EUROPEAN SYNCHROTRON RADIATION FACILITY

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Experiment Report Form

	Experiment title: Real-time imaging of phase transitions in CsPbBr3 perovskite nanowires using temperature-dependent Full Field Diffraction X-ray Microscopy	**Experiment number**: MA-4714
Beamline: ID01	**Date of experiment**: from: 03 Mar 2021 to: 07 Mar 2021	**Date of report**: 22 Oct 2021
Shifts: 12	**Local contact(s)**: Edoardo Zatterin / Ewen Bellec	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

Due to the COVID-19 pandemic, this experiment was carried out fully remote, which imposed some limitations. We used most the of night shifts for searching, which was necessary in the beginning, but not so much in the last days of beamtime. The time management could have been much better if we had users on-site, but the succees of the experiment was only achieved thanks to the high effort employed by beamline staff.

In this experiment, we used temperature-dependent Full-Field Diffraction X-ray Microscopy (FFDXM) to probe the evolution of nanodomains in a single $CsPbBr_3$ nanoplatelet. The sample used here consist on $CsPbBr_3$ nanostructures directly grown on SiO_2 glass substrate. We mounted it inside the furnace provided by the ID01 beamline in transmission geometry.

Initially, we positioned the MAXIPIX detector at the theoretical orthorhombic 004 Bragg reflection and scanned the X-ray beam with the sample, for several incident angles theta, until find a well aligned structure. It was relatively simple to cover a large area of the sample, since we were using a 70 x 70 μm^2 beam. After aligning the nanoplatelet, the MAXIPIX detector was moved out of the way, and the compound refractive lenses (CRLs) were positioned so we could project a real space image of the sample on the Andor camera, inside the vacuum pipe and 6.5 m away from the sample.

The projected image shows all domains aligned in Bragg condition and by scanning the incident theta angle, *i.e.* a rocking curve, we were able to visualize tilted domains. One fixed incident angle was selected and the temperature was slowly increased, so we could track the domains evolution with high time resolution (6 s, limited by the detector readout time). We had a surprisingly strong signal at the Andor detector, and after discussing with bamline staff, we believe that the time resolution could have been even better with an improved readout system for the detector.

Smooth domains rearrangement could be noted as the temperature was increased, until a dramatic change near 75 °C, close to the theoretical orthorhombic to tetragonal $CsPbBr_3$ crystal phase transition. We continued to increase the temperature, and a second sudden change was seen near 83 °C. Such result was unexpected, and can help us better understand metal halide perovskite phase transition properties. Both events are depicted in Figure 1.

Later, the temperature was decreased until room temperature, but only gradual changes could be seen during the process. In the last step of the experiment, we cycled the temperature between 20 °C and 80 °C multiple times, to check for domain configuration reproducibility. Rocking curves were performed near critical points along the whole experiment.

We are now finishing to write a manuscript with the results, which will be submitted for publication very soon. We have also submitted a new proposal where we want to read the data with the Andor camera in a smarter way, improving a lot the time resolution achieved here.

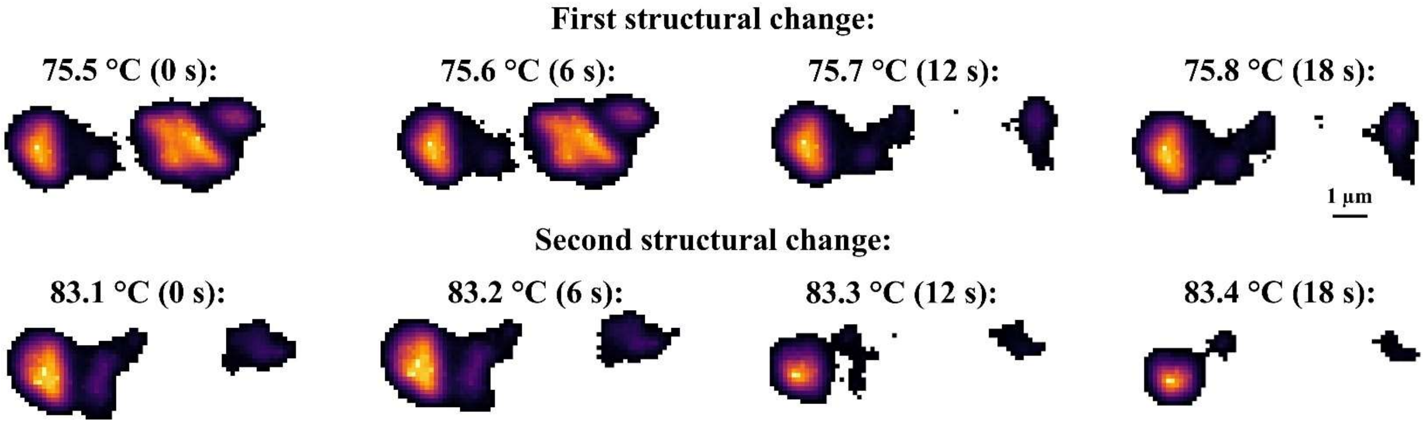

Fig. 1: Two separate sudden changes in the nanoplatelet domain configuration seen during sample heating. Top panels show the structural change near 75 °C, while bottom panels depict the change close to 83 °C.