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Preparation and properties of transparent cordierite-based glass-ceramics with high crystallinity

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Abstract

Transparent cordierite-based glass-ceramics with high crystallinity were prepared successfully. The crystallization kinetics of parent glass was deeply investigated by differential scanning calorimetry (DSC). After heat-treatment at 1030 °C for 6 h, a large amount of α-cordierite (indialite) crystals devitrified from the parent glass, which was confirmed by the X-ray diffraction and TEM results. The crystallinity of the obtained transparent glass-ceramics reached up to 87.5%, which can be ascribed to the composition and heat treatment of parent glass. The high transparency of glass-ceramics mainly resulted from the small difference of refractive index between α-cordierite and the parent glass. In addition, these transparent glass-ceramics possessed low density (2.477 g/cm^3), low thermal expansion coefficient (1.435×10^{-6} °C^{-1}) and high Vickers hardness (8.1 GPa). The combination of excellent physical, thermal and optical properties makes this new family of transparent glass-ceramics exhibiting potential applications in the fields of base materials for rare-earth ions.
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1. Introduction

Transparent glass-ceramics can be prepared by means of the following conditions: their crystal sizes are much smaller than the wavelength of light or the difference of refractive index between crystal phase and matrix glass is very small in these glass-ceramics [1]. These conditions can be realized in several aluminosilicate glass systems and much attention has been paid to that in the last thirty years. Till now, a large amount of papers on transparent glass-ceramics have been published, and these transparent crystalline materials have been applied widely due to their excellent optical properties, high thermal and chemical stability, and high mechanical strength [2,3]. However, the majority of traditional transparent glass-ceramics contained small to moderate crystallized volume fraction, usually between 3% and 70%, and the content of glass was higher than 30%, which influenced their mechanical or thermal properties [4].

Cordierite ($2MgO \cdot 2Al_2O_3 \cdot 5SiO_2$) and cordierite-based glass-ceramics have been widely studied in recent years owing to their low thermal expansion coefficient (TEC), low density, low dielectric constant and high mechanical strength [5,6]. Many investigations on glass-ceramics of this system have been published, especially focusing on the effect of different nucleating agents (TiO_2, ZrO_2 or P_2O_5) and the heat treatment on their mechanical, thermal and electrical properties [7–10]. In addition, studies concerned with the nucleation sites and kinetics of cordierite-type glass have been reported by Zanotto [11].

Small amounts of additives in parent glass significantly influence the crystallization and properties of this glass-ceramic system. Hwang et al. [12] has studied the effect of composition on microstructural development in this system, and they concluded that compositions richer in SiO_2 than the stoichiometric composition of cordierite suppressed the formation of μ-cordierite, yet enhanced the crystallization of α-cordierite, resulting in a higher content of α-cordierite. B_2O_3 and P_2O_5 as additives added into cordierite glass has been studied extensively by Wu and Hwang [13], who believed that the addition of B_2O_3 promoted the formation of α-cordierite, either from crystallization of the residual

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glass or from transformation of μ-cordierite, whereas P_2O_5 had the opposite effect. Crystallization kinetics of cordierite-type glass containing B_2O_3 has been well studied by Watanabe et al., they suggested that the crystallization mechanism was believed to be a volume diffusion process governed by the thin boundary layer produced at the glass-crystal interface followed with surface nucleation [14]. However, relatively few fundamental studies have focused on the optical properties of cordierite-based glass-ceramics. It is pertinent to note that there is a small difference of refractive index between cordierite and some aluminosilicate glass [1]. Through designing the composition of parent glass appropriately, it is probable to prepare a new kind of transparent glass-ceramics with high crystallinity.

In this investigation, we aimed to develop a new type of transparent glass-ceramics with high crystallinity from MgO–Al_2O_3–SiO_2 system. Some of the characteristic properties of these transparent glass-ceramics were presented below together with an analysis of their crystallization process.

