



The preparation of the lithium disilicate glass-ceramic with high translucency

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ARTICLE INFO

Article history:

Received 14 October 2016

Received in revised form 23 November 2016

Accepted 28 November 2016

Available online 6 December 2016

Keywords:

Lithium disilicate

Translucency

Two-stage heat treatment

Glass-ceramics

ABSTRACT

To develop a lithium disilicate glass-ceramic with high translucency, the effects of the single-stage and two-stage heat treatments on the crystalline phases and microstructure of the $\text{Li}_2\text{O}-\text{SiO}_2-\text{Al}_2\text{O}_3-\text{K}_2\text{O}-\text{P}_2\text{O}_5$ glass system were investigated. It was found that the nucleation of the two-stage treatment was more effective than that of the single-stage treatment, resulting in the smaller lithium disilicate crystals (referred as the LD hereafter). Besides, the size of the LD crystals increases with increasing the second heat treatment temperatures, so the second heat treatment temperature should be as lower as possible than the LD crystallization peak temperature on the premise that the LD is the main crystalline phase. Finally, the lithium disilicate glass-ceramic with the crystalline size of about 100 nm was developed, and the real in-line transmission (RIT) value was measured to reach 27.3% at the wavelength of 550 nm ($d = 2.0$ mm), which is much greater than that of the commercial lithium disilicate glass-ceramics.

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1. Introduction

Lithium disilicate glass-ceramics are polycrystalline materials consisting of lithium disilicate crystals and glass matrix, formed by an appropriate heat treatment process [1]. Up to the present, the glass-ceramics have been widely used in dental restorations [2], mainly for dental inlays, onlays, crowns and bridges [3].

As one of the promising materials for dental restorations, the investigation of its mechanical property has aroused much interest [4–7] and the commercial lithium disilicate glass-ceramics with the high-bending strength of 400 ± 40 MPa have been developed [8]. Besides the mechanical property, the aesthetic property, especially the translucent characteristic of the glass-ceramics, is another important property which needs to be concerned [9,10], as the translucency is one of the primary factors affecting the esthetics and is critical for dental restorations. Dental restorations require different translucency for different applications, resulting in more translucency requirements for the lithium disilicate glass-ceramics [11]. It has been reported that the transmittance for the commercial lithium disilicate glass-ceramic is relatively low [8,12], no more than 10% ($\lambda = 550$ nm, $d = 0.8$ mm). McLaren et al. [11] reported that they would not use the lithium disilicate crowns

as the anterior teeth because of its relatively poor translucency. It is therefore of great significance to improve the transparency of the lithium disilicate glass-ceramics.

The translucency of glass-ceramics largely depends on light scattering [13], which is affected by the relative refractive index difference and the crystalline size [9,14]. For the lithium disilicate glass-ceramics, the size of the lithium disilicate crystals (referred as LD hereafter) can be adjusted to tune the translucency. According to Heffernan et al. [9], the materials composed of small crystals (~ 100 nm) are usually more translucent than those of large crystals. While, the crystal size in the commercial lithium disilicate glass-ceramic is about $0.5\text{--}4$ μm [9], leading to the relatively low translucency [15–17]. Therefore, adjusting the size of the LD crystals has drawn much attention for the lithium disilicate glass-ceramics [17–19]. Wang et al. [17] investigated the influence of the P_2O_5 nucleating agent on the size of LD crystals in a $\text{SiO}_2-\text{Li}_2\text{O}-\text{K}_2\text{O}-\text{Al}_2\text{O}_3-\text{ZrO}_2-\text{P}_2\text{O}_5$ glass system. When the P_2O_5 content is 2.0 mol%, the size of LD crystals is 200–400 nm. Zhang et al. [14] studied the effect of heat treatment procedure on the size of LD crystals. They achieved the glass-ceramic with the crystal size of 400 nm after 650 °C for 72 h and 830 °C for 3 h. Although much effort has been made to tailor LD crystalline size, lithium disilicate glass-ceramic with the LD crystalline size of ~ 100 nm has not been attainable yet. It is therefore of great significance to further reduce the LD crystalline size to improve the translucency of the lithium disilicate glass-ceramics and enlarge their application in dental restorations as well.

In the present investigation, the crystallization behavior, microstructure and translucency of the lithium disilicate glass-ceramics were

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characterized in the $\text{Li}_2\text{O}-\text{SiO}_2-\text{Al}_2\text{O}_3-\text{K}_2\text{O}-\text{P}_2\text{O}_5$ glass system. Based on the results, the effects of single-stage and two-stage heat treatments on the nucleation and crystallization mechanism of the LD crystals were carefully investigated. Finally, the disilicate glass-ceramic with superior translucency was developed.

2. Experimental procedures

2.1. Glass preparation

The investigated lithium disilicate glass was prepared using analytic grade Li_2CO_3 , SiO_2 , Al_2O_3 , K_2CO_3 and $(\text{NH}_4)_2\text{HPO}_4$, which were weighed by a balance with an accuracy of ± 0.1 g. Table 1 summarizes the content of each component. Homogeneous mixture of powder was melted in air at 1400°C for 2 h and the melt was quenched into water to obtain a glass frit. The received glass frit was milled for a few minutes and sieved to obtain the particles less than $74\ \mu\text{m}$.

2.2. Samples preparation and analyses

Differential scanning calorimetry (DSC, Linseis, STA PT-1600, Germany) was used to investigate the crystallization temperatures for the lithium metasilicate crystals (referred as LMS hereafter) and the lithium disilicate crystals, with the nominal accuracy of $\pm 0.05^\circ\text{C}$ for temperature. The parent glass powder was heated from room temperature to 1200°C with a heating rate of $10^\circ\text{C}\ \text{min}^{-1}$ in the static air.

