Engineering Nanocrystalline Microstructures in GeS_2-Sb_2S_3-PbS Glasses

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Chalcogenide glasses (ChG) are known for their wide transmission range from λ=1-14 μm and for their high refractive index. However, applications for ChG are often limited by their poor thermal-mechanical properties including limited thermal shock resistance, low thermal conductivity, and modest thermo-optic coefficients. Nucleating and growing a secondary crystalline phase in the glass matrix can improve these properties, but too much crystallization and/or large or multiple phase crystallites can lead to a loss in transmission. Controlled crystallization in ChG can be quite difficult due to their low thermal conductivities. This leads to a temperature gradient across the glass in traditional furnace heat treatments, causing different rates of crystallization across the sample and leading to an uncontrolled, random microstructure. In order to control the nucleation and growth of crystals, it is necessary to fully characterize the nucleation and growth behavior of the defined glass-crystal system.

Two ChG compositions in the Sb_2S_3-GeS_2-PbS glass system, with GeS_2:Sb_2S_3 ratios of 17:3 and 11:4, were evaluated for their suitability in an application that required an optical glass-ceramic with uniform distribution of crystal nuclei in the glass matrix. Composition selection was aided by the mapped regions of crystallization stability in this glass system by Xia *et al.*[1] The glasses were characterized through Raman spectroscopy, thermal analysis, FTIR, UV-Vis spectroscopy, and microhardness. The 11:4 composition was found to be a better candidate because of its crystallization behavior and higher percent transmittance. After a down selection to the 11:4 composition, research focus shifted to creating nucleation-like and growth-like curves for the primary crystalline phase. The nucleation-like and growth-like curves were created for the primary crystal phase of the 11:4 glass-ceramic through a process adapted from the works of Marotta *et al.*[2] and Ray *et al*.[3] Points for the nucleation-like and growth-like curves were generated through DSC runs with specific isothermal holds to induce nucleation/growth at different rates. The completed nucleation-like and growth-like curves were used to identify appropriate heat treatments to nucleate and grow nanocrystals uniformly within the glass matrix. The methodology to define these material attributes and the resulting findings are discussed.

References

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