EUROPEAN **S**YNCHROTRON **R**ADIATION **F**ACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

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 using the **Electronic Report Submission Application:**

http://193.49.43.2:8080/smis/servlet/UserUtils?start

Reports supporting requests for additional beam time

Reports can now 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:** Paraelectric-ferroelectric frustrated transition in $BaTiO_3$ with Nickel inclusions: Correlations with crystallographic structure	**Experiment number**: **2502718**
Beamline: BM25B	**Date of experiment**: from: September 24th, 2010 to:September 28th, 2010	**Date of report**:
Shifts: 18	**Local contact(s)**: Pilar Ferrer	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Carlos Pecharromán García.

Gustavo Mata Osoro

Fátima Esteban Betegón

Report:

Ferroelectric phase transition of $BaTiO_3$ (BT) implies coupled changes in the crystalline structure and electric properties. It was believed that this transition was due to a distortion in the distance Ti-O in the tetragonal phase. It has been observed that structural and compositional changes affect the phase transition in a manner that is far from clear[123]. In this regard, the inclusion of metal nanoparticles suppresses the ferroelectric behavior of BT but increases notably the relative permeability. Previously, we have found that in BT samples with high Ni content, the dielectric permittivity was reduced at high applied bias voltage[4], as it happens in the paraelectric phase, but nothing was said about any possible structural change near the transition. In this context, the obtained results have supplied new information about the nature of ferroelectricity suppression by metal particles.

Samples were prepared in the Instituto de Ciencia de Materiales of Madrid (ICMM-CSIC) with Ni concentration ranging from 0 to 30%. X-ray diffraction measurements of these samples were taken using the six-circle diffractometer in the B branch of BM25 beamline. Simultaneously, we performed electrical measurements of capacitance and dielectric losses (using a LCR meter Agilent e4980) applying bias Voltages from 0 to 40 V at temperatures from 50º to 130º. All the measurements were performed using 18 shifts.

We expected to affirmatively answer the question if the suppression of the ferroelectricity in BT due to the inclusion of Ni particles had a counterpart in the crystalline structure. This is so in basis of the results obtained in this experiment. We have observed that:

- The phase change carries a change in the crystalline structure in the BT matrix. In samples of BT with Ni concentrations of 28% it was observed that above 70º, the material present a paraelectric behavior, observed a diffraction peak corresponding to the cubic phase. Below that temperature, the sample showed ferroelectric behavior with diffraction peaks corresponding to the tetragonal phase. The

change of interplanar distance vs. temperature for Ni and BT under an applied electric field appears in figure 1. When temperature reaches 90º, the relative difference between (002) and (200) planes in BT reduces until they take the same value becoming equal in the cubic phase. In that case of Ni, the atomic spacing always increases under increasing electric field. These data indicate that Ni particles suffer expansive stresses as a consequence of a net compressive of BT grains.

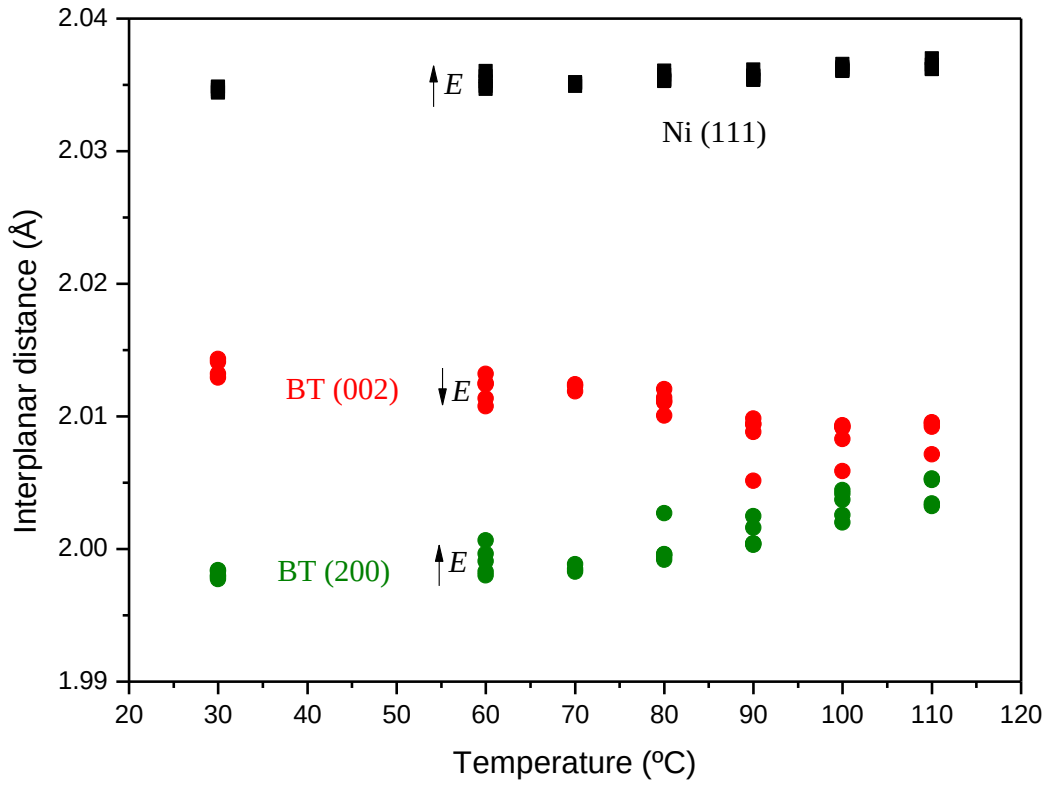

Figure 1: Variation of (002) and (200) interplanar distance in BT (red and green circles, respectively) and (111) in Ni(black squares) vs. temperature under different external electric field for a 28% Ni/BT sample. Arrows indicate the increasing external electric field.

All these data and the subsequent analysis is being processed and will be published shortly.

[1] S. Wang, G.O. Dayton, J. Am. Ceram. Soc., 82, [10], 2677, (1999).
[2] A. I. Frenkel, M. H. Frey, D. A. Payne, J. Synchrotron Rad., **6**, 515, (1999).
[3] S. Wada, T. Suzuky, M. Osada, M. Kakihana, T. Noma, J. Appl. Phys., **37**, 5385, (1998).
[4] R. Jiménez, F. Esteban-Betegón, C. Pecharromán, J. S. Moya, C. Alemany, Ferroelectrics, **268,** 456, (2002).