## **Functional Materials**

## MS.3.P073 TEM and other chracterizations of PbNb<sub>2</sub>O<sub>6</sub> for high temperature piezoelectric applications

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Materials with much higher Curie temperature than that of presently popular piezo-electric materials like barium titanate (BaTiO<sub>3</sub>) and lead zirconate titanate (PZT) are often needed. A high temperature piezo-electric material like Lead Meta-Niobate, PbNb<sub>2</sub>O<sub>6</sub> (PNO), must be used in the ultrasonic imaging system that can "view" nuclear fuel rods under molten metal (often, liquid sodium) in a Fast Breeder Reactor (FBR). This helps eliminate mis-alignment of the rods and ensures highrer safety of FBRs that simultaneously produce energy and nuclear fuel. One has to look for such fuels due to the limited stock of natural uranium globally and some countries in particular. PNO is piezoelectric only in its orthorhombic phase. It is one of the potential functional materials - in form of sensors and actuators - also for other high temperature applications like car exhaust. We have perfected the steps for the preparation of the meta-stable orthorhombic PNO, avoiding the competing rhombohedral phase and other compounds, leading to Curie temperatures (vide next paragraph), higher than those in recent reports. Check of no second phase has been concluded from XRD Rietveld Analysis and, at granular level, by High Resolution Transmission Electron Microscopy (TEM) on different grains. We discovered that starting materials govern the end product in PNO preparation. Microstructure of our ceramic PNO in orthorhombic and rhombohedral phases has been studied. Lattice planes and ferroelectric domains show up clearly in TEM pictures. Real part, Z', and imaginary part, Z", of electrical impedance, Z, (related to real and imaginary parts of permittivity) have been measured in an Impedance Spectrometer - up to ~ 700 °C over 20 Hz to 5.5 MHz range. The real part of permittivity peaks sharply, at  $(580 \pm 1)^{\circ}$ C at 5.5 MHz and at  $(573 \pm 1)^{\circ}$ C at 20 Hz, indicating the Curie Temperatures. Differential Scanning Calorimetry [1] showed an interesting endothermic minimum at ~ 570°C during heating in orthorhombic PNO but not in non-piezoelectric (rhombohedral) PNO, confirming the Curie temperature. Electron diffraction rings from the two phases (Figure 1) are indeed very different, and the symmetry of the pattern is lower in the rhombohedral sample than in the orthorhombic sample.

1. K.R. Sahu, and Udayan De, Thermochimica Acta 2009; 490: 75.



**Figure 1.** Diffraction rings seen by 200 kV Transmission Electron Microscopy of orthorhombic PNO (left figure) and of rhombohedral PNO (right figure).