Synthesis of a nano-crystalline lithium-mica glass-ceramic via sol-gel method

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Glass- ceramics are polycrystalline ceramic materials, derived through the controlled nucleation and crystallization of glass, where the content of residual glassy phase is usually less than 50%. In addition to the ease of flexibility of forming the glassy state, glass- ceramics have a uniformity of microstructure and reproducibility of glass [1-3]. Of the many types of obtainable microstructures in glass- ceramics, those based on uniformly dispersed crystals <100nm in size provide unique attributes for the current products and offer promise for many potential new applications [1].

Mica glass-ceramics are such typical machinable ceramics where the crystals of mica disperse within a glassy matrix. Apart from being machinable, the mica-based glass-ceramics exhibit heat resistance exceeding 800 °C, electrical insulating properties as well as high mechanical strength [2].

Recently Taruta et al. [3] successfully prepared a novel mica glass-ceramics where the separated micas are lithium-mica type with a mean crystallite size of 20 nm in which the interlayer cation is lithium ion. Thus, the novel mica glass-ceramics have potential applications not merely as mechinable ceramics and optical materiales but also as lithium ion conductors, used in many application fields.

In recent years, glasses prepared through a sol-gel route are found to have advantages over conventional melted method such as: good homogeneity, better purity and lower stoichiometric losses [4]. So far, no research has been reported in the area of lithium- mica sol- gel synthesis hence; the present investigation intends to prepare the nano-crystalline lithium-mica glass-ceramic applying the aqueous sol-gel process, to characterize the synthesized powders and compare it with the one provided by Taruta et. al. through the conventional melted method.

Experimental procedures of the present work can be highlighted as follows:

- Sample preparation: Fig. 1 shows the flowchart of the synthesis process. The dried gel was heated under different conditions to obtain glass-ceramic materials.
- Thermal analyses: Thermal traces such as decomposition of residual organic materials or nitrate groups and crystallization process were conducted by using DSC and TG methods.
- X-ray diffraction (XRD) analyses: X-ray diffraction was used for phase analysis of different samples. The average crystallite size of mica was estimated using the Scherrer formula on the base of XRD patterns.
- TEM investigation: The morphology and crystallite size of nano-crystalline mica were investigated by transmission electron microscopy (TEM) along with EDS spectra equipment for chemical analysis.
- Evaluation of crystallization kinetic: The kinetic factors such as activation energy for crystallization and Avrami parameter were estimated through DSC diagrams using different heating rates according to the equations proposed by Marotta et al.[5].

The obtained results can be summarized as follows:

- The mean crystallite size of mica was determined as 13nm, which is seen in the TEM micrograph (Fig. 2), is in good agreement with those of the calculated value by means of the Scherrer formula. This is confirmed that the average crystallite size of mica synthesized via sol-gel process is smaller than the one prepared by conventional melted method and reported by Taruta et. al.
- Crystallization of the mica is derived from MgF₂ at 675 °C while, at next stages, there will occur the transformations of mica to norbergite and norbergite to chondrodite.
- According to the Marotta formula, the activation energy for crystallization and Avrami parameter are measured at 376.7 kJ mol⁻¹ and 2.3, respectively. Based on this model it seems that the bulk crystallization along with the combination of a needle and flake like growths of the crystals is the predominant mechanism to describe the crystallization behavior of the sol-gel synthesized lithium-mica nano glass- ceramics.
References


Figures

Fig. 1. The flow chart of lithium-mica synthesis applied sol-gel technique.

Fig. 2. TEM photograph of (a) the specimen heat treated at 700 C and corresponding SAED pattern with the attached EDS spectra taken from the marked particle, (b) HRTEM image of mica nano-crystal.