	Experiment title: Domain structure induced phase of PZN-PT	Experiment number: ME - 1039
Beamline: ID15A	**Date of experiment**: from: 8.5.2005 to: 18.5.2005	**Date of report**: 31. 8. 2005
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Names and affiliations of applicants (* indicates experimentalists):
Ross Piltz*, Bragg Institute, ANSTO, Lucas Heights, Australia
Klaus-Dieter Liss*, Bragg Institute, ANSTO, Lucas Heights, Australia
Simon Redfern, Earth Sciences, Cambridge, UK

Relaxor ferroelectrics are a hot scientific topic, with interest partially driven by the fundamentals of their extraordinary piezoelectric properties and by the billion dollar industry in which they find application. Many versions [1] of the phase diagram of the lead-based relaxor PZN-PT ($Pb(Zn_{2/3}Nb_{1/3})O_3$-$PbTiO_3$) exist, though it now appears that much of the confusion is due to a skin effect (~50 um) and the use of low energy X-ray studies on such lead-rich materials [2]. For less than 11% PT it was generally accepted that the stable phase was rhombohedral at room temperature transforming to cubic at high temperature. However, a stability band for a tetragonal phase between these two phases has been found for PT proportions of 4.5 % [3] and 8 % [4]. Recently, it has been proposed that for pure PZN a frustrated cubic phase exists and this phase has been denoted "Phase X" by the authors [5]. The authors attribute its unusual properties to structural frustration resulting from the interaction of nanometer sized polar domains found in PZN-PT and other relaxor ferro-electric systems.

Similar experimental observations [3] have already been seen in PZN-4.5%PT as it is cooled from the cubic phase, namely the apparent cubic symmetry and the diffuse phase transition spread over more than 100 K. In this case, details of the peak broadening and peak shifts are consistent with a transition from a cubic to tetragonal phase that is partially inhibited by the mechanical interference between the tetragonal and remanent cubic phase. The aim of the experiment was to obtain high-resolution data on PZN and PZN-4.5%PT to test if phase X is in fact the rhombohedral or tetragonal phase distorted by a twin domain structure. The ID15A diffractometer was used in triple-crystal mode using the perfect Si crystal sets in non-dispersive setup [6,7]. PZN and PZN-4.5%PT samples were heated from room temperature to the cubic phase and returned to room temperature while measuring high-resolution scans to investigate the emergence of the domain structure and any induced strain.

Our experimental results show that Phase X at room temperature is in fact a cubic phase combined with a strained rhombohedral phase. Typical results at room temperature are shown in Figure 1. The central peak is actually composed by two peaks slightly offset corresponding to the cubic and rhombohedral phases while the peak to the right is from the rhombohedral phase. Unfortunately, a technical problem with sample heating limited our high-resolution scans to room temperature which meant we were unable to answer the greater question of how such a dual phase situation arose, and what was the origin of the extended phase transition.

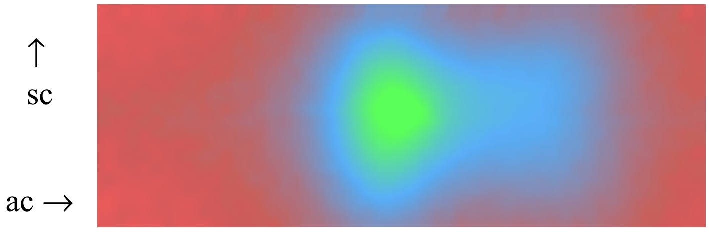

Figure 1. High-resolution scan of motor *sc* (sample angle) versus *ac* (analyser angle) for the 222 reflection at room temperature.

The sample heating problem arose from the "blower" furnace used for heating and our method of mounting the sample. As relaxor ferroelectrics are very sensitive to external stress the sample was mounted using high flexibility glue so that minimal stress occurred during thermal expansion. It was observed that the blower furnace oscillated in temperature by ±0.1 degree. Unfortunately, during these small oscillations the sample could suddenly rotate by as much as 4 arc-seconds. Different gluing methods reduced this rotation to 2 arc-seconds, though even this did not result in useful high-resolution scans at high temperatures. Only at room temperature was the temperature was sufficiently stable to perform useful high-resolution scans such as in Figure 1.

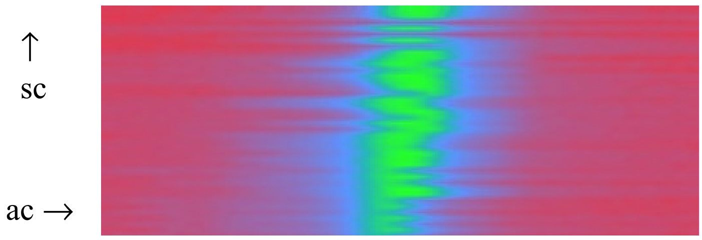

Figure 2. Example of random rotations in sc during an ac versus sc scan. The correct image should be a straight vertical line in the center of the image.

A future ID15 experiment is planned using a high stability vacuum furnace to eliminate the above random rotations. Combining this with the experience obtained in this experiment it is expected that a definitive result will be obtained on the origin of the "Phase X" behaviour.

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