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ESRF

Experiment Report Form

The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.

Once completed, the report should be submitted electronically to the User Office via the User Portal:

https://wwws.esrf.fr/misapps/SMISWebClient/protected/welcome.do

Reports supporting requests for additional beam time

Reports can be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

Reports on experiments relating to long term projects

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

Published papers

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

ESRF	**Experiment title:** Real-time X-ray diffraction of the crystallization of mixed (FAPbI3)1-x (MAPbBr3)x (x=0-1) perovskites	**Experiment number**: **SC-4668**
Beamline: ID03	**Date of experiment**: from: 21 June 2017 to: 27 June 2017	**Date of report**: 03. March 2018
Shifts: 15	**Local contact(s)**: Maciej Jankowski	*Received at ESRF:*

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Report:

Compositional engineering of organic-inorganic lead halide perovskites is one of the key methods to achieve high solar cell efficiency. As stated in the proposal, we have measured the formation of crystalline perovskite thin films during the formation of mixed $(FAPbI_3)_{1-x}$ $(MAPbBr_3)_x$ (x=0-1) (FA= $(HN=CHNH_3^+)$, $(MA=(CH_3NH_3^+))$ perovskites from a precursor solution.

The following X-ray scattering experiments were done at beamline ID03 of the ESRF with a photon energy of 24 keV under ambient conditions. The beam size was 50 µm in vertical direction and 100 µm in horizontal direction. GIXD data was measured under an angle of incidence of 0.1° with a Maxipix area detector. GIWAXS data was measured with a PILATUS 300k area detector under the same angle of incidence.

Since the analysis of the data is still in progress, we present only some important findings in this report. As an example in Fig.1 we show postgrowth GIXD data of mixed perovskite films dependent on mixing ratio. All films are crystalline with reflections matching a single perovskite structure. Depending on mixing ratio we find a slight shift of the lattice constant of the cubic perovskite structure. The shift of the lattice constant is induced by a larger molecular cation, which expands the lattice.

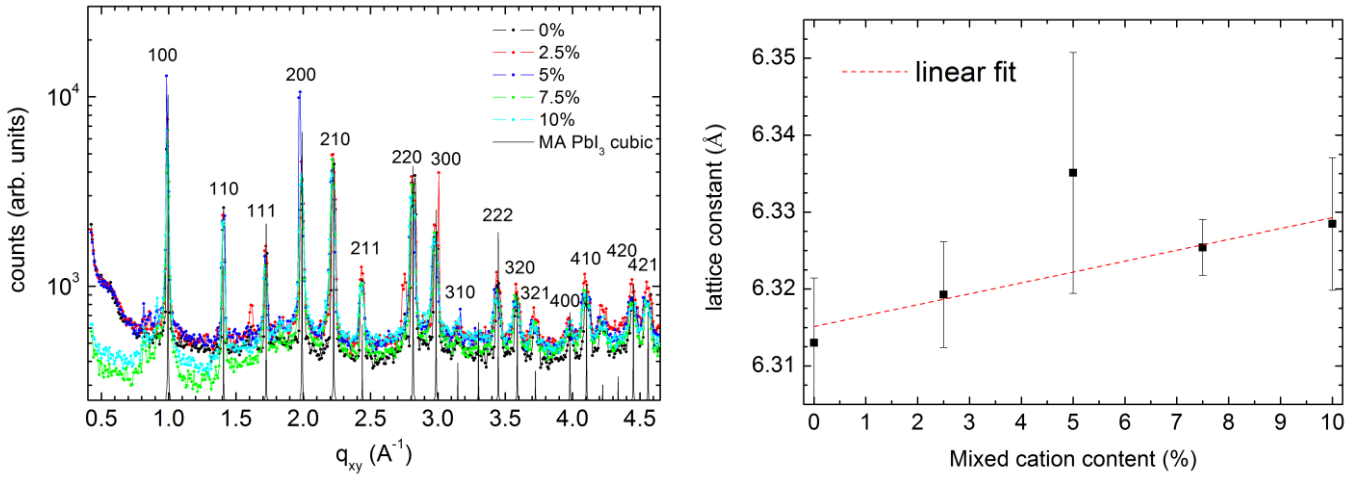

Fig. 1: Left: GIXD data of mixed organic cation perovskites depnedent on mixing ratio. Right: Linear shift of the cubic lattice constant of the perovskite thin film dependent on mixing ratio.

Fig. 2 shows shows GIWAXS data from two films with different molecular mixing ratios. Both films show cubic perovskite crystal structure. For low mixing ratio (Fig. 2a) the diffraction rings exhibit a pronounced substructure (small dots distributed over the ring) resulting from large crystalline domains. This is a hint that the domain size distribution is very broad. For high mixing ratio (Fig. 2b) we find nearly homogenous rings without the substructure hinting to a situation where we have smaller domains, but with a more homogenous domain size distribution.

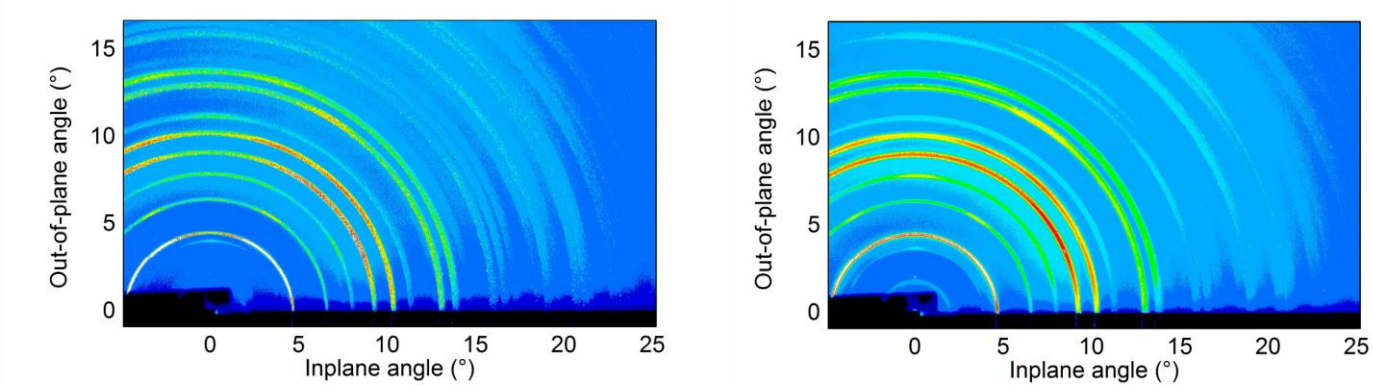

Fig 2: Comparison of GIWAXS data from two films with different mixing ratios (left: low mixing ratio, right: high mixing ratio) of molecular cations. Each image was assembled from 6 images measured with a PILATUS 300k detector.

As stated in the proposal, we were able to measure the crystal formation dynamics for several material compositions. The crystal formation dynamics were measured in real-time during the drying process by grazing incidence X-ray diffraction (GIWAXS) to determine the crystal quality, in terms of peak width and orientation distribution. The analysis of these data is still ongoing. With these *in situ* real-time measurements, we expect to obtain a detailed understanding of the structural aspects of perovskite crystal formation from solution.

We wish to acknowledge the excellent collaboration with the beamline staff, which made this challenging experiment a success.