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:** **Structural study of the Yttrium-Doped Barium Zirconate thin films interface upon insertion of protons.**	**Experiment number**: **MA-3069**
Beamline: **ID03**	**Date of experiment**: from:29 Nov 2016 to: 05 Nov 2016	**Date of report**: **13 Sept 2017**
Shifts: 18	**Local contact(s)**: Raja Znaiguia	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

Aim of the experiment was to determine if the misfit (MS) dislocation network present at the interface between a thin film of BaZr0.8Y0.2O3-x (BZY) grown onto the (110) surface of NdGaO3 (NGO) substrate was sensitive to proton loading.

This substrate is normally chosen for the growth of perovskites because of its stability. The (110) face is the one showing the standard cubic perovskite structure with a lattice constant of 3.91Å.

Four samples have been analised during the experiment differing mainly in thickness of the BZY film in the range from 6 to 100 nm. For the experiment we used the flow reactor in order to control the gas environment of the samples from humid atmosphere (Ar saturated with water at 1 bar) to O2 treatment (300 mbar) and CO (at 200 mbar). In the first sample Gas exposure was carried out at 450°, 480° and 500°C while on the other samples the exposure was carried out only at 480°C.

In figure 1 we show a typical intensity 2D map from such systems measured at L=0.15 rlu in the UHV chamber of the ID03 beamline. As reference system we used the square approximant surface unit cell of the NdGaO3 (110) surface. In the map several families of peaks are visible. The ones at H and K integers and H half integers are truncation rods of the substrate. The peaks at H and K multiple of 0.92 rlu are peaks from the BZY film. The other small peaks at H and K = n ± m*.08, with n and m integers (in the case of H, n also half integer) are due to the MF dislocation network.

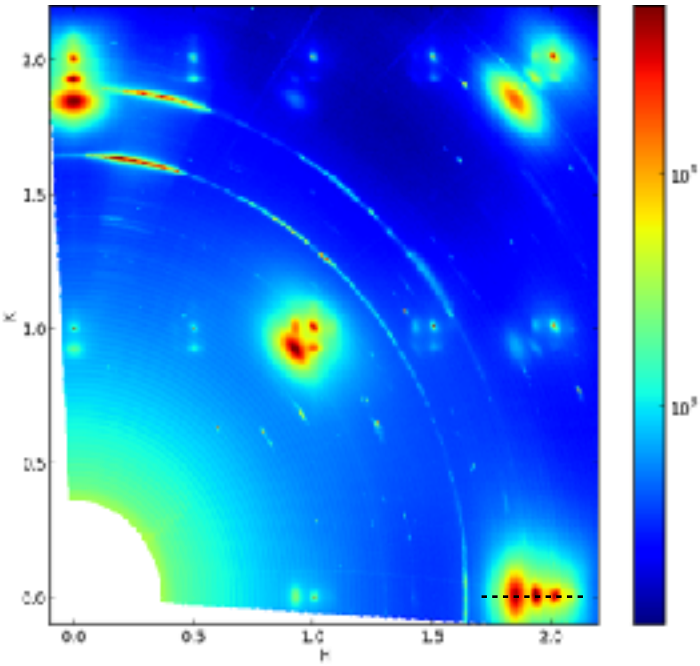

Figure 1) X-ray intensity map collected at L=0.15 rlu from a 6 nm thick film of $BaZr_{0.8}Y_{0.2}0_{3-y}$ film grown on the pseudo cubic (110) surface of NdGaO3. See text for more details.

The rings are due to some residual of silver print paint we used to perform impedance spectroscopy measurements.

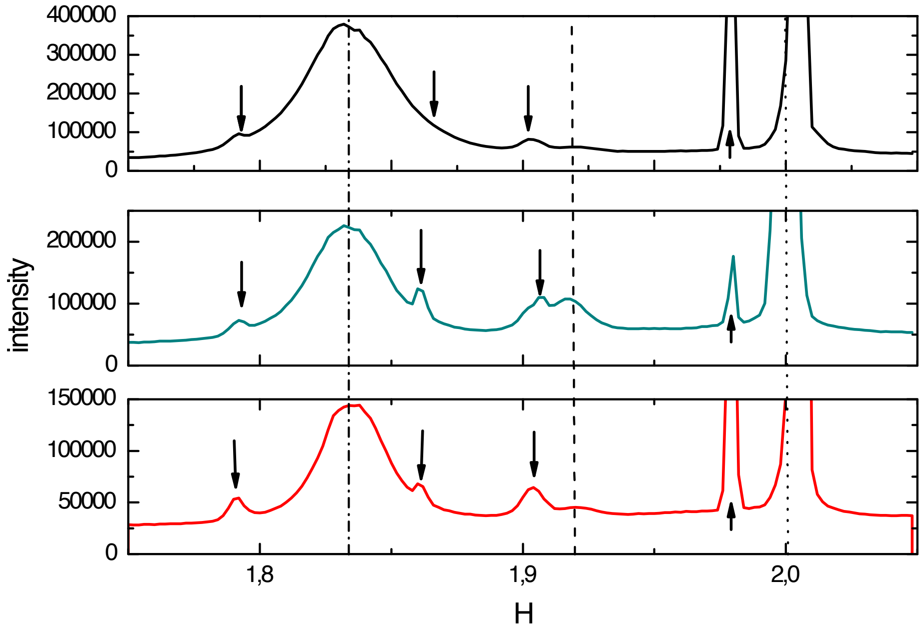

Figure 2) X-ray intensity plot along the dashed line of fig. 1. The diffraction peaks of the BZY film and NGO substrate are the ones at ~1.835 rlu and ~2.0 while the MD peak is at about 1.92 rlu. On the top is the intensity plot of the pristine film, in the centre after exposure at a humid atmpsphere for 4 hours at 450 °C and in the bottom after annealing at 450° in a dry environment. From the above plots it results that the intensity of the MD peak depends strongly on the exposing atmosphere highlighting the role of the MD in the protonization of the interface.

In figure 2 we show an intensity plot along the dashed line of fig. 1 at H=0 and K in the range 1.75-2.05 rlu. The plot includes intensity from the BZY film, the substrate (2,0,0.15) CTR and the dislocation network at (1.92, 0, 0.15). The other small maxima present in the plot and indicated with arrows are are due to the Be dome of the flow reactor.

Here we only report the data for the 6nm film. Similar results have been obtained for the 20 nm films.

In the two other thicker samples (50 and 100 nm) we have studied the MD peak was not visible.

The beamline worked fine and we have been able to answer to most of the questions listed in the proposal. We plan to submit a new proposal to better understand the loading dynamics of this class of systems and to better determine the stability upon cycling. These two last questions resulted from the results of this experiment.

The support from the ID03 staff was of very high quality.