EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

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

Deadlines for submission of Experimental Reports

Experimental reports must be submitted within the period of 3 months after the end of the experiment.

Experiment Report supporting a new proposal ("relevant report")

If you are submitting a proposal for a new project, or to continue a project for which you have previously been allocated beam time, you must submit a report on each of your previous measurement(s):

- even on those carried out close to the proposal submission deadline (it can be a *"preliminary report")*,
- even for experiments whose scientific area is different form the scientific area of the new proposal,
- carried out on CRG beamlines.

You must then register the report(s) as "relevant report(s)" in the new application form for beam time.

Deadlines for submitting a report supporting a new proposal

- 1st March Proposal Round - **5th March**
- 10th September Proposal Round - **13th September**

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.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report in English.
- include the experiment number 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:** Light-induced halide ion segregation in hybrid perovskites	**Experiment number**: SC-5363
Beamline: ID10	**Date of experiment**: from: 10.11.22 to: 14.11.22	**Date of report**: 18.11.22
Shifts: 12	**Local contact(s)**: Federico Zontone	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

We investigated the changes in thin films of $(CH_3NH_3)PbBr_{1.8}I_{1.2}$ induced by light in real time. We used our own custom-made cubic sample cell with kapton windows for X-rays and a powerful LED (λ=450 nm) from ThorLabs. The perovskite sample was constantly flushed with dry nitrogen to improve the sample stability. To make sure that the structure of the film is not affected by X-rays, we used attenuators, which allowed us to take a series of 900 X-ray diffraction patterns over 1.5 hours without any visible changes in the measured diffraction pattern.

We utilized an X-ray beam (E=9.94 keV) focused to approximately 2x2 μm^2 and studied the speckle pattern formed around the 001 reflection in Bragg geometry. The speckles arize from segregation of halogene ions caused by light. During the long ilumination period (90 minutes) we observed splitting of the initial 001 diffraction peak into 3 weaker peaks, corresponding to the mixed $(CH_3NH_3)PbBr_{1.8}I_{1.2}$ structure and domains with higher concentration of Br^- ions, $(CH_3NH_3)PbBr_{1.8+X}I_{1.2-X}$, as well as domains with higher concentration of I^- ions, $(CH_3NH_3)PbBr_{1.8-Y}I_{1.2+Y}$ (Fig. 1). Careful analysis of the peak positions and the widths will allo us to estimate the ratio of halide ions in all type of domains, as well as estimate the size of these domains. This analysis is presently ongoing.

In addition to studing the formation of domains (dynamics), we are using the X-ray photon correlation techniques (XPCS) to study the movement of domains (kinetics). For this, we will correlate the speckle patterns around the 001 diffraction peak at different times, and from the decay of the correlation function

$$C(t_1, t_2) = \frac{\langle I(t_1) I(t_2) \rangle_q}{\langle I(t_1) \rangle_q \langle I(t_2) \rangle_q}$$

we will extract the characteristic times of domain movement. Unfortunately, this type of measurement is very sensitive to the stability of the incident X-ray beam, which was not perfect during our measurements (Fig. 2). Since the experiment was conceptually successful, we are presently in the process of checking how much information on short timescales can be extracted from the data after careful normalization, but it might turn out that the instabilities cannot be normalized out, and we might need to repeat our XPCS measurements in the future with a more stable beamline configuration.

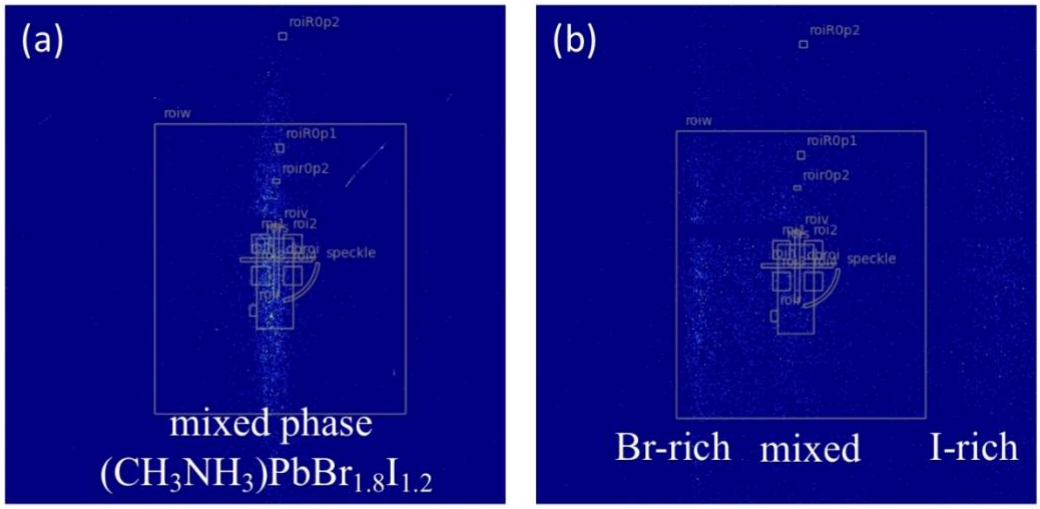

Figure 1. (a) Diffraction pattern from the mixed $(CH_3NH_3)PbBr_{1.8}I_{1.2}$ perovskite before the illumination with LED. Signle diffraction peak is visible. (b) Diffraction pattern from perovskite film with the light induced domain segregation after illumination with LED for 90 minutes. Two new peaks corresponding to Br-rich and I-rich perovskite phases has appeared.

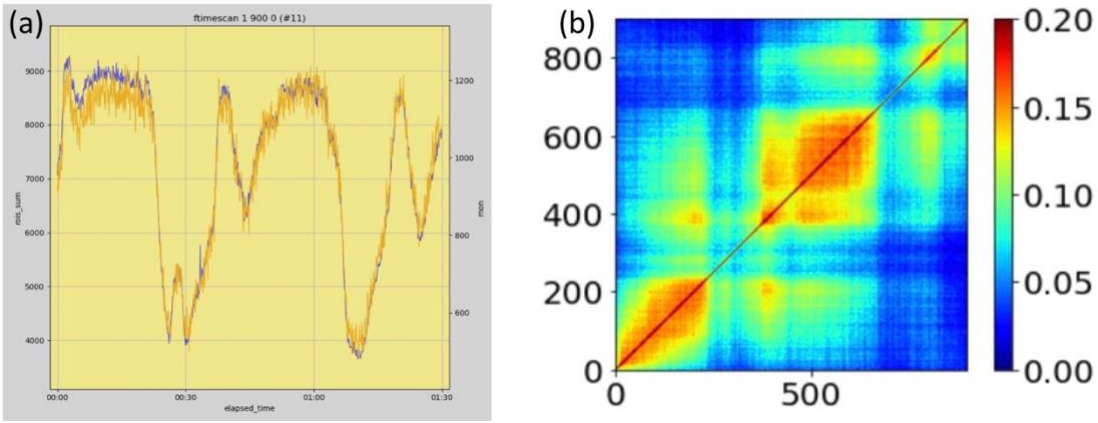

Figure 2. (a) Blue line – an average intensity of the diffraction pattern during the 90-min scan. Orange line – intensity of the incident X-ray beam. (b) corresponding two-time correlation function $C(t_1, t_2)$ showing strong artefacts corresponding to the drops of the intensity of the incident X-ray beam.