

High Resolution Neutron Powder Diffraction a Singular Application

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There has been continuing controversy concerning the crystal structure(s) of materials exhibiting the Giant Piezoelectric Effect, particularly in the $\text{PbZn}_{1/3}\text{Nb}_{2/3}\text{O}_3-x\text{PbTiO}_3$ (PZN-PT) system at $x < 9\%$. The materials are relaxor ferroelectrics with perovskite based crystal structures that depart only slightly from a cubic metric although the ion coordinates are far from cubic. It has been variously claimed that, after electric poling, the poly-domain single crystals are rhombohedral, monoclinic, orthorhombic and even a cubic core with rhombohedral exterior called the X-phase. Our own high resolution neutron powder diffraction experiments on as-grown PZN-PT have unequivocally supported the older more conventional view that the materials are primarily rhombohedral even when quite large 0.5-1mm powders' were used. However, there is always the possibility that either a macroscopic crystal may behave differently, or that claimed electric field induced phase transitions may cause a different symmetry to be adopted which is then retained upon removal of the field due to hysteresis (remanent polarisation). We have therefore exploited the unsurpassed resolution of the High Resolution *Powder* Diffractometer at the ISIS facility, Rutherford Appleton Laboratory, UK in a *single crystal* neutron diffraction study of PZN-4.5%PT. Crystals were rotated with a low-index zone-axis vertical and time-of-flight neutron diffraction patterns recorded using only the equatorial segment of the backscattering detector. The data so recorded are analogous to the zero layer of a single crystal rotation photograph, however with superb resolution. The recording of complementary zones allows a complete structure to be investigated. Despite the relatively low intensity in this demonstration experiment, it has proven beyond doubt that the crystals are rhombohedral at room temperature even after electric poling. The upgraded HRPD will be able to record such patterns in just 1-2 hours.