

Poster Sessions

solvents were grown in situ on a diffractometer from a mixture of two or more liquid at ambient conditions compounds and structurally characterized. This method, pioneered in [1], differs from standard in situ crystallization technique as crystallization from melt differs from solution crystallization and may produce different polymorphs. The structures of new polymorphs of several compounds (cyclobutanone, ethylacetate, butanone) were obtained by this method. Their molecular structures and packing of molecules are described and compared with those of known polymorphs of the same or similar compounds. The intermolecular interactions determining the packing of molecules are analysed by various methods as well as possible factors affecting the packing. The technical details of experimental setup and data processing are discussed as well.

I. J. Bennet-Buchholz, T. Haumann, R. Boese; J. Chem. Soc. Chem. Commun., 1998, 2003-2004

Keywords: low-melting compounds, polymorphic structures, crystal growth

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Hydrothermal synthesis of (K,Na)NbO₃

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Lead-free perovskite KNbO₃-based solid solutions can exhibit piezoelectric properties comparable to that of actuator grade lead zirconate titanate piezoelectrics in the vicinity of the morphotropic phase boundary. Conventional high temperature processing of KNbO₃-based solid solution involves energy intensive and laborious processes with risk of getting non-stoichiometric compound due to potassium volatilization. In this study, crystalline (K,Na)NbO₃ solid solutions are synthesized hydrothermally at significantly lower temperature of 200 °C for the first time close to the morphotropic phase boundary by using a mixed NaOH and KOH solution. Above a certain critical ratio of NaOH to KOH concentration, a secondary NaNbO₃ perovskite phase always formed alongside the solid solution. Details of NKN phase formation, structural and composition in soft chemistry solution route were studied, including presence of different intermediate phases which potentially be technical barrier to synthesize pure NKN phase using single step hydrothermal synthesis. Despite this difficulty, a short 2 hour heat treatment at 800 °C was successfully used to convert the mixture of (K,Na)NbO₃ and NaNbO₃ powder to a single solid solution phase powder close to the morphotropic phase boundary. This opened a new possibility of reducing energy requirement in NKN powder compact synthesis via solid state reaction.

Keywords: hydrothermal synthesis, sodium potassium niobate, ferroelectric materials

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Perovskite-type LnFeO₃ (Ln= Y, Pr, Nd, Sm, Gd, Tb, Dy, Ho) prepared by mild hydrothermal method

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A series of the rare-earth orthoferrites LnFeO₃ (Ln= Y, Pr, Nd, Sm, Gd, Tb, Dy and Ho) that were characterized by powder X-ray diffraction with the orthorhombic perovskite lattice symmetry have been prepared under mild hydrothermal conditions. In hydrothermal processing of these materials it is found that their formation are mainly affected by the reaction temperatures, times, and appropriate alkaline solution range for different lanthanide elements, while the particle sizes of the samples which are applied to image by means of scanning electron microscopy vary significantly with the amount of OH⁻ anions in the reaction systems.

Keywords: hydrothermal, perovskite, orthoferrites

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Effect of anion adsorption on the hydrothermal growth of boehmite

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The effect of the adsorption of the anions as nitrate, chloride and sulfate on the hydrothermal growth of boehmite (AlOOH) was investigated in this paper. The experimental results indicated that boehmite nano-flakes with a width of about 50 nm and boehmite nano-fibres with a preferential growth along [100] were formed after hydrothermal treatment (240 °C, 16h) of the freshly precipitated alumina gel at pH=10.5 and pH=4.0, respectively. The anions were difficult to be adsorbed on the boehmite surface under alkaline condition, thus had little influence on the hydrothermal formation of the corresponding boehmite nano-flakes. The formation of the boehmite nano-fibres was promoted by the adsorption of the anions on boehmite surface under acidic condition, and the increase of the aspect ratio of the nano-fibres was identical with the adsorption tendency of the anions (nitrate < chloride < sulfate).

Keywords: hydrothermal, boehmite, anions

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Using of Taguchi method for experimental design of crystallization processes of inorganic compounds

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Taguchi design of experiments (DOE) method has been used for to plan a minimum number of experiments and optimization of crystallization processes. This method decrease the number of experiment that need to received to good crystals. Taguchi method can be designed for a wide range of specific functions. Using a special orthogonal array only a small set from all the possible experiment is selected. The simultaneous variation of the main crystallization parameters and their interaction were investigated using orthogonal array techniques. The relative magnitude of the effect of different

factors can be obtained by decomposition of variance, called (analysis of variance) ANOVA. After developing some special criteria, which depend on performance objectives, the optimal levels of the design factors were determined.

