computational investigation of the relationship between the resolution efficiency of ephedrine (E) as a resolving agent for 2-phenylpropionic (hydratropic) acid (PP) and the physical properties of the diastereomeric salt pairs E***R***PP and E***S***PP. The crystal structure of the least soluble salt (E***R***PP) and a polymorph of the most soluble salt (E***S***PP form IV) have been determined by low temperature single crystal X-ray diffraction and E***S***PP form II by powder X-ray diffraction (PXRD). Two further polymorphs and a hydrate of E***S***PP have been identified by infrared spectroscopy and PXRD. Differential scanning calorimetry with thermogravimetric analysis was used to determine the relative stability of the salt pairs and polymorphs. HPLC and solubility measurements have been used to measure the resolution efficiency of E***R***PP and the most stable polymorph (form I) of E***S***PP.

The experimental crystal structures reveal that the resolution efficiency is affected by the competition between intra- and intermolecular interactions, as in the most stable salt (E***R***PP), the ephedrine forms an intramolecular N-H···O interaction, in addition to having different intermolecular hydrogen bonding motif to both E***S***PP polymorphs. CrystalOptimizer [2] has been used to calculate the lattice energy, $E_{latt}=U_{inter}+\Delta E_{intra}$, the balance between the intramolecular energy penalty, ΔE_{intra}, for distortions of the ions within the crystal, and the intermolecular lattice energy, $U_{inter.}$ The experimental work on this system shows both the challenges to, and potential benefits of, computational assessment of resolving agents.

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Keywords: chirality, resolution, computation

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Microstructure evolution in MgF_2 and ZnO doped LAS Glass-Ceramics

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The microstructure evolution, including glassy phase separation, crystallization and phase transformation plays the most important role in practical mechanical property and application of lithium aluminosilicate (LAS) glass-ceramic materials. The main goal of this study is to demonstrate the correlations among phase development, microstructure, thermal expansion coefficient and mechanical strength associated with the content of MgF_2 and ZnO with commercial-like recipes. In this work, the result of flexural strength of LAS glass-ceramics strongly depends on the concentration of additives, MgF_2 and ZnO. When about 6% ZnO are added, the strength of a sample increases from about 62 MPa to 78 MPa. However, MgF_2 bearing samples accompany with a ~50% decreased in flexural strength. This is because the morphology, phase composition and transformation temperature are changed via doping. X-ray powder diffraction (XRD) results shows that the main phase follows the regular way, hexagonal β-quartz solid solution formed at lower temperature and transform to tetragonal β-spodumene solid solution at higher temperature. The transformation temperature of β-quartz solid solution to β-spodumene solid solution is lowered for several tens of degrees when the ZnO or MgF_2 is doped. It might be due to the weakening effect of silicate network by these additives. Also, various secondary phases, such as $ZrTiO_4$, and spinel of zinc and magnesium are usually precipitated at higher temperatures for samples with higher content of MgF_2. In addition, X-ray absorption near edge structure (XANES) feature is also used to certify the existence of some minor phases and the structural role of the doped elements. The significant change in XANES features of Zn K-edge indicates that Zn ions are usually involved in the crystallization process for samples doped with > 1% ZnO. Zn^{2+} ions might substitute Li^+ site in the main crystalline phase. This substitution phenomenon results in the formation of secondary phases. Thermal expansion mismatch from the microstructure non-uniformity is responsible for the strength drop of MgF_2 bearing samples.

Keywords: glass-ceramic, XANES, X-ray diffraction

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Evolution of the microstructure of bimetallic valves

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New our casting methods using heightened pressure of inert gas allow to obtain complex-form products from a variety of combination traditional and new high-temperature materials (G. Teterin at al. 1996). Distribution of the alloy components as well as phase composition across the valve section was obtained by means of energy-dispersive analysis and X-ray diffraction. The difference X-ray method has been used to investigate the evolution of the phase composition and microstructural characteristics of highly effective bimetallic engine valves. The head of the valves is made of Ti or Ti-Al, and the main rod is made of different materials. Bragg angle and the width of Bragg intensity profiles of reflection of high and low order for the phases were measured. The assessment of the change in the microstructure of the strain level at the predominant phase has been made on the basis of data of the angular dependence of the width of the Bragg reflection. As a reference standard of an instrument shape of reflections SRM 660 (LaB6) is used. Reliability and composition of the joining of head and stem was investigated by various method also. Hardness measurements were undertaken using 15-g, 50-g and 100-g loads using Micromet-2001 and PMT-3 testers. After casting the head of the valve it has a tetragonal gamma-phase and hexagonal alpha-phase; the ratio of the phases changes during the subsequent thermal treatment, and as a result the mechanical properties of the valve head become better. It should be noted that it is necessary to perform the control over the rod junction area because during some processing modes the martensitic beta-phase could appear, which can reduce the fracture toughness of the material and lead to a higher level of strain and as a result may appear some kind of micro-cracks in the junction of the valve. The exhaust valve were tested as a part of engine under the real-life condition of operation. It has been suggested the model described the processes

Keywords: strain-stress, phase composition, casting methods

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Analytical methodology for the quantification of respirable crystalline silica (RCS) in occupational environments using a CIP 10-R sampler

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