nanomaterials, 12(5), 835.
[2] Schneider, T., Cardoletti, J., Komissinskiy, P., & Alff, L. (2023). Impact of strain engineering on antiferroelectricity in NaNbO3 thin films. ACS omega, 8(26), 23587-23595.
[3] Zhou, W., Liu, Y., Yang, Y., & Wu, P. (2014). Band gap engineering of SnO2 by epitaxial strain: experimental and theoretical investigations. The Journal of Physical Chemistry C, 118(12), 6448-6453.

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