The received glass powders were remelted at 1200°C for 2 h and cooled to room temperature to obtain the glass blocks. Then the single-stage and two-stage heat treatments were respectively performed according to the designed schedules. Four temperatures around the crystallization temperature of the LD crystal were selected in the single-stage treatment. Compared with the single-stage treatment, an additional heat treatment at 588°C for 30 min was added for the two-stage heat treatment. The detailed heat treatment schedules were described in Table 2 and Table 3. The sample only annealed at 588°C for 30 min (referred as G_{2-0} hereafter) was also prepared for comparison.

After the heat treatments at an Electrical Resistance furnace (YFFX15/160-CC, China, $\pm 3^\circ\text{C}$), the crystalline phases were determined by X-ray diffraction analysis (XRD, PANalytical, X'PERT-PRO MPD, Netherlands), using the copper $K_{\alpha 1}$ radiation ($\lambda = 0.154059\ \text{nm}$) at 40 kV and 40 mA and the samples being scanned from 10° to 90° with a 2θ -step of $0.033^\circ/\text{s}$. And then the microstructures were characterized on polished surface of all the samples by scanning electron microscopy (SEM, Semicon, JSM-7001F, Japan).

The ultraviolet visible spectrum spectrometer (UV, Hitachi, U-3010, Japan) was applied to measure the translucent values, which were expressed by real in-line transmission (RIT) [14]. The instrument has an accuracy of $\pm 0.3\ \text{nm}$ for wavelength and $\pm 0.4\%$ for RIT values. The resultant disks were 17 mm in diameter and about 2 mm in thickness. The RIT was measured in the wavelength range of 380 nm–780 nm.

3. Results

3.1. Heat treatment process

To obtain a lithium disilicate glass-ceramic with high translucency, it is important to control the size of the as-precipitated LD crystals through a proper heat treatment. The heat treatment temperatures can generally be determined by the DSC characterization.

Table 1
The composition of the parent glass (mol %).

Composition	Li_2O	SiO_2	Al_2O_3	K_2O	P_2O_5
Content (mol %)	26.6	66.4	3	2	2

Table 2
Single-stage heat treatment schedule.

Samples	Heat treatment	
	Temperature/ $^\circ\text{C}$	Time/min
G_{1-1}	750	30
G_{1-2}	800	30
G_{1-3}	850	30
G_{1-4}	900	30

3.1.1. DSC measurement

Fig. 1 shows the DSC trace of the investigated glass. The glass transition temperature and melting temperature are about 448°C and 967°C respectively. Two exothermic crystallization peaks at 588°C and 814°C are respectively associated to the formation of LMS and LD [14].

3.1.2. XRD results

Fig. 2 presents the XRD patterns of the specimens after the single-stage heat treatment in Table 2. As shown in Fig. 2, the temperature has a significant effect on the crystallization behavior. Single phase LMS precipitated when the temperature was 750°C . As the temperature increased to 800°C , LMS completely disappeared and LD formed. Besides, the SiO_2 phase appeared in the samples when the temperature exceeded 800°C . When the temperature exceeded 850°C , the crystalline Li_3PO_4 as a minor phase formed.

After annealed at 588°C for 30 min (G_{2-0}), only the diffraction peak of LMS could be detected, indicated in Fig. 3(a). Fig. 3(b) shows the XRD patterns of the samples after the two-stage heat treatment in Table 3. When the second stage treatment temperature was 750°C , the LMS and LD both formed with the LMS being the dominant one. When the temperature exceeded 800°C , the LMS peaks disappeared and the LD became the primary phase. Besides, the SiO_2 peaks emerged when the temperature exceeded 800°C . The Li_3PO_4 phase appeared when the temperature exceeded 850°C .

The results show that the single-stage treatment and two-stage treatment have no significant effect on the finally obtained crystalline phase. Besides, the LMS crystal acts as the dominating phase at the lower temperature, but the LD crystal becomes the main phase when the temperature exceeds 800°C . The XRD results also confirm the validity of the prediction of the DSC measurement.

3.1.3. Microstructure features

The microstructures of the glass-ceramics after the single-stage heat treatment at different temperatures are presented in Fig. 4. Fig. 4 (a) shows the crystalline LMS dendrites [20]. The rod-shaped LD crystals are observed (Fig. 4(b-d)) in the other samples. And with increasing the heat treatment temperatures, the length of the LD crystals increases from about $0.6\ \mu\text{m}$ to about $1.5\ \mu\text{m}$.

For the sample treated at 588°C (G_{2-0}), only the diffraction peak of LMS could be detected. Because the LMS crystals have high solubility in HF solution [21] and very small size in the sample G_{2-0} , it is very difficult to analyze their microstructures under the secondary electron mode or the electron backscatter mode. G_{2-1} is similar to G_{2-0} , so the SEM photographs of G_{2-0} and G_{2-1} are not provided in this paper. For the specimens G_{2-2} , the morphology of the LD crystals is difficult to

Table 3
Two-stage heat treatment schedules.

Samples	Heat treatment			
	Temperature/ $^\circ\text{C}$	Time/min	Temperature/ $^\circ\text{C}$	Time/min
G_{2-1}	588	30	750	30
G_{2-2}	588	30	800	30
G_{2-3}	588	30	850	30
G_{2-4}	588	30	900	30

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