EUROPEAN SYNCHROTRON RADIATION FACILITY

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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:** In situ analysis of the crystallization process of CH3NH3PbX3 (X= Cl, Br, I) perovskite by real-time X-ray diffraction.	**Experiment number**: **SC-4438**
Beamline: ID10	**Date of experiment**: from: 31 Aug 2016 to: 05 Sep 2016	**Date of report**: 02. Jan 2017
Shifts: 15	**Local contact(s)**: Andrei Chumakov, Federico Zontone	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

As stated in the proposal, we have measured the formation of crystalline perovskite thin films during the conversion of a precursor material after drop-casting CH_3NH_3X in 2-propanol. The crystal quality and grain orientation were measured in real-time during the drying process by grazing incidence diffraction GIXD to determine the crystal quality, in terms of peak width and orientation distribution.

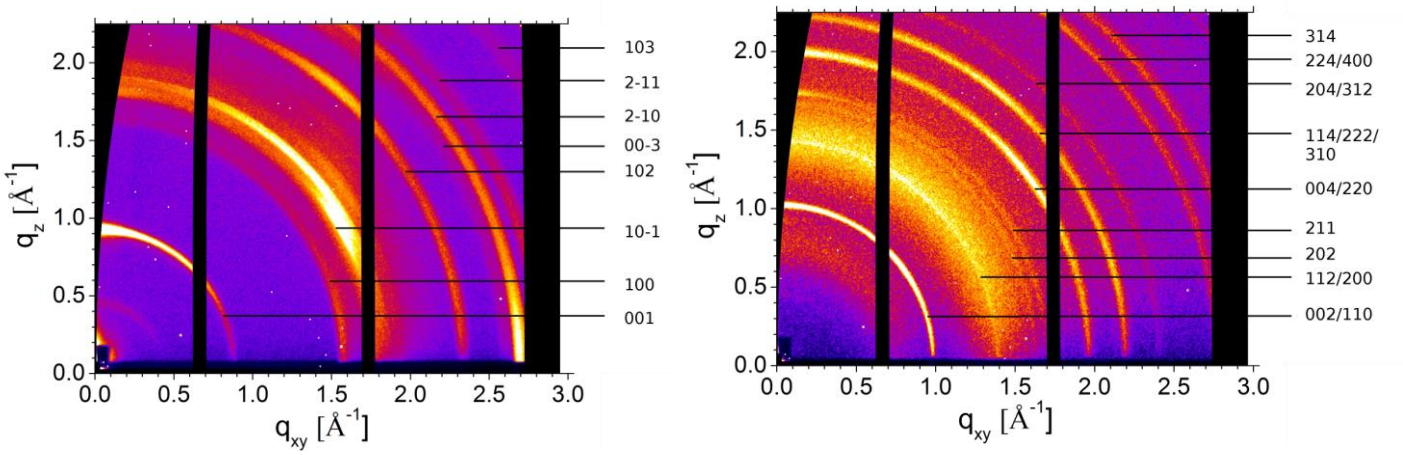

Fig. 1: Reciprocal space maps of a precursor material (left) and a perovskite (right) thin film after ~15 min of the dropcasting of CH3NH3X in 2-propanol.

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 two reciprocal space maps of the precursor material and the resulting perovskite thin film. The data is of high quality and all reflections can be indexed according to the known perovskite crystal structure.

As a first step we analysed the intensity distribution of the Bragg reflections versus time through the complete time series. In Fig. 2 the evolution of perovskite and precursor Bragg reflection intensity is shown for one sample. It is clearly visible that the perovskite Bragg intensity is strongly increasing and the precursor Bragg intensity is increasing on a time scale of minutes. These reaction dynamics are different depending on precursor material and drop casted solution.

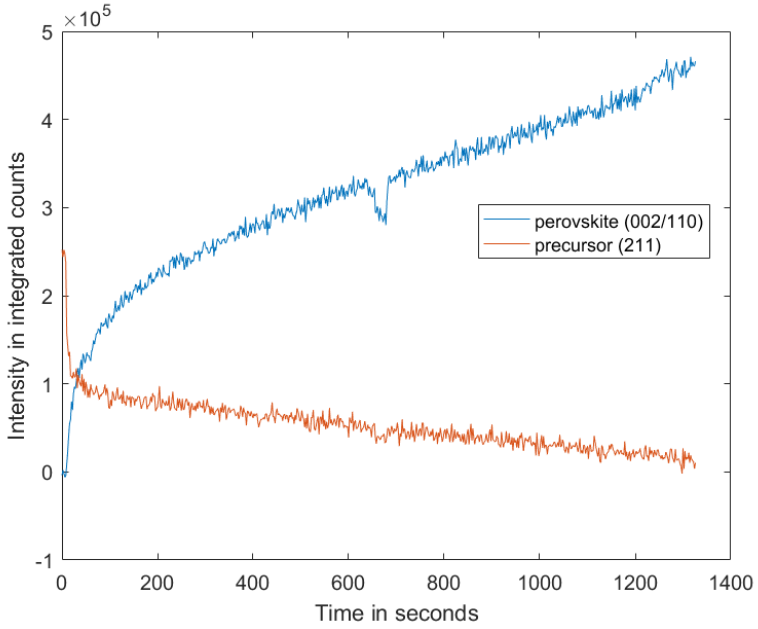

Fig 2: Real-time intensity evolution of the Bragg reflections belonging to the precursor material (orange) and the perovskite (blue) thin film.

As stated in the proposal, we were able to measure the crystal formation dynamics for several material compositions. With these *in situ* real-time measurements, we expect to obtain a detailed understanding of the structural aspects of perovskite crystal formation from solution.

These measurements will be compared with the performance of the devices based on the films deposited under similar conditions. Finally, we should be able to tailor the properties of the perovskite material in a way to fabrication high-efficiency solar cells.

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