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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 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:** Study of Local Atomic Arrangement in Relaxor Ferroelectric Na0.5Bi0.5TiO3 by Means of X-Ray Diffuse Scattering Holography	**Experiment number**: HS-3423
Beamline: ID11	**Date of experiment**: from: 7/2/2008 to: 13/2/2008	**Date of report**: 25/2/2008
Shifts: 18	**Local contact(s)**: Dr. Jonathan P. Wright	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Andrea Lausi*

Sincrotrone Trieste, S. S. 14 – km 163.5 in Area Science Park, 34012 Basovizza – Trieste, Italy

Miloš Kopecký*, Jan Fábry*, Jiří Kub*

Institute of Physics, Academy of Sciences of the Czech Republic, Na Slovance 2, 182 21 Prague 8, Czech Republic

Report (preliminary):

The aim of this experiment was to determine the short-range arrangement of atoms in $Na_{0.5}Bi_{0.5}TiO_3$ (NBT) relaxor ferroelectric by means of the x-ray diffuse scattering holography. Our plan was to measure the x-ray holograms using the anomalous diffuse scattering from the Bi atoms at photon energies close to the bismuth L_{III} absorption edge (13.419 keV). The real-space reconstruction of these holograms results in the local neighbourhood of bismuth cations providing thus the short-range order information.

Finally, with respect to the possibility to reach very high energies at the ID11 beamline, we decided to carry out the experiment using the bismuth K-edge at 90.526 keV. Although the variations of the real-part of the form factor at the K-edge are smaller than those at the L-edge, this disadvantage seems to be compensated by negligible absorption in the sample and by very low scattering in the air.

The sample was the NBT single crystal (rhombohedral, space group R3c) in the form of a 50 μm thick plate, the sample surface was parallel to the (111) crystallographic plane (using the notation of a pseudocubic unit cell). The sample was illuminated by a monochromatic beam of the cross-section of 200×200 μm^2. The intensity patterns were recorded in the transmission mode using a large CCD detector (array of 2048×2048 pixels of the size of 51×51 μm^2) positioned at the distance of 200 mm behind the sample. The reciprocal space was scanned by rotating the sample around the axis perpendicular to the incident beam in the range from -180° to 180°. We measured such a large region of the reciprocal space because we want to reconstruct the local atomic structure without using the

"long-range" symmetry deduced on the basis of Bragg reflections. The sample was moving during each exposure in 0.5° steps. The acquisition time for each step was chosen between 3 s and 25 s depending on the energy; the aim was to get diffuse scattering patterns without an oversaturation of detector pixels when passing through the Bragg reflections. Data were collected at photon energies E_1 = 90.5 keV (at the absorption edge), E_2 = 90.9keV (above the edge), and E_3 = 80 keV (below the edge). Several data sets were collected for each energy in order to improve the data precission.

The most critical part of holography experiment consists in the data processing. First, the background and the scattering from the air have to be subtracted using scattering patterns collected with no sample present. The data have to be corrected carefully also for the fluorescence, the inelastic scattering, the beam polarization, the absorption in the sample, and the detector efficiency. It is also necessary to remove numerically the intense Bragg reflections using a median filter. Then sets of the corrected experimental data points can be transformed into a regular grid in the reciprocal space resulting in 3D diffuse scattering patterns. Finally, the difference of the intensity patterns collected at two energies with different contributions of the anomalous scattering can be treated as a hologram and the real-space image can be reconstructed by using the inverse Fourier transform. This procedure is time-consuming and it has not been finished yet, because all corrections have to be done with the precision of the order of 1 percent of the measured signal in order to get a good hologram. However, we have already some preliminary results. Fig. 1 shows a three-dimensional map of the reciprocal space constructed by using a set of 720 diffuse scattering patterns collected at the energy of 80 keV. Fig. 1 represents the mesured intensities partially corrected using the procedures described above, it is not a hologram. Inspite of "non-ideal" data processing, the inverse Fourier transform provides the local pair distribution function (see Fig. 2) with clear peaks corresponding to all atomic pairs. Fig. 2 shows that there is no chess-board alternation of Na and Bi cations. But the main question, i.e. wheather the bismuth atoms are grouped together or not, requires more extensive data processing.

