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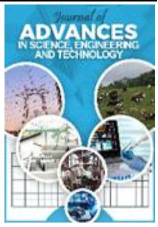

Advances in Beamline in situ Spin-Coating for Perovskite Thin Films and Beyond

Elly Shatsala*

Department of Physics, School of Natural Sciences, Masinde Muliro University of Science and Technology, Kakamega, Kenya.

ABSTRACT

This research letter presents a beamline-integrated in situ spin coater that enables real-time observation of thin-film formation and crystallization during spin coating and annealing. The system integrates temperature-stable heating, low-vibration motors, and a helium-purged inert chamber with synchronized anti-solvent injection and detector acquisition at millisecond resolution. Controlled through a LabView interface, synchronized triggers coordinate spin speed, anti-solvent delivery, and data collection within the beamline environment. Applied to Cs0.1FA0.9PbI3 films passivated with PEACl, time-resolved grazing-incidence X-ray scattering (GIWAXS) reveals transient Pb-Cl-I intermediates that vanish as 3D perovskite phases form. The evolution of quasi-2D domains, lattice contraction, and crystallite orientation directly correlates with nucleation and passivation dynamics. This approach provides a general framework for linking processing conditions to structural evolution in perovskite and other functional thin films.

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1. Introduction

Synchrotrons are circular accelerators that propel electrons using sequences of magnets until they reach relativistic speeds, producing radiation millions of times brighter than conventional light sources (Willmott, 2019). This intense, tunable X-ray light enables structural and chemical analysis at atomic and molecular scales (Godwin, 1969; Fan & Zhao, 2018). Modern synchrotron facilities have become indispensable for in situ and operando studies of material processes, enabling high-resolution mapping of crystallization, phase transitions, and strain evolution (Liu et al, 2021).

In situ spin-coating techniques at synchrotron beamlines allow direct tracking of thin-film formation in real time. Early ex situ studies have evolved into advanced glovebox-integrated systems equipped with motorized spin coaters, transparent X-ray windows, and synchronized triggers coordinating spin speed, anti-solvent injection, and detector acquisition with millisecond precision (Manley et al., 2017; Steele et al., 2023). This capability is especially critical for complex systems such as metal- halide perovskites, where solvent coordination, nucleation, and intermediate phases evolve rapidly (Yang et al., 2024).

Alternative thin-film deposition methods (Wafer World, 2025), such as Pulsed Laser Deposition (PLD) (Fujioka, 2015; Krebs et al., 2003), Chemical Vapor Deposition (CVD) (Carlsson & Martin, 2010; Zhang et al., 2013), Physical Vapor Deposition (PVD) (Mattox, 2010), and Atomic Layer Deposition (ALD) (Johnson, Hultqvist, & Bent, 2014) can, in principle, be integrated into synchrotron beamlines for in situ studies. Yet, each presents intrinsic constraints: PLD often produces non-uniform films with particulate contamination (Fujioka, 2015; Krebs et al., 2003); CVD requires hazardous precursors and high substrate temperatures (Carlsson, & Martin, 2010; Zhang et al., 2013.); PVD is limited by its line-of-sight geometry and slow growth rates (Mattox, 2010); and ALD, though exceptionally precise, remains inherently slow and restricted to small substrates (Johnson, Hultqvist, & Bent, 2014). These limitations highlight the need for a more versatile and accessible platform for in situ and operando X-ray measurements. Spin-coating provides a simple, rapid, and scalable alternative that can be readily adapted for beamline integration, enabling direct observation of crystallization, phase evolution, and interfacial processes under realistic processing environments.

Despite recent progress, reproducibility, beam-induced effects, and large data handling remain challenges. Emerging operando platforms integrate GIWAXS with optical or electrical probes, and machine-learning-driven control schemes are being developed for

* Corresponding author. e-mail: millershatsala@gmail.com

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automated, reproducible deposition. Once driven primarily by perovskite research, in situ spin-coating is now extending to polymeric, colloidal, and oxide thin films, establishing it as a broadly applicable technique for studying real-time structure formation (Bauer et al., 2023; Toolan & Howse, 2013).

