EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

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:** Spatially-resolved phase distribution in solvent-deposited perovskite photovoltaic films	**Experiment number**: HC-2204
Beamline:	**Date of experiment**: from: 08 November 2015 to: 13 November 2015	**Date of report**: 10 May 2016
Shifts: 12	**Local contact(s)**: Thomas Dane	*Received at ESRF:*

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Report:

Scanning nanofocus X-ray diffraction (nXRD) was used for the first time to investigate the structure of $CH_3NH_3PbI_3$ (MAPI) perovskite films for thin film photovoltaic devices. A monochromatic beam of λ = 0.8349 Å was focused to a spot size of ~200 × 200 nm^2, across which the MAPI films on silicon substrates were scanned in transmission geometry. The results could be compared directly with scanning electron microscopy (SEM) and grazing incidence x-ray diffraction (GIXRD) results, showing clear benefits for nXRD:

- nXRD probes the full depth of the film, unlike SEM, enabling us to identify grains that overlap at different depths (fig. 1).
- By structurally characterising individual perovskite grains, the distribution of grain dimensions is directly mapped, providing clear benefits over peak width analysis. Samples at different points in an anneal sequence show clear differences which cannot be detected in GIXRD.

The results have been submitted as a paper to Advanced Functional Materials with the following abstract:

Scanning nanofocus X-ray diffraction (nXRD) performed at a synchrotron is used for the first time to simultaneously probe the morphology and the structural properties of spin coated $CH_3NH_3PbI_3$ (MAPI) perovskite films for photovoltaic devices. MAPI films are spin coated on a Si/SiO_2/PEDOT:PSS substrate held at different temperature during the deposition to tune the perovskite film coverage and then investigated by nXRD, scanning electron microscopy (SEM), and grazing incidence wide angle X-ray scattering (GI-WAXS). The advantages of

nXRD over SEM and GI-WAXS are discussed. A method to visualize, selectively isolate, and structurally characterize single perovskite grains buried within a complex, polycrystalline film with a spatial resolution of 200 nm is developed. The results of nXRD measurements are correlated with solar cell device measurements, and it is shown that spin casting the perovskite precursor solution at elevated temperature leads to improved surface coverage and enhanced solar cell performance.

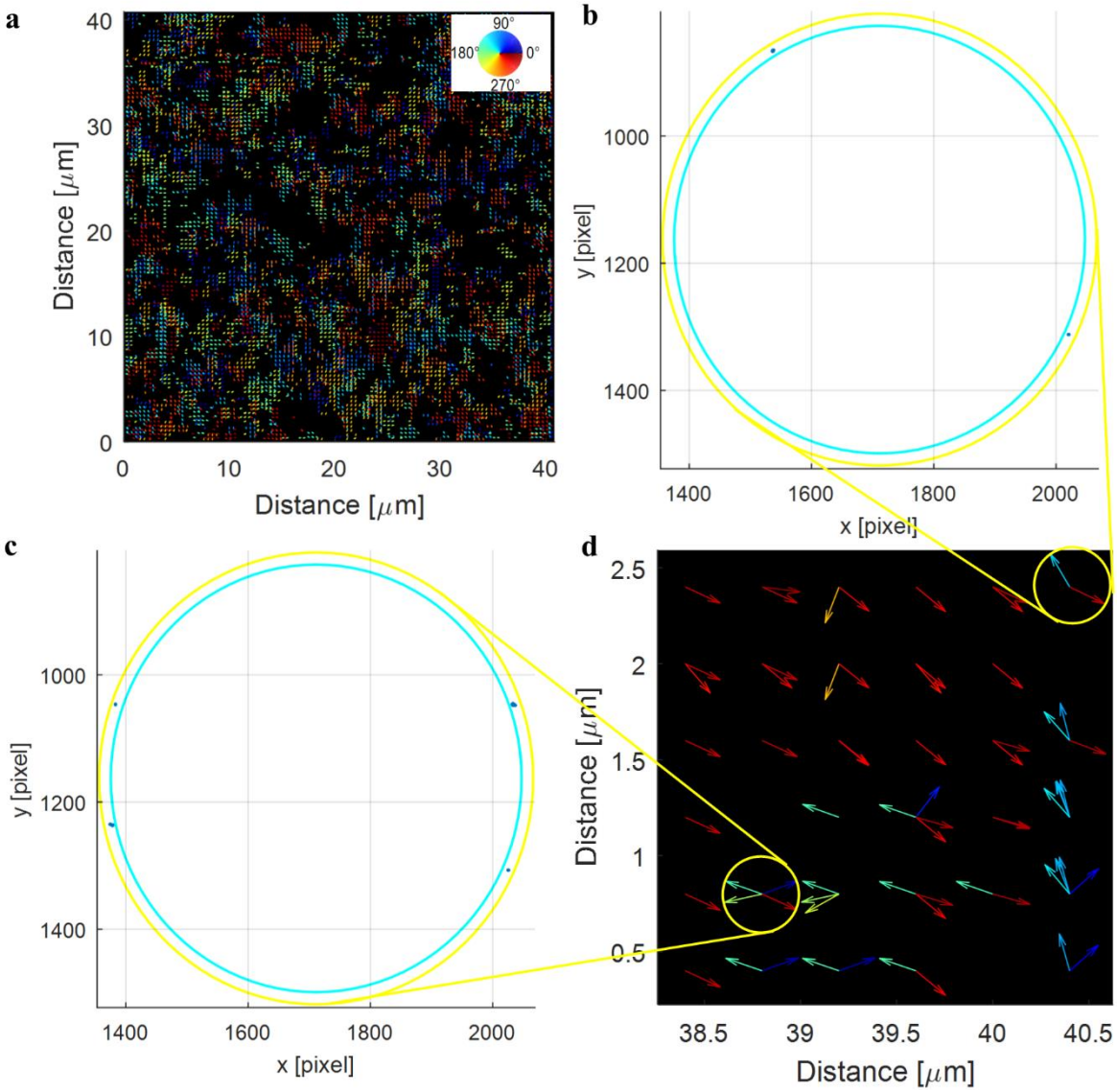

Figure 1. A representation of the grain orientation from nXRD in terms of the angular coordinate of peaks extracted from diffraction spots (a). Examples of sparse patterns extracted from diffraction peaks for two (b) and four (c) overlapping grains from region (d), which represents a zoom of the bottom right hand corner of image (a).