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$NaF-CaF_2-Al_2O_3-SiO_2$微晶玻璃的析晶动力学和显微组织

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摘 要： 采用传统熔体冷却法制备了掺铒$NaF-CaF_2-Al_2O_3-SiO_2$体系玻璃，并通过优化热处理工艺获得了透明氟氧化物微晶玻璃。通过DSC、XRD和TEM等方法研究了引入碱金属氧化物和碱土金属氧化物对玻璃形成能力的影响，采用动力学方法分析了$NaF-CaF_2-Al_2O_3-SiO_2$系统玻璃的析晶机制，探讨了热处理制度、玻璃析晶行为和显微结构的关系。研究表明：碱金属氧化物的引入降低了该系统玻璃的形成能力，而碱土金属氧化物的添加提高了玻璃的析晶稳定性；该系统玻璃的析晶过程主要受扩散控制，其主晶相为CaF_2，析晶活化能为345.8 kJ/mol，晶粒尺寸随晶化温度升高逐渐增大，晶粒数量随保温时间延长逐渐增多。通过优化热处理制度，获得了晶粒尺寸小于50 nm、结晶度约为30%的透明微晶玻璃。

关键字： 氟氧化物玻璃；析晶行为；热处理工艺；显微组织；透明微晶玻璃

Crystallization kinetics and microstructures of $NaF-CaF_2-Al_2O_3-SiO_2$ glass-ceramics

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Abstract: The Yb^{3+}-doped transparent oxyfluoride glass in $NaF-CaF_2-Al_2O_3-SiO_2$ system was prepared by conventional melt-quenching method and the transparent oxyfluoride glass-ceramics was obtained by optimized heat treating. The influences of introducing alkali metals oxides and alkaline earth oxides on glass-forming ability were investigated by differential thermal analysis (DTA), X-ray diffraction (XRD) analysis and transmission electron microscopy (TEM). The crystallization mechanism of $NaF-CaF_2-Al_2O_3-SiO_2$ system glass was analyzed by kinetics method, and the influences of heat treating conditions on the crystallization behavior and the microstructures of this system glass-ceramic were studied. The results show that the glass-forming ability is weakened with introducing alkali metals oxides, while the crystallization stability is improved by introducing alkaline earth oxides. In addition, the main crystal phase of the glass-ceramics is CaF_2 and its crystallization activation energy is 345.8 kJ/mol. The size of CaF_2 grains increases with increasing crystallization temperature and the amount of crystals increases with increasing holding time. A novel transparent Yb^{3+}-doped glass-ceramics is obtained by optimizing heat treating, in which the grain size is less than 50 nm and the crystallization degree is about 30%.

Key words: oxyfluoride glass; crystallization behavior; heat treating process; microstructure; transparent glass-ceramics

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