2. Results Highlight and Discussion

2.1. Beamline-integrated design and synchronization

The developed in situ spin coater at micro-diffraction beamline 12.3.2 at Advanced Light Source incorporates temperature-controlled heating (up to 180 °C), low-vibration direct-drive motors, and a helium-purged chamber with adjustable gas flow and humidity control. The compact geometry allows direct mounting on diffractometer stages without beam misalignment. Thin Kapton windows (7–25 µm) ensure minimal parasitic scattering, while piezo-driven anti-solvent injection is synchronized with detector readout via transistor – transistor logic (TTL) triggers using an Arduino.

Multimodal data acquisition is enabled through high-speed optical imaging and optional electrical contacts for operando testing. The schematic and mounted spin-coater are shown in Fig.1 This configuration achieves sub-millisecond synchronization, ensuring precise temporal correlation between deposition events and GIWAXS signal evolution. As a result, the coater captures both rapid nucleation processes and slower annealing dynamics with exceptional fidelity.

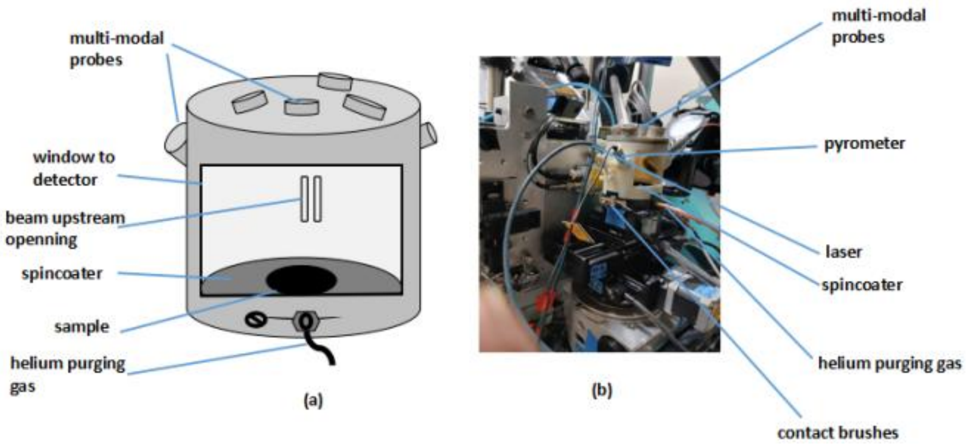

Fig. 1: (a) Schematic of the *in-situ* spin-coater setup. (b) Spin-coater mounted at BL 12.3.2

2.2. Real-time crystallization dynamics in PEACl-passivated $Cs_{0.1}FA_{0.9}PbI_3$

Time-resolved GIWAXS measurements reveal transient diffraction peaks at distinct *q*-values during early annealing, which vanish as 3D perovskite reflections grow (Fig. 2 (a)). These features correspond to Pb-Cl-I intermediate complexes or chloride-rich low-dimensional phases that mediate nucleation before converting to the α-phase perovskite. These findings are similar to other reports elsewhere (Steele et al., 2023; Kodalle et al., 2024; Maqsood et al., 2021). Integrated intensity plots shown in Fig. 2 (b) confirm a direct correlation between the decay of intermediates and the rise of the α-phase signal (Yadavalli et al., 2020).

A gradual shift of Bragg peaks to higher *q*-values ("blue-shift") indicates lattice contraction, likely due to solvent evaporation, Cl^- release, or partial incorporation of smaller cations (Reddy et al., 2011). Fig. 2(c) sediment the Time-resolved process in which early stages of spin coating do not produce significant peaks, but there is a mild brightening due to the solvate. A delta phase peak appears after the anti-solvent drop, which transitions to alpha on the onset of annealing.

