

parameters $a_1 = 10.534(2)$, $a_2 = 11.683(2)$, $a_3 = 20.407(4)\text{\AA}$, $\alpha_2 = 93.33(3)^\circ$. When lowering the temperature from 293 K, a strong attenuation of certain ultrasonic resonances occurs reaching a maximum at 288.4 K. Below this temperature the attenuation of ultrasonic waves, in which the elastic constants c_{11} , c_{22} , c_{12} and c_{66} are involved, is drastically reduced. If the low temperature phase is heated to room temperature, the attenuation reaches a maximum at 291.5 K indicating a hysteresis. This hysteresis is confirmed in DSC measurements, where a transition enthalpy of 2 J/g is observed. In the low temperature phase, domains with several nm thickness parallel to (001) are formed. In the direction [010] the angle of optical extinction of these domains deviates by about $\pm 6^\circ$ from the extinction in the high temperature phase. Both phases exhibit a topotactic structural relation, which however, does not prevent the single crystalline state of the mother crystal to be maintained after the transition. The phase transition is considered to be of weakly first order.

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Keywords: nitrilotriacetates, elasticity, phase transition

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Thermal Transformations of Some Alicyclic β -amino Acid Hydrates

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Alicyclic β -amino acids were recently used for the construction of two-dimensional hydrogen-bond grids that link molecules into bilayer structures [1]. While the crystallization of several β -amino acids yielded the expected bilayer type structures, others (*cis*-2-hydroxycyclohex-4-ene-1-carboxylic acid, all-*cis*-amino-4-hydroxycyclohexanecarboxylic acid, *trans*-2-aminocyclopentane-carboxylic acid and 3-*exo*-aminobicyclo[2.2.1]hept-5-ene-2-*exo*-carboxylic acid) crystallized as hydrates and did not form the expected grid pattern. These hydrates were analysed by thermogravimetry, differential scanning calorimetry, powder and single crystal X-ray diffraction in an attempt to prepare and characterize their anhydrous crystal forms. The results show that each powder loses the hydrogen-bonded solvent before decomposition. The powder patterns of the solvent-free forms show similarity to those of the bilayer structures.

[1] Fábrián L., Kálmán A., Argay Gy., Bernáth G., Gyarmati Zs. Cs., *Cryst. Growth Des.*, 2005, 5, in press.

Keywords: thermal analysis, hydrates, hydrogen bonding

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X-ray Diffraction Study on the Phase Transitions of Barium Titanate

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Structural phase transitions in as-grown single crystal of BaTiO₃ were studied by X-ray precession method in the temperature range between 420 K and 90 K. It was found that high temperature cubic phase, designated here as cubic I, is transformed into the room temperature phase (RTP at $T_1=403$ K), and further into the low temperature phase (LTP at $T_2=278$ K), in both of which closely related two structures coexist to form domains with a shape of multi-domain 'hybrid' structure. In RTP the coexisting structures are tetragonal I and monoclinic I and in the LTP tetragonal II and monoclinic II. On the other hand, the lowest temperature phase (below $T_3=183$ K) is in a single domain of a tetragonal III form. These observations are in disagreement with the phase transition sequence, proposed by previous studies, cubic→tetragonal→orthorhombic→rhombohedral.

The lattice constants of the tetragonal I form are $a_{\text{TI}}=0.4009(10)$ nm and $c_{\text{TI}}=0.4048(10)$ nm, and those of the monoclinic I form are $a_{\text{MI}}=0.4059(10)$, $b_{\text{MI}}=0.4009(10)$, $c_{\text{MI}}=0.5700(10)$ nm and $\beta_{\text{MI}}=135.33(10)^\circ$ at room temperature. Those of the tetragonal II form are $a_{\text{TII}}=0.4027(10)$ and $c_{\text{TII}}=0.3996(10)$ nm, and those of the monoclinic II form are $a_{\text{MII}}=0.3994(10)$, $b_{\text{MII}}=0.4027(10)$, $c_{\text{MII}}=0.5678(10)$ nm and $\beta_{\text{MII}}=134.77(10)^\circ$ at 233 K. Evolution and crystallographic relationship of the four phases forming the multi-domain 'hybrid' structures are presented.

Keywords: BaTiO₃, phase transitions, 'hybrid' structure

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X-ray and Neutron Scattering Study of as-grown Pb(In_{1/2}Nb_{1/2})O₃ Single Crystals

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Characters of static and dynamic structures of as-grown Pb(In_{1/2}Nb_{1/2})O₃ (PIN) at room temperature (RT) have been investigated by a complementary use of x-ray and neutron scatterings. The PIN crystal has different chemical ordering ($S_2 = 0 \sim 1$) in the arrangement of In³⁺ and Nb⁵⁺ on the B-site by a different thermal treatment.

The diffuse scatterings showing the relaxor state have been observed in the outer-layer of the crystals, while $h/4$ $k/4$ $l/4$ reflections showing the long-range antiferroelectric (AFE) state in the inside [1,2]. Phonon dispersion of the as-grown PIN crystals has also been measured, which is the first time for the PIN series. We have measured two transverse optic modes (TO1 and TO2) at zone center and concluded that the major part of the as-grown crystal at RT is ferroelectric (FE). This is consistent with the dielectric measurement performed on the as-grown PIN crystal [3].

These results show that the degree of B-site order S_2 is widely distributed in the as-grown PIN crystal from $S_2 = \sim 0$ to ≥ 0.7 . That is, the AFE grain locates in the FE matrix, while the outer-layer is Relaxor in the as-grown PIN.

[1] Nomura K., et al., *J. Phys. Soc. Jpn.*, 1999, 68, 39. [2] Nomura K., et al., *J. Phys. Soc. Jpn.*, 1997, 66, 1856. [3] Ohwa H., et al., *J. Phys. Soc. of Jpn.*, 2000, 69, 1533.

Keywords: ferroelectric properties, neutron and X-ray scattering, phase transitions

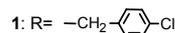
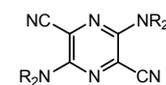
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Phase Relationship of Two Polymorphs of a Diaminodicyanopyrazine Dye

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2,5-Diamino-3,6-dicyanopyrazine dyes are novel fluorescent dyes for opto-electronic applications [1]. These dyes have been found to have crystal polymorphs with different colours depending on the amino substituents. The colour difference is considered to be ascribed to the different molecular conformation among the polymorphs. In this study, the stability relationship of two polymorphs (red and yellow) of **1** were determined by melting data and correlated with their molecular and crystal structures.



The red phase was found to transform to the yellow phase at room temperature. DSC measurement showed that the yellow phase has a higher melting point than that of the red phase. The heat capacities were also determined by DSC measurement. These results suggest that the red phase and the yellow phase are a monotropic relationship. The phase relationship will be also discussed on the basis of their