Although a lot of work has to be done before extracting all the information from the experimental data, the use of the diffuse scattering holography seems to be very promising for the study of the short-range ordering in ferroelectric materials.

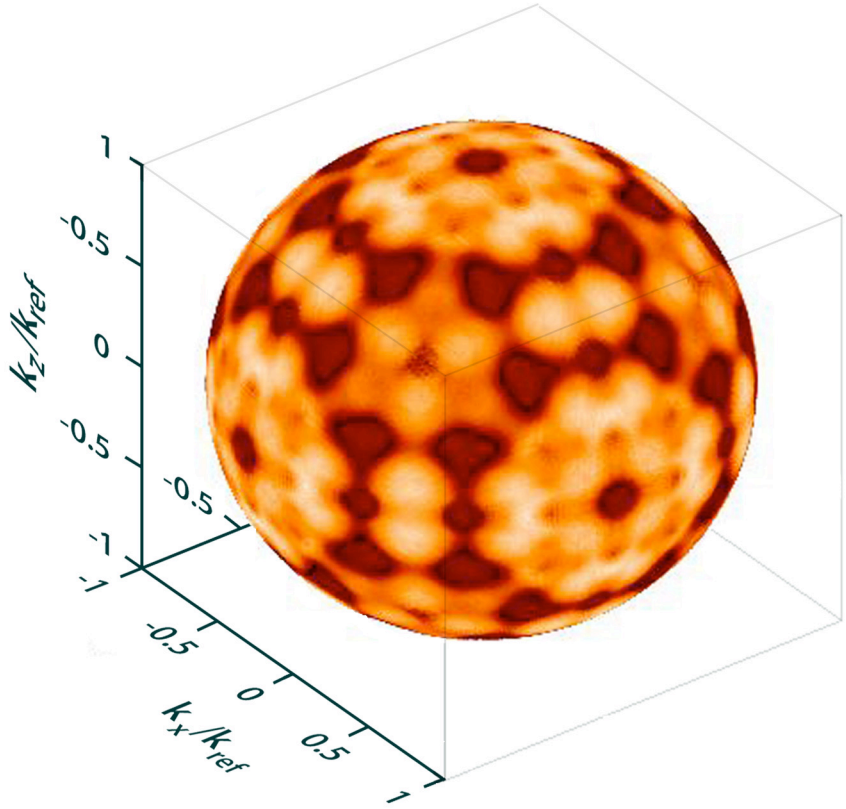

Figure 1: The three-dimensional x-ray diffuse scattering pattern of the $Na_{0.5}Bi_{0.5}TiO3$ single crystal measured at the photon energy of 80 keV. The scale is related to the wave number k_{ref} of 20 keV photons, $k_{ref} = 10.136$ Å^{-1}.

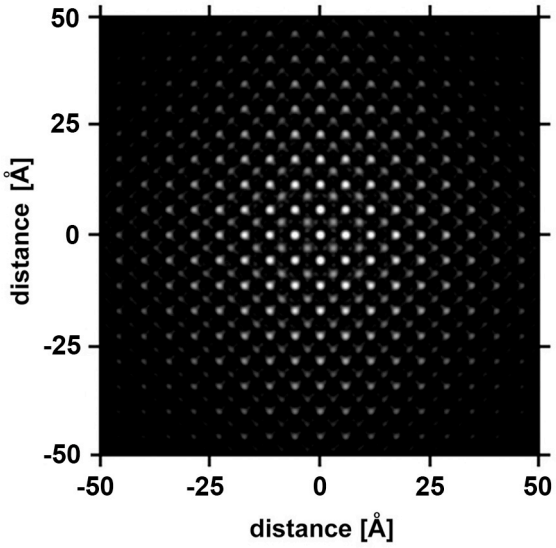

Figure 2: The pair-distribution function reconstructed from the pattern in Fig. 1 in the plane parallel to the crystallographic plane (001) at $z = 2a$ ($a = 5.94$ Å). The prominent peaks represent the Bi(Na)-Bi(Na), Ti-Ti, and O-O pairs of atoms whereas the weaker ones correspond to the Bi(Na)-O, Ti-O, and O-O pairs.