

FA4-MS25-P01**TEM heating experiment on lead-free piezoceramics.**

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Lead-free piezoceramics are in the focus due to environmental concerns about lead-containing materials. $(\text{Bi}_{1/2}\text{Na}_{1/2})\text{TiO}_3$ (BNT)-based materials doped with other perovskite systems like BaTiO_3 (BT), $(\text{Bi}_{1/2}\text{K}_{1/2})\text{TiO}_3$ (BKT) or $(\text{K}_{0.5}\text{Na}_{0.5})\text{NbO}_3$ (KNN) are of special interest. They show depending on composition extraordinary electromechanical properties. Especially $(1-x-y)\text{BNT}-x\text{BT}-y\text{KNN}$ with $0.05 \leq x \leq 0.07$ and $0.01 \leq y \leq 0.03$ show high strain under applied electric fields [1]. For room temperature the structure is a mixture of tetragonal and rhombohedral phase with phase fractions depending on composition [2]. Both phases are present in single grains [3] and exhibit oxygen octahedral tilting which leads to superlattice reflections in the diffraction patterns (DP).

To clarify the phase diagram and to understand the phase evolution for heating up to the Curie temperature (T_C) and cooling down from T_C , transmission electron microscopy (TEM) heating experiments were performed on different compositions of the BNT-BT-KNN system. Bright-field images and associated DP for different temperature steps were recorded to trace the change in crystal structure by appearing or disappearing superlattice reflections and correlated to the visible microstructure. These results are discussed in context of electromechanical measurements.

[1] S.-T. Zhang, A. B. Kounga, E. Aulbach, H. Ehrenberg, J. Rödel, *J. Appl. Phys.* 2007, 91, 112906. [2] L. A. Schmitt, M. Hinterstein, H.-J. Kleebe, H. Fuess, *J. Appl. Cryst.*, 2010, submitted. [3] L. A. Schmitt, H.-J. Kleebe, *Funct. Mater. Lett.*, 2010, 3, 55

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FA4-MS25-P02**Comparative diffraction studies of lead-free piezoceramics**

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A comparative study of two lead-free piezoceramics, $(\text{Bi}_{0.5}\text{Na}_{0.5}\text{TiO}_3)_{0.92}-(\text{BaTiO}_3)_{0.06}-(\text{K}_{0.5}\text{Na}_{0.5}\text{NbO}_3)_{0.02}$ and $(\text{Bi}_{0.5}\text{Na}_{0.5}\text{TiO}_3)_{0.94}-(\text{BaTiO}_3)_{0.05}-(\text{K}_{0.5}\text{Na}_{0.5}\text{NbO}_3)_{0.01}$, hereafter termed 92-06-02 and 94-05-01, is presented. Samples were investigated by X-ray, neutron and electron diffraction. Transmission electron microscopy (TEM) and powder diffraction experiments clearly revealed the presence of a rhombohedral and a tetragonal phase with space group R3c and P4bm, respectively, both on a nanoscale level [1].

Using the technique of selected area electron diffraction combined with dark field imaging, the coexistence of tetragonal nanosized platelets and rhombohedral domains within one single grain was verified [2].

It is found that sample 92-06-02, with a high amount of nonpolar tetragonal phase, shows a grainy contrast, whereas specimen 94-05-01 feature domain like contrast, related to a higher rhombohedral phase fraction.

Further in situ cold-stage TEM investigations are in progress.

[1] Schmitt, L.A., Hinterstein, M., Kleebe, H.-J., Fuess, H., *J. Appl. Crystallogr.* 2010, submitted. [2] Schmitt, L.A., Kleebe, H.-J., *Functional Materials Letters*, Topical issue on lead-free ferroelectrics, 2010, 3, 55-58.

Keywords: diffraction, domains, ferroelectrics, piezoelectric ceramics, transmission electron microscopy