ESRF

ESRF	**Experiment title: High pressure XAS investigation of relaxor ferroelectrics**	**Experiment number**: HS2115
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Report:

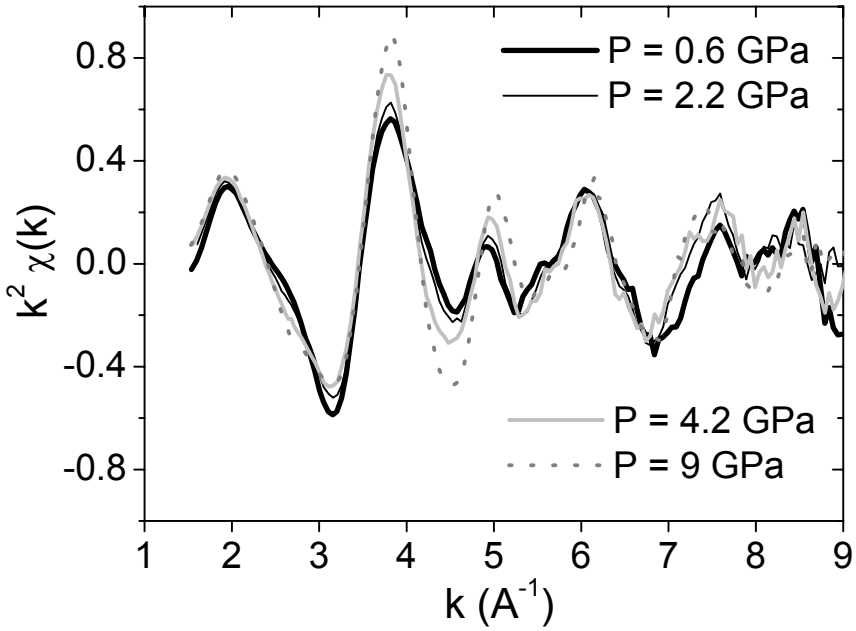
The aim of the experiment was to investigate the effect of pressure on the local structure in relaxor ferroelectrics. Relaxors form a special class among ferroelectric materials with outstanding electromechanical properties of which the understanding remains a challenging problem. It is generally admitted that their dielectric properties are related to the existence of different nanoscale polar regions. Therefore local order studies of these materials are of tremendous importance. The interest of applying pressure to such systems has been previously demonstrated from our Raman studies [1] and from our recent X-ray diffraction studies performed on beamline ID30 at ESRF [2,3,4] which both revealed clear structural evolutions under pressure.

In the initial project, we planned to collect EXAFS data as a function of pressure , using a diamond anvil cell, on two typical relaxor systems : $(Na_{0.5}Bi_{0.5})TiO_3$, (NBT) at the Ti (K) and Bi (LIII) edges and $Pb(Mg_{1/3}Nb_{2/3})O_3$ (PMN) at the Pb LIII and Nb K edges. We first had to find experimental conditions leading to reproducible EXAFS spectra. Spectra were recorded in transmission mode. In order to select an homogeneous part of the sample, we had to reduce considerably the beam size to 50 μm (diameter of the pressure cell window : 200 μm). We succeeded in studying the effect of pressure at the Bi LIII edge (13.4 keV) in the NBT sample but had no time to study neither the Ti edge nor the PMN sample. Since the sample

thickness for an absoption edge equal to one at the Bi LIII edge is 43μm. The mass of the NBT sample in the beam was ≈ 1 μg.

XAS spectra were measured at 0, 0.9, 1.5, 2.3, 3.2, 4.6, 5.5, 7.2, 8.2, 9.5 and 11.9 GPa at room temperature. After a careful procedure, all contributions of diffraction peaks, originating from the diamonds of the pressure cell, could be avoided in the interesting energy range [13.3-13.9 keV]. The acquisition of 4 spectra at each pressure was necessary in order to eliminate non reproducible artefacts. The time duration of one spectrum was about 30 minutes (counting time : between 2 and 4 seconds per point)

Several EXAFS oscillations($k^2\chi(k)$) at the Bi LIII edge are represented in the figure below. A marked evolution is observed under pressure. From the comparison with X-ray diffraction data under pressure [4], it is clear that the pressure evolution of the local order around the Bi cation and of the average long range structure are different. Especially, the rhombohedral to monoclinic transition occuring between 1.6 and 2 GPa has no important consequences on the EXAFS spectra, which can be understood if the local order symmetry does not change when the crystal undergoes an average structural phase transition, as it is the case in $KNbO_3$ under pressure [5]. A paper presenting these results and their quantitative analysis is in preparation.



Pressure evolution of the EXAFS oscillations at the Bi LIII edge in $(Na_{0.5}Bi_{0.5})TiO_3$ at room temperature

The success of this experiment, despite its difficulties, opens the route towards investigations of local order under pressure in relaxor ferroelectrics.

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[2]J. Kreisel et al, ESRF experiment report HS1813 (2002).
[3] Chaabane B., Kreisel J, Dkhil B, Bouvier P., Mezouar M. 2003, Phys. Rev. Lett 90 : 257601
[4] Kreisel J, Bouvier P, Dkhil B,Thomas PA, Glazer AM, Welberry TR, Chaabane B, Mezouar M. 2003, Phys. Rev. B 68: 014113
*[5] Frenkel AI, Wang, FM, Kelly S, Ingalls R, Haskel D, Stern EA, Yacoby Y, Phys.Rev. B **56** 10869 (1997).*