Keywords: crystal growing, Taguchi method, inorganic compounds

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Hydrothermal synthesis of yttrium silicate based phosphors using new water soluble silicon compounds

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We developed a new stable water soluble silicon compound, which can provide up to 1M concentration of Si in water. It can be prepared by the reaction between tetraethoxysilan (TEOS) and propylene glycol. Analysis of its precise structure is currently under way. The potential of the water soluble silicon source for preparation of multicomponent oxide materials by solution based methods was investigated in synthesis of Y-Si based phosphors such as Ce³⁺ activated Y₂SiO₅(YSO:Ce) phosphor. YSO:Ce is a blue emission material used in the field emission displays. Synthesis of single phase compound by conventional solid-state reaction method is very difficult, and even a small amount of impurities significantly suppresses phosphor's emission intensity. In this work Hydrothermal Gelation Method in the combination with the prepared new water soluble silicon compound was used for synthesis of YSO:Ce. The method was previously developed by the authors, and it is based on the formation of the silicate gel network involving metal ions. For synthesis, aqueous stock solutions of soluble silicon compound, yttrium and cerium nitrates were mixed to obtain the required stoichiometry (Y:Si:Ce=1.98:1:0.02). The solution was put into hydrothermal reactor and kept at 200°C for 24 hours. The obtained colorless transparent gel was heat treated at 500°C and 800°C to remove organic compounds. Final annealing was carried out at 1400°C for 2 hours. The highest quality YSO:Ce materials up to date were obtained by the described method using new water soluble Si source. The intensity of the blue light emission from the obtained phosphor was three times higher compared to the samples synthesized by the similar method using TEOS.

Keywords: soluble silicon compounds, hydrothermal sol-gel method, phosphors

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Morphological control of meso- and single- crystals of Perovskite under solvothermal conditions

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The preparation, dielectric properties, optical properties and

temperature-dependent phase changes of CaTiO₃ have been attracted extensive research interest in last decades. However, CaTiO₃-based materials are in common fabricated by sol-gel chemistry and solid-state reactions at elevated temperatures and their morphologies are not favorable to be controlled. CaTiO₃, the nomenclature Perovskite named from, existing as a unique pseudocubic but orthorhombic symmetry, might be appearing as a variety of morphological derivatives in solvent. Herein, we will present the growth of meso- and single-crystals of Perovskite CaTiO₃ with tunable morphology by the assistant of organic molecules under solvo- and hydrothermal conditions. Several CaTiO₃ nanostructures, such as textured hollow mesocages built by self-assembled nanocrystals, mesoboxes with twinned or single-crystal nanowalls, as well as sub-microrods, butterfly-like nanodendrites and cross-linked nanotubes were synthesized under different growth conditions. According to the detailed structural analyses by X-ray and electron diffraction, cubic phase and orthorhombic {112} twined nanostructure were detected for hollow CaTiO₃ nanostructures obtained via a facile growth procedure. In addition, a "top to down" growth mechanism of the butterfly-like CaTiO₃ nanodendrites has been observed by varying experimental conditions. The growth of these CaTiO₃ crystals might shed new light on crystallographic structure-related morphology-evolution and open a new window to modify nanostructure-related physical and chemical performances. For example, unique Raman spectra were obtained from the as-prepared CaTiO₃ mesoboxes with twinned structures, which is obviously different from previous CaTiO₃ bulk materials.

Keywords: perovskite oxides, nanocrystals, hydrothermal mineralization

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Single crystal growth of nonlinear optical chalcone derivative

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A donor substituted chalcone derivative has been synthesized by Claisen-Schmidt condensation reaction. Single crystals of chalcone derivative have been grown by isothermal solution growth technique. The X-ray diffraction analysis indicated that the new chalcone derivative crystallizes in noncentrosymmetric space group and generates optical second harmonic of Nd: YAG laser operating at 1063nm. The relative second harmonic generation (SHG) efficiency of the donor substituted chalcone is determined to be eight times greater than that of urea. The ease of crystal growth, high SHG efficiency, good thermal stability and transparency down to 440nm makes it to be a promising material for blue light generation from current laser diodes.

Keywords: crystal growth from solution, X-ray diffraction, second harmonic generation

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In situ observation of the joint gel/impurity effect on protein crystal growth kinetics

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