

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://www.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.



| | | |
|---|--|--------------------------------------|
| | Experiment title: High pressure structural studies of $\text{CH}_3\text{NH}_3\text{PbX}_3$ X=(I, Br, Cl) photovoltaic perovskites | Experiment number: |
| Beamline: Id27 | Date of experiment: from:15.09.2016 to:17.09.2016 | Date of report: 15.03.2017 |
| Shifts: 6 | Local contact(s): Volodymyr Svitlyk | <i>Received at ESRF:</i> |
| Names and affiliations of applicants (* indicates experimentalists): Balint Nafradi* All Arakcheva* Gaetan Giriat* EPFL/LPMC | | |

Report:

The high-pressure (HP) single-crystal XRD experiments for MAPbX_3 with X = I and Cl have been performed with Ar and Ne pressure transmitting mediums (PTM) up to 20 GPa.

The most successful XRD experimental data have been obtained for MAPbI_3 in both Ne and Ar. Structural studies based on these data allow finding of new $\text{Ne}_x\text{-MAPbI}_3$ and $\text{Ar}_x\text{-MAPbI}_3$ compounds with pressure dependent compositions. The corresponding article is under preparation for mature Materials.

Details of a structure of the MAPbI_3 HP transformation in Ne and AR pressure transmitting medium.

Comparison of the pressure-induced changes to the composition, relative unit cell volume and density of the MAPbI₃ crystal exposed to high pressure in two PTMs, Ne and Ar

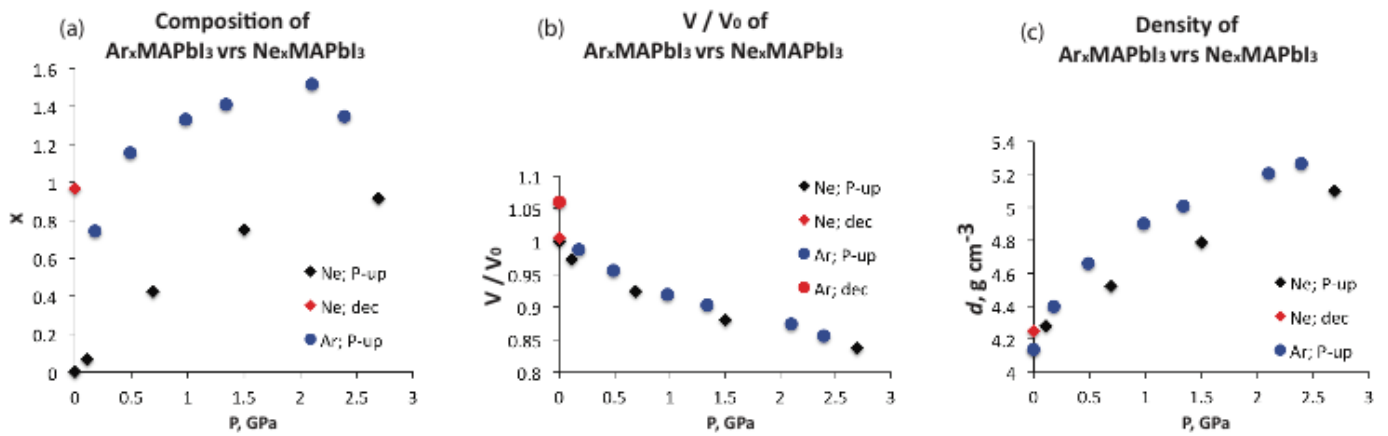


Fig. 1 Pressure-induced changes to: (a) the crystal composition, (b) the relative unit cell volume normalized to a single formula, and (c) the density of the MAPbI₃ crystal for two PTMs Ne and Ar with the same pressure range.

Influence of crystal quality on the HP-transformation of MAPbI₃

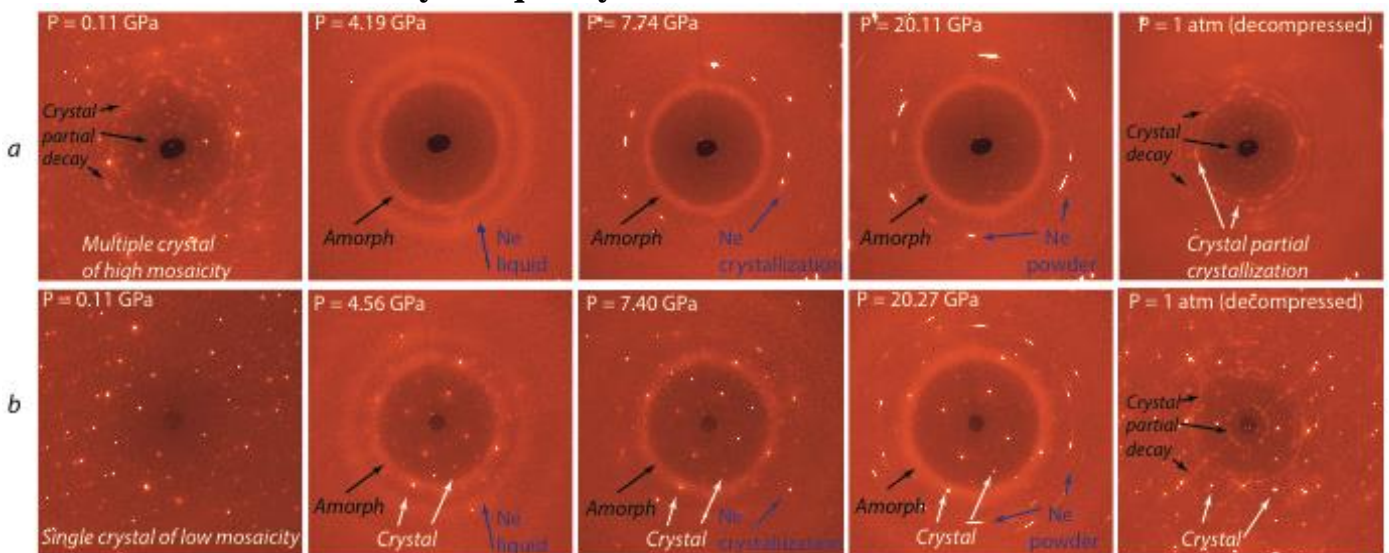


Fig. 2 The high-pressure transformation of the MAPbI₃ crystal of different quality with Ne-PTM. The single crystal diffraction patterns are shown for different pressure (P). (a) A multiple crystal of high mosaicity and traces of its decay. The crystal is completely amorphous at about 4 GPa and show only a partial recrystallization after decompression. (b) Crystal of low mosaicity and without any traces of decay. A part of the crystal keeps its crystalline state up to about 20 GPa and completely recovers its crystalline state after decompression. Two light circles observed at P=4.19GPa appear from two amorphous fractions: the outer one is due to Ne-liquid: the inner one is to the MAPbI₃ amorphous fraction. At P>7.4GPa, only the amorphous circle is observed since Ne becomes crystalline. The pressure-induced increasing of its intensity indicates the increase of the amorphous fraction volume.