

Controllable Modification of Glass-Crystal Composites with Ion-Exchange Technique

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Abstract : The presented research is related to the development of recently proposed technique of the formation of composite materials, like optical glass-ceramics, with predetermined structure and properties of the crystalline component. The technique is based on the control of the size and concentration of the crystalline grains using the phenomenon of glass-ceramics decrystallization (vitrification) induced by ion-exchange. This phenomenon was discovered and explained in the beginning of the 2000s, while related theoretical description was given in 2016 only. In general, the developed theory enables one to model the process and optimize the conditions of ion-exchange processing of glass-ceramics, which provide given properties of crystalline component, in particular, profile of the average size of the crystalline grains. The optimization is possible if one knows two dimensionless parameters of the theoretical model. One of them (β) is the value which is directly related to the solubility of crystalline component of the glass-ceramics in the glass matrix, and another (γ) is equal to the ratio of characteristic times of ion-exchange diffusion and crystalline grain dissolution. The presented study is dedicated to the development of experimental technique and simulation which allow determining these parameters. It is shown that these parameters can be deduced from the data on the space distributions of diffusant concentrations and average size of crystalline grains in the glass-ceramics samples subjected to ion-exchange treatment. Measurements at least at two temperatures and two processing times at each temperature are necessary. The composite material used was a silica-based glass-ceramics with crystalline grains of $\text{Li}_2\text{O}\cdot\text{SiO}_2$. Cubical samples of the glass-ceramics (6x6x6 mm³) underwent the ion exchange process in NaNO_3 salt melt at 520 oC (for 16 and 48 h), 540 oC (for 8 and 24 h), 560 oC (for 4 and 12 h), and 580 oC (for 2 and 8 h). The ion exchange processing resulted in the glass-ceramics vitrification in the subsurface layers where ion-exchange diffusion took place. Slabs about 1 mm thick were cut from the central part of the samples and their big facets were polished. These slabs were used to find profiles of diffusant concentrations and average size of the crystalline grains. The concentration profiles were determined from refractive index profiles measured with Max-Zender interferometer, and profiles of the average size of the crystalline grains were determined with micro-Raman spectroscopy. Numerical simulation were based on the developed theoretical model of the glass-ceramics decrystallization induced by ion exchange. The simulation of the processes was carried out for different values of β and γ parameters under all above-mentioned ion exchange conditions. As a result, the temperature dependences of the parameters, which provided a reliable coincidence of the simulation and experimental data, were found. This ensured the adequate modeling of the process of the glass-ceramics decrystallization in 520-580 oC temperature interval. Developed approach provides a powerful tool for fine tuning of the glass-ceramics structure, namely, concentration and average size of crystalline grains.

Keywords : diffusion, glass-ceramics, ion exchange, vitrification

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