

the use of *in situ* method on X-ray diffractometer. The phase changes observed may be explained in the following way: at low synthesis temperatures and relatively short-time calcinating (2 hours) the generation of KNbO₃ as least(possibly nano) particles. This supposal is proved by the fact that known ferroelectrics (BaTiO₃, PbTiO₃ etc.) in nanocrystal condition are characterized by the increased cell sizes and lessened values of spontaneous deformation. The increased cell volumes could be the consequence of large concentrations of defects. On increase of calcinating temperatures, the reduce of the cell volume might be the consequence of either increase of crystalline sizes or of the reduce of defects number. Until now the phenomenological study of ferroelectric phase conditions was observed from the point of view of outer effects(temperatures, pressures, electric fields) that's why including the parameters of real conditions of objects into complete theoretical observation.

Keywords: ferroelectrics, phase transitions, synthesis

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Modulated crystal structure of RE_{1.67}AE_{0.33}NiO₄ in charge ordering state

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Stripe-type charge ordering (CO) [1], as found in some layered perovskite-type transition-metal oxides, is a typical example of self-organized electrons and also responsible for novel electric properties, such as the colossal magnetoresistance and electroresistance that are expected to be applied to the next generation memories [2]. Therefore, investigation of the CO structure is important for material design in layered transition metal oxide systems [3]. In this work, we report on the modulated crystal structure associated with the CO in the single-layered nickelates RE_{1.67}AE_{0.33}NiO₄ (RE and AE are rare-earth and alkaline-earth ions, respectively). The relation between A-site cations and CO structure has been investigated systematically by means of transmission electron microscopy (TEM). Long-range ordered charge stripes with a commensurate modulation wave number along the b-axis (the longer axis in the ab plane) are observed in the orthorhombic single crystal Pr_{1.67}Ca_{0.33}NiO₄ with minimal disorder effect. Change of A-site combination to Sm and Sr, the CO correlation along the b-axis becomes short-range. An abrupt decrease of the CO correlation length along the c-axis with the increase of disorder revealed that the charge stripe ordering collapses via the stacking faults of the charge stripes, indicating that the quenched disorder affects the CO structure in the layered nickelates.

Keywords: modulated crystals, transition-metal oxides, TEM

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Investigation of differently synthesized Co-Ni-Al spinels and precision of the crystallographic data

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Spinelns comprise a large group of compounds with high importance in industry. Our investigations focus on the influence of different synthesis methods on both, the crystallization path and on the crystal chemistry of the spinel product. Additionally to the materials aspects, the precision and reproducibility of the crystallographic analyses are of major interest. (Co,Ni)Al₂O₄ spinels with four different Ni:Co ratios were synthesized by 4 different methods: from impregnated carbons, by sol-gel methods, and by calcination of oxides and of salts. Samples were calcined at temperatures between 550°C and 1300°C. All samples were measured in Bragg-Brentano geometry with Cu-K α radiation. Additionally, selected samples were analyzed in Debye-Scherrer geometry with Mo-Kα₁ radiation and by neutron diffraction at the E6 instrument at the Hahn-Meitner facility (Berlin). Rietveld refinements of diffraction patterns revealed that the synthesis path indeed has an influence on the spinels formed. Due to the different precursor materials spinels with different chemical compositions and varying amounts of by-phases are obtained. Neutron diffraction experiments performed on selected samples calcined between 1050°C and 1300°C show only minor changes of the Co and Ni distribution on the octahedral and the tetrahedral position with temperature. This indicates that already at lower temperatures the spinels formed are in equilibrium. Structure refinement strategies as well as the selection of instrument geometry and radiation source shows a strong influence on the refined structure data.

Keywords: spinel, crystallization of materials, data precision

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Phase transition studies in multiferroic Pb(Fe_{1/2}Nb_{1/2})O₃ and its solid solution with PbTiO₃

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Recent years have witnessed enormous interest in the multiferroic materials because of the presence of more than one ferroic/ anti-ferroic orders: such as ferroelectric, ferroelastic and ferromagnetic/ antiferromagnetic. Pb(Fe_{1/2}Nb_{1/2})O₃ (PFN), is a multiferroic exhibiting a diffuse ferroelectric phase transition at 385K and two antiferromagnetic transitions at 143K and 9K, respectively. It is an attractive material for use in multilayer ceramic capacitors and other electronic devices due to its high dielectric constant, diffuse phase transition behaviour and low sintering temperature. The phase diagram of its solid solution with PbTiO₃ (i.e. (1-x)Pb(Fe_{1/2}Nb_{1/2})O₃-xPbTiO₃ or PFN-xPT) shows a morphotropic phase boundary (MPB) similar to that in the well known Pb(Zr_xTi_{1-x})O₃ (PZT), (1-x)Pb(Mg_{1/3}Nb_{2/3})O₃-xPbTiO₃ (PMN-xPT) and (1-x)Pb(Zn_{1/3}Nb_{2/3})O₃-PbTiO₃ (PZN-xPT) ceramics. We present here the results of synchrotron powder x-ray diffraction, dielectric and magnetization studies on 'phase-pure' PFN and PFN-xPT. It was found for the