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:** STRUCTURAL EVOLUTION OF FERROELECTRIC BIFEO3 UNDER INTENSE ELECTRICAL EXCITATION	**Experiment number**: MA-1570
Beamline: BM08 GILDA	**Date of experiment**: from: 15-5 to: 21-5 from: 17-9 to: 24-9	**Date of report**: 2-10-13
Shifts: 18 +21	**Local contact(s)**: F. d'Acapito	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Andrea Martin (*) (CNR-IOM)

Report:

Exp MA-1570 originally aimed at studying the local order in ferroelectrics under intense electrical fields, in particular in the case of $BiFeO_3$. However, due to unexpected difficulties with sample preparation, $Pb(Ti_xZr_{1-x})O_3$ (PZT) was studied at its place. The sample consisted in a $DyScO_3$ substrate (roughly 10*10mm^2) supporting a lower $SrRuO_3$ electrode (50 nm thick) and the PZT layer (100nm) topped by a 40nm Pt electrode (spatially patterned in patches of 300*800 μm^2). The experiment presented several critical points like: realization of a suitable circuit for electrical excitation, preparation of a suitable metallic pattern on the sample in order to limit the sample capacitance to a few nF, use of the low intensity 4 bunch mode and use of a microbeam (200*200μm^2) for the analysis of the single capacitor. In the two experimental sessions the overall feasibility of the experiment has been demonstrated. It has been possible to apply fields up to 200 MV/m on the sample synchronous with the X-ray flashes and with a time duration of 300ns. XANES spectra of good quality at the Zr-K edge with and without excitation were collected. Some differences have been evidenced, however, further glitches prevented us from safely drawing conclusions namely:

- a deterioration of the sample was observed after one night of data collection under excitation. It was not possible to determine the exact moment as no continuous sample check protocol was adopted.

– it was not possible to repeat the measurement in order to confirm the observations on a different capacitor, as the remainder of the capacitors delivered had their contact unwired, presumably during to the trip from Univ. Wisconsin to the ESRF. It was not possible, during the experiment, to find a micro-welding facility.

In conclusion it will be necessary to solve the welding problem and to investigate in more detail the deterioration process prior a new application for beamtime.

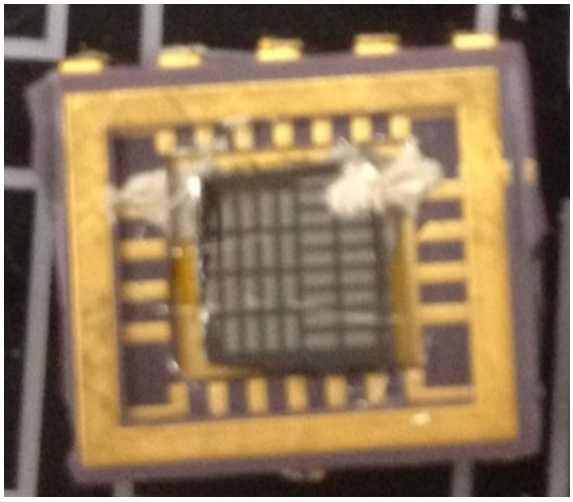

Fig. 1 *Picture of one of the patterned samples mounted in a chip carrier*

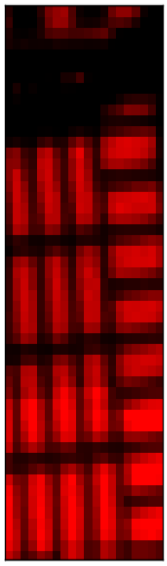

Fig. 2 *Elemental map (Pt Lα line) of the sample surface used to select the capacitor to investigate. The single capacitor dimensions are 300*800 μm².*

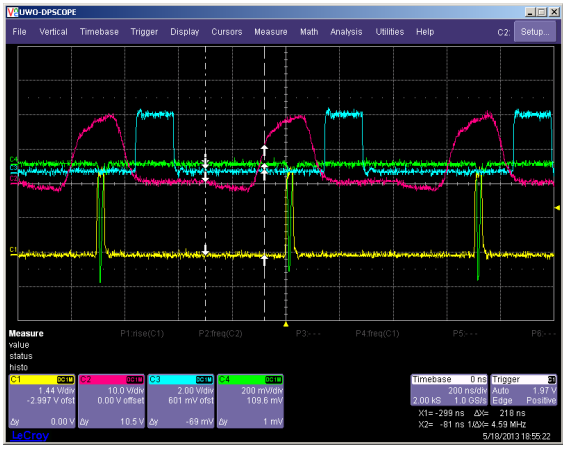

Fig. 3: *Oscilloscope screenshot of the various signals: yellow=time reference, Green=x-ray pulses read by a scintillator in the measurement chamber (non visible with the sample on the beam), blue= trigger, Purple= excitation pulse on the sample.*

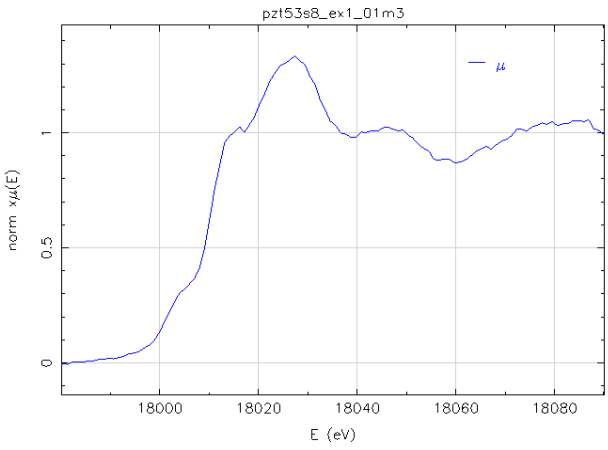

Fig. 4 *XANES spectrum at the Zr-K edge of a capacitor under 200MV/m excitation.*