2. Experimental procedures

2.1. Preparation of materials

The parent glass with a composition of $20MgO–20Al_2O_3–55SiO_2–5B_2O_3$ (in mol%) was prepared from high purity MgO, Al_2O_3, SiO_2 and H_3BO_3 by conventional melt quenching method. The reason for choosing this composition is that it was close to the stoichiometric composition of cordierite, $2MgO \cdot 2Al_2O_3 \cdot 5SiO_2$. Proper batches of each powder were thoroughly mixed and then melted at 1580 °C for 2 h in a covered platinum crucible in an electric furnace. The homogenous melts were poured onto a preheated iron plate, and thereafter transferred into a muffle furnace and annealed at 680 °C for 2 h to remove the internal stress. The obtained clear and transparent glass was cut into desired dimension or grinded into fine powder in order to perform different measurements.

2.2. Analytical methods

A Differential Scanning Calorimetry (DSC, Netzsch 404PC, Germany) was used to determine the glass transition temperature (T_g) and crystallization temperature (T_c) of the parent glass under air atmosphere. Powdered samples (less than 50 μm) weighed 10–15 mg were placed in an alumina crucible to perform the DSC measurement. The DSC curves were obtained within the temperature range of 30–1100 °C at different heating rates of 5, 10, 15, 20 °C/min. And the measurement error was ± 2 °C. Besides, the kinetic study of crystallization mechanism was investigated by Johnson-Mehl-Avrami (JMA) equation.

The crystalline phase precipitated in the parent glass was investigated by an X-ray Diffractometer (XRD, D/max 2500 model, Rigaku, Japan). The diffractometer scanned the powder at a rate of 8°/min within Bragg angle (2θ) from 5° to 80°, which operated with working voltage and current of 40 kV and 50 mA. The crystalline phase was identified through matching the peak positions of the intense peaks with PCPDF standard cards. In addition, the crystallized volume fraction of glass-ceramics was calculated by Rietveld analysis of the diffractometer, within the error range of about ± 5%.

In this investigation, we used the transmission electron microscope (JEOL JEM 2100F, Japan) to characterize the crystalline microstructure of glass-ceramics. Prior to TEM, the glass-ceramic samples were ground (less than 100 nm) and then dissolved into the alcohol solution homogeneously. Appropriate amount of solution was dropped on the carbon film and subsequently dried under an infrared lamp. Each TEM micrograph has a scale, which is used to calculate the grain size by measuring the map range.

Transmittance spectra of the parent glass and glass-ceramics were measured with a HITACHI U-3310 UV spectrophotometer (Hitachi Ltd., Japan) during the wavelength range of 200–780 nm. The surfaces of glass-ceramics were polished (2000 mesh) and cleaned by Ultrasonic Bath with alcohol as the clearing agent. The refractive indexes of parent glass and glass-ceramics were studied through prism minimum deviation method by an Abbe refractometer (2 W, Shanghai) with accuracy of $n_D \pm 0.002$. Five measurements were made on each sample and averaged.

As the open porosity could be ignored, the bulk density was determined according to Archimedes principle with distilled water as the immersion medium at room temperature, $\rho = m_1\rho_0/(m_1 - m_2)$, where, m_1 was the weight of the sample in the air, m_2 was the weight of the sample in the distilled water, and ρ_0 was the density of the distilled water.

The thermal expansion coefficients of the parent glass and glass-ceramics with dimensions 5 mm × 5 mm × 20 mm were obtained by a thermo-mechanical analyzer (Netzsch DIL 402EP, Germany) within the temperature range 20–600 °C at a heating rate of 5 °C/min. And the final values were obtained by averaging the values among the temperature range of 20–600 °C.

A micro-hardness tester (DHV-1000-CCD, Beijing) with a pyramid shaped diamond indenter was used to carry out the Vickers hardness measurements, applying loads of 4.9 N for 10 s. At least ten different positions of each sample were tested to get the average value. Then the average value was transformed into GPa.

3. Results and discussions

3.1. Crystallization kinetics

Fig. 1(a) shows the DSC curve taken on the parent glass at a heating rate of 10 °C/min. The glass transition temperature (T_g) occurs at about 780 °C, and followed by a single exothermic peak which corresponds to the crystallization of the parent glass. The DSC curves with different heating rates are shown in Fig. 1(b), it is clear that the crystallization peak temperatures (T_p) of different heating rates are 983 °C, 1008 °C, 1023 °C and 1039 °C, respectively. Both of the crystallization peak temperature and the peak height increased

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