ESRF	**Experiment title:** Temperature dependence of ferroelastic domain wall structures from diffuse scattering in perovskite polycrystalline ferroelectrics.	**Experiment number**: 25-01-932
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Report: The experiment focused on determining the temperature evolution of the ferroelectric domain wall structure in the morphotropic phase boundary $0.94Bi_{0.5}Na_{0.5}TiO_3$-$0.06BaTiO_3$ (BNT-6BT) ceramic system by mean of high resolution X-ray diffraction. We measured XRD patterns at room temperature of powders and bulk ceramics of each tested composition. Due to beamtime constrains only some characteristic difraction peaks were selected to measure the diffuse scattering as a function of the temperature over the range from 100 K to 400 K. A beam energy of 30 keV was selected in order to measure the difraction pattern in transmition mode for bulk ceramics.

We measured the following samples:
(1) BNT-6BT (powder) obtained by solid-state reaction
(2) BNT-6BT (bulk) sintered from 1)
(3) BNT-6BT (bulk) obtained by sol-gel
(4) La-doped BNT-6BT (bulk) obtained by solid-state reaction
(5) Mn-doped BNT-6BT (bulk) obtained by solid-state reaction

Three different analysis can be done from the obtained results:
(i) to reveal the crystallographic structure of the studied materials, which is particularly relevant in morphotropic phase boundary (MPB) compositions where a (pseudo)cubic phase is detected from conventional X-ray diffraction analysis, as in the case of the samples (1) and (2). The ferroelectric behaviour of the BNT-6BT system must be correlated with a low symmetry (ferroelectric) phase. The structural origin of the ferroelectric behaviour of bulk BNT-6BT ceramics (sample (2)) could be revelead from the obtained measurement.
(ii) to detect temperature-driven phase transformations in MPB composition in order to reveal the scope of the phase boundary, i.e. the temperature stability of the boundary.
(iii) to determine changes in the ferroelectric domain wall structure as a function of temperature. This study may contribute to underestand the correlation between domain wall structure and macroscopic properties in MPB systems.

Figure 1 shows two characteristic peaks of the BNT-6BT XRD pattern for the samples (1) and (2). Splitting for the 111 and 200 reflexions are expected in rombohedral and tetragonal structures, respectively. However, no splitting are detected for the powder sample (1) confirming a (pseudo)cubic structure in this case. However, a notably different shape is observed for the bulk sample (2). Here, both the 111 and 200 reflexions seem to be splitted. This result postulates that a phase transformation occurs when the pseudocubic powder is consolidated as a bulk polycrystal. Complementary studies have been carried out by using Raman confocal. Results from both high energy XRD and Raman confocal evidence that a stress-induced phase transformation occurs in MPB BNT-BT, which is the base of the observed ferroelectricity in this compound. This result has been presented at the 14th International Conference of the European Ceramic Society (ECerS XIV) with a title “Crystallographic and diffuse scattering studies in BNT-BT system from high energy X-ray radiation in temperature”. A research article is now in preparation with a tentative title “Strain induced ferroelectric phase in morphotropic phase boundary BNT-BT piezoceramics”.

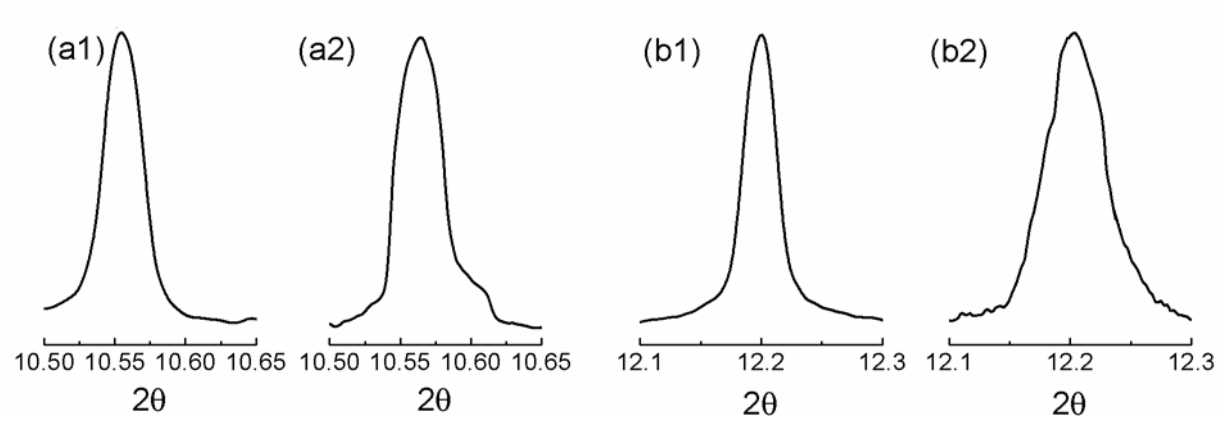

Fig.1. Reflexions 111 (a) and 200 (b) for powder (1) and bulk (2) BNT-6BT ceramics at room temperature.

Figure 2 shows the temperature dependence of the 110 diffraction peak for powder sample (1). This reflexion would exhibit splitting for both tetragonal and rhombohedral structure. As can be observed no splitting is detected taking into account the measurement resolution. Only a shift toward lower 2-theta values is identified as a result of the thermal effect, i.e. a higher d-spacing is observed with increasing temperature as a consequence of the thermal expansion of the lattice. Thus, no temperature-driven phase transformation is detected in MPB BNT-BT system, indicating that the boundary is thermally stable in a wide range of temperatures.

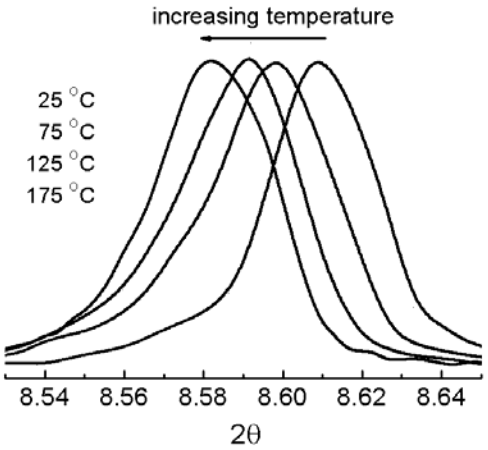

Fig.2. Temperature evolution of the 110 reflexion of powder BNT-6BT ceramics.

Unfortunately, the collected data do not allow to determine changes in the ferroelectric domain wall structure because statistically negligible differences was obtained for the diffuse scattering as a function of temperature in the study samples.