movement of a molecule can induce polymorphic transition into a new metastable form. The study has been supported by a grant from BRHE (RUX0-008-NO-06) and Integration projects 13 & 109 from the SB RAS.


Keywords: phase transitions in solids, drug polymorphism, low-temperature structure

FA4-MS35-P10

Unprecedented solid-state chemical reaction – from(C3N2H5)3SbBr6⋅H2O to (C3N2H5)3SbBr6; polymorphism of substrate and products, A. Gągor[a], A. Piecha[b], R. Jakubas[b] and A. Pietraszko[b]. Institute of Low Temperature and Structure Research PAS, Wroclaw, Poland. b University of Wroclaw-Jolio-Curie 14 50-383 Wroclaw, Poland
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A novel organic-inorganic hybrid material, tris(imidazolium) hexabromoantimonate(III) hydrate, (C3N2H5)3SbBr6⋅H2O that leads to multiphase crystallites dominated by ferroelectric (C3N2H5)3SbBr6. The room temperature structure of TIBA consists of discrete SbBr6 anions, disordered imidazolium cations, and water molecules forming a 3D array of hydrogen bonds. The solid-state chemical reaction leads from [SbBr6]3− to [Sb2Br11]5−. The creation of ferroelectric [C3N2H5]3SbBr6 through an “annealed” sample of (C3N2H5)3SbBr6⋅H2O is confirmed by X-ray diffraction phase analysis, dielectric spectroscopy, and pyroelectric measurements. The dielectric response of the electric permittivity and the critical slowing down of the process observed near 140 K in the “annealed” sample of TIBA are treated as a “fingerprint” of a neat (C3N2H5)3SbBr6 ferroelectric. Furthermore, TIBA undergoes isosstructural discontinuous phase transition at 212/221 K (cooling-heating) (P21/c ↔ P21/c). The phase transition mechanism is characterized by two contributions: an order-disorder (cationic substructure) and a displacive one (water molecules). Crystal structure of two polymorphs of second product of reaction: (C3N2H5)Br that crystallizes from water solution below 273 K are also presented along with the order-disorder phase transition mechanism.


Keywords: ferroelectric, halogenaoniminates(III), phase transitions

FA4-MS35-P11

Topotactic phase transformation of 4H-CuTa1.10S2 into 4H-Ta1.1S2, Bernd Harbrecht, Wenjie Yan, Otfrid Lemp, Steffen Zörb, Kirsten Bohnen
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Tantala forms a series of polytypic ternary sulfides of general composition CuTa1+xS2. The layeried-type phases are good electronic and ionic conductors. The mobile Cu ions (y) and excess Ta (x) are inserted in spatially separated van der Waals gaps of a layered TaS2 host structure the type of which depends upon x, y and T. Oxidative deintercalation of Cu with iodine affords new polymorphs of Ta1-0.5S2. The new polymorphs differ from those accessible from the elements at temperatures above 1250 K by higher order staging, i.e. excess Ta (x) is not found in all van der Waals gaps what confers lubricant-like properties to these Ta-rich disulfides. Such polypotypes can be classified by a code nH in which n counts the number of TaS2 slabs in the repeat unit and N refers to the lattice symmetry, e.g., H for hexagonal. Here, we present the structure of a ternary 4H polytype that – upon progressive deintercalation of Cu at ambient temperature - topotactically transforms into a differing 4H polytype. The transition is reflected in divergent X-ray diffractograms showing an offset of the reflections and a drastic change in the intensity modulation of h0l zonal reflections. Structural features of the two polypotypes derived from the distinctive intensity modulations are highlighted and discussed in the light of known polymorphs. Electronic and mechanistic aspects of the transformation are discussed.


Keywords: transition metal dichalcogenides, polytypes, topotactic phase transformation

FA4-MS35-P12

Elucidation of Crystallization Processes – The Polymorphism of Nifedipine, Maria Klimakow[a], Klaus Rademann b, Franziska Emmerling a, b BAM Federal Institute for Materials Research and Testing, Berlin, Germany. a Humboldt-University Berlin, Germany
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The crystallization process of the polymorphic compound nifedipine (4-(2-nitrophenyl)-2,6-dimethyl-3,5-dicarbomethoxy-1,4-dihydropyridine, C17H18N2O6) using different solvents was investigated in situ by X-ray diffraction and Raman spectroscopy. In medical treatments, nifedipine acts as dihydropyridine calcium antagonist. Despite the pharmaceutical importance, the wide use of the substance, as well as knowledge of its polymorphism, the crystal structures of most of the nifedipine polymorphs still remain unknown. To investigate the formation of intermediate phases, which are kinetically favored and formed preferentially under fast evaporation conditions, an acoustic levitator was employed as sample environment for the combination of time-resolved X-