

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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The duality of agarose gels as either impurity or impurity filter in protein crystal growth experiments has been discussed frequently. But, up today, we don't know if agarose gels operate as an impurity, as an impurity filter or both. Therefore a series of experiments have been performed using gelled solutions (low concentration agarose) and ungelled solutions to simultaneously study the effect of gels and impurities on the growth kinetics of biomacromolecule crystals. The growth of crystals from highly purified (99.99% purity) and commercial grade (98.5%) lysozyme was observed by Laser Confocal Microscopy combined with Differential Interference contrast Microscopy (LCM-DIM). Step velocities, 2D nucleation rates and normal growth rates were measured. These growth parameters were separately assessed for crystals growing from pure and commercial grade solutions. It was found that 2D nucleation rates are enhanced by the presence of gel fibers that act as heterogeneous nucleation sites. These results also show that the specific surface energy is similar for the gel fiber/crystal interface and for the gel fiber/solution interface. This is consistent with the observed incorporation of agarose fibers into the lysozyme crystal lattice and the small effect of gel fibers on step velocity for crystals growing from highly purified solution. The presence of agarose significantly modifies the step velocity in crystals growing from impure solutions, shifting these values closer to the velocities measured in purified solutions. This velocity increase corresponds to a 7 fold reduction in the concentration of adsorbed impurities at the crystal surface with respect to ungelled experiments and can be considered as direct evidence of the diffusive impurity filtering concept.

Keywords: protein crystal growth, agarose gel, impurity filtering

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Self-organized eutectic microstructures towards photonic crystals and metamaterials

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Eutectics are special materials which are both a MONOLITH and a MULTIPHASE MATERIAL.[1] They may find application in the field of photonic crystals and metamaterials.[2, 3] The eutectic microstructure can exhibit many geometrical forms. It can be regular-lamellar, regular-rod-like, irregular, complex regular, quasi-regular, broken-lamellar, spiral and globular. The most interesting from the point of view of photonic crystals would be the microstructures with regular shapes, i.e. lamellar and rod-like. For metamaterials applications the other shapes could be also of interest - for example the percolated structures (for giant dielectric constant); or the spiral one for chiral metamaterials. The general overview of the road of eutectics towards photonics as well as new experimental data will be presented.

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Keywords: eutectic crystalization, self-organization, photonic crystals and metamaterials

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Desktop Minstrel UVTM: A novel protein crystal monitoring automation system using UV fluorescence

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Identifying protein crystals in crystallization droplets has long been considered a challenging step in the field of protein crystallography. Although there are numerous automated crystallization robots readily available, none have been able to successfully monitor crystal growth by distinguishing protein crystals from non-protein crystals and detecting crystals from drops that are otherwise difficult to see with visible light. In order to fulfill this critical need, Rigaku has developed a novel protein crystal monitoring automation system, the Desktop Minstrel UVTM, which uses UV fluorescence microscopy. The system includes an ultraviolet microscope with at least one ultraviolet light emitting diode, providing illumination with the wavelength matching the absorption of the fluorescing amino acids, such as tryptophan. To greatly decrease photo-damage to the protein crystals, the fluorescing light illuminated on the sample is reduced to the minimum and is then digitally recorded by a camera with a CCD sensor. We have conducted crystallization experiments with various proteins in order to evaluate this system. The resulting UV images from these experiments clearly reveal the protein crystals from non-protein crystals, such as salts. In addition, this UV crystal monitoring system is built upon the platform of Rigaku's state-of-art imaging automation technology, the Desktop Minstrel, which makes the evaluation of a large number of crystallization experiments possible. The Desktop Minstrel UV enables researchers to accurately harvest protein crystals for data collection or design follow-up experiments.

Keywords: crystallization robots, imaging, UV fluorescence

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The microcapillary protein crystallization system

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The Microcapillary Protein Crystallization System (MPCS) embodies a new, semi-automated, microfluidic plug-based crystallization technology which allows researchers to conduct nanoliter-volume screening of crystallization conditions and to perform in-situ X-ray diffraction studies on crystals that form. The MPCS integrates formulation of crystallization cocktails with preparation of the crystallization experiment. Within microfluidic Teflon tubing or the microfluidic circuitry of a plastic CrystalCard, ca.10-20 nL volume droplets can be generated, each representing a traditional