As annealing proceeds, diffuse diffraction rings transform into sharper arcs (also seen in time-resolved diffraction panel in 2 (c)), indicating improved crystallite orientation. The surfactant action of PEA^+ and Cl-mediated kinetics fosters anisotropic growth, forming large, well-aligned grains (Alasiri et al., 2024). The primary perovskite reflections sharpen and intensify, consistent with grain coarsening and enhanced coherence length (Cao et al., 2024).

A decrease in d-spacing seen in Fig. 2 (d) is generally considered good in layered (quasi-2D) perovskites as it often leads to improved charge transport and enhanced structural stability, which are critical for high-performance devices like solar cells and X-ray detectors (Jung et al., 2018).

Collectively, these results demonstrate that PEACl-assisted crystallization promotes structural order and orientation favorable for charge transport and device stability.

3. Conclusion

The developed *in situ* spin-coating system enables synchronized, high-speed GIWAXS measurements that uncover dynamic intermediates and orientation transitions during perovskite film formation. In $Cs_{0.1}FA_{0.9}PbI_3$:PEACl, the detection of transient Pb-Cl-I phases, and lattice contraction reveals a clear mechanistic link between Cl-mediated nucleation and improved crystal orientation. Beyond perovskites, the method establishes a platform for understanding structure-processing relationships across diverse functional thin films.

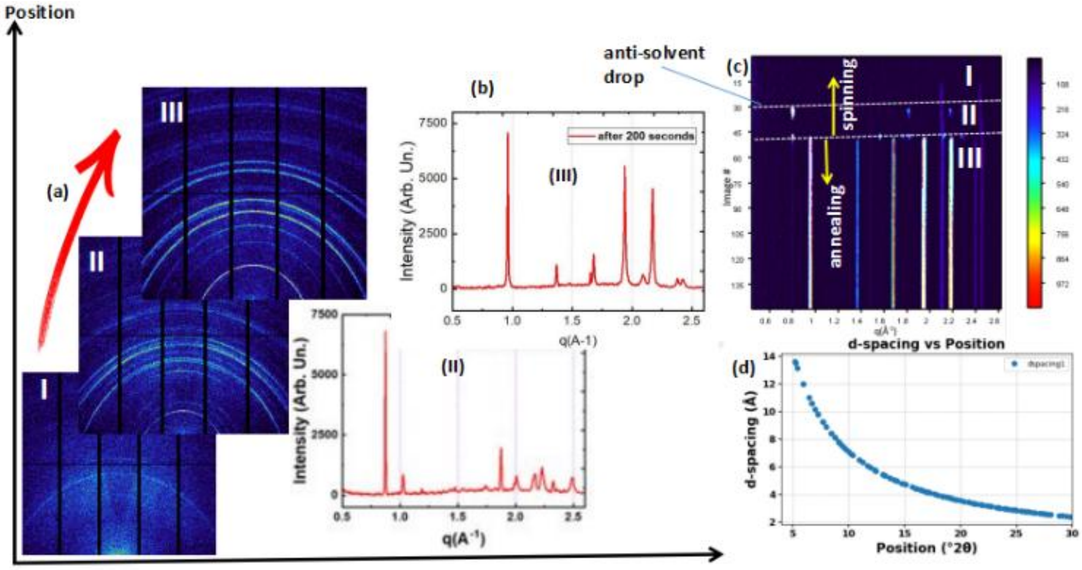

Fig. 2: Time-resolved structural evolution during in situ spin-coating. (a) diffuse diffraction GIWAXS rings (b) 1D *q*-integrated traces (*I(q)*) stacked over time showing transient intermediate and low-*q* peaks; (c) integrated intensity versus image number for key reflections - intermediate, α-perovskite, and PbI_2 (if present)- on a common time axis; (c) evolution of peak positions ($2\theta^0$) converted to *d*-spacing, revealing absolute lattice changes (Å)

4. Impact and Outlook

Beamline-integrated *in situ* spin-coating represents a key step toward reproducible, multimodal, and automated thin-film characterization. With advances in data analytics, machine-learning-driven deposition control, and scalable *operando* architectures, these tools will accelerate materials discovery by uniting synthesis, structure, and function in real time

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