
Thermal properties and crystallization mechanism of undoped and Nd³⁺-doped calcium aluminosilicate glasses

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Abstract

In the preparation of glass-ceramics, with specific micro-structure and properties, the knowledge about nucleation rate and crystal growth is of great relevance, since can enable controlled crystallization. A well-established method that uses differential thermal analysis (DTA) to determine the nucleation-rate-like curves in glasses has been proposed by Ray and Day¹. In this regard, DTA measurements can be used as a rapid method for determining the temperature where the nucleation rate in a glass is a maximum. The scope of this work had two main strands. The first was focused on the role of crystallization mechanism in the synthesis of calcium aluminosilicate glasses doped with Nd³⁺. The second strand of research was the obtaining of transparent glass-ceramics through controlled crystallization of the precursor glasses. Accordingly, this study demonstrates the effect of composition on the crystallization kinetic of SiO₂-Al₂O₃-CaO-MgO-Nd₂O₃-doped glasses. Two matrices with quite distinct compositions and degree of polymerization were synthesized. The melting was performed under vacuum atmosphere at 1600C. In rare-earth doped glasses, this vacuum synthesis provides ions in reduced oxidation states and with minimal, or absent, presence of OH⁻ in their structures. These OH⁻-free glasses, prepared under vacuum atmosphere, present potential application as solid state lasers and white light (WL) generation devices. Differential thermal analysis was used to evaluate the crystallization kinetic parameters, such as the glass transition temperature (T_g) and the activity energy of glass transition (E_t) were also measured with different methods. Glass stability was evaluated by means of the characteristic temperatures of thermal events in the DTA measurements. Non-isothermal methods of Kissinger and Ozawa were used to obtain the apparent activation energy. The results showed that in this system surface crystallization was more favorable than bulk. By confocal Raman microscopy it was estimated that the crystallized region was 20-30 microns from surface to center of sample. The structural characterization, carried out by X-ray diffraction (XRD), revealed calcium aluminum oxide as the major phase.

Keywords: Transparent glass ceramics, rare earth, Crystallization kinetic parameters

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