

Microstructure and mechanical properties of zirconia-toughened lithium disilicate glass–ceramic composites

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HIGHLIGHTS

- The influence of zirconia on microstructure and properties was studied.
- The stress relationships between glass and crystalline phases were analyzed.
- Mechanical properties of composite with 3Y-TZP (15 wt%) increased significantly.
- The main reinforcing and toughening mechanisms were discussed in detail.

ARTICLE INFO

Article history:

Received 8 January 2013

Received in revised form

16 October 2013

Accepted 19 October 2013

Keywords:

Glasses

Ceramics

Microstructure

Mechanical properties

ABSTRACT

The lithium disilicate glass–ceramics composites reinforced and toughened by tetragonal zirconia (3Y-TZP) were prepared by hot-pressing at 800 °C with varying zirconia content from 0 to 30 wt.%. In the case of the composites of small zirconia content (below 10 wt.%), zirconia acted as nucleation agent primarily, and the microstructure was refined continuously. The morphology of $\text{Li}_2\text{Si}_2\text{O}_5$ crystals transformed from rod-shaped to spherical structure, and the mechanical properties decreased inevitably. For the composites of large zirconia content (from 15 wt.% to 30 wt.%), however, zirconia restrained the phase separation of glass. The morphology of $\text{Li}_2\text{Si}_2\text{O}_5$ crystals transformed to rod-shaped structure again. The mechanical properties of the composite at zirconia content of 15 wt.% increased up to 340 MPa and $3.5 \text{ MPa m}^{1/2}$ which were much higher than those of zirconia-free glass–ceramics. The improved properties were attributed mainly to compressive stress reinforcement, phase transformation and bridging toughening mechanisms.

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1. Introduction

Obvious considerations in evaluating dental ceramics include esthetics, mechanical performance, economic feasibility and utilization. All-ceramic dental materials are acknowledged for their biocompatibility and esthetic features. However, it has been claimed that these materials also exhibit a number of disadvantages, such as their inability to accommodate tensile forces by plastic deformation, and to disseminate such stressed in the vicinity of the cracks or defects. Efforts to improve the life time and to extent their application fields of all-ceramic materials have been directed toward improving their strength and fracture toughness [1]. Lithium disilicate glass–ceramics is one of the most

representative dental restoration systems because of the better mechanical properties and excellent esthetic features [2–4]. The trade name is IPS Empress 2 (Ivoclar Ltd.) which was developed in 1998. Although lithium disilicate glass–ceramics have many outstanding advantages, they cannot meet the demands of posterior dental crown and three-unit fixed bridges which must subject to higher external pressure. Similarly to other all-ceramic system, microcracks or flaws can be generated during the laboratory phase of fabrication and these flaws can later lead to clinical failure. As a result, it is necessary to improve the mechanical properties of lithium disilicate glass–ceramics.

Several methods have been successfully employed to strengthen and toughen glass–ceramics such as the formation of surface compressive stresses through ion exchange [5], fiber reinforcement using both brittle [6] and ductile fibers [7], and the addition of Ti particles to bioactive glass–ceramics [8]. Zirconia-reinforced ceramics have also been extensively investigated to improve the mechanical properties of alumina, mullite, Si_3N_4 , dental porcelain

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and glass–ceramics [9–14]. It is in 1980s that the potential for ZrO₂ toughening of glass and glass–ceramics has been explored [15–18]. In this case, some systems have been studied such as MgO–Al₂O₃–SiO₂ (cordierite) [19], Na₂O–Al₂O₃–SiO₂ [16], Li₂O–Al₂O₃–SiO₂ [20], Li₂O–SiO₂ [21] and bioactive glass–ceramics [22–28]. These glass–ceramics were fabricated by melting method and pressureless sintering technique, and so on. The microstructure, crystallization process and mechanical properties were investigated in the above reports. Nevertheless, few works in the literature devoted to zirconia-reinforced glass–ceramics composites have been conducted on lithium disilicate glass–ceramics [29].

With regard to the preparing technology of glass–ceramics by incorporation of ZrO₂, as we known, the content of zirconia is very limited by melting process. Apel et al. [22], investigated the ZrO₂-containing lithium disilicate glass–ceramics by melting method. Zirconia influenced the crystallization by hampering crystal growth and the resulting biaxial strength decreased comparing with ZrO₂-free glass–ceramics. In the case of pressureless sintering, the densification of glass–ceramics can be restrained by crystallization inevitably during sintering. Therefore, in this study, the zirconia-toughened lithium disilicate glass–ceramics composites designed by ourselves with a variable weight fraction of zirconia were prepared by hot-pressing technology in vacuum. The aim of this study is to improve the mechanical properties of lithium disilicate glass–ceramics. Furthermore, the interrelations between porosity, crystallinity and microstructure with mechanical properties of the composites were discussed. Meanwhile, the mainly reinforcing and toughening mechanisms were analyzed in detail.

2. Experimental

The lithium disilicate base glass with a composition of 31 mol% Li₂O, 62 mol% SiO₂, 2 mol% ZnO, 3 mol% K₂O, 1 mol% CaO and 1 mol% P₂O₅ was prepared from well-mixed powder containing appropriate amount of Li₂CO₃, SiO₂, ZnO, KNO₃, CaCO₃ and NH₄H₂PO₄ as raw materials [30]. K₂O, CaO and ZnO were added in order to reduce the melting temperature and to increase the chemical stability of the glass. Beside above oxides, P₂O₅ was added as the nucleation agent. A batch of 150 g powders were melt in a high-alumina crucible in an electric heated furnace at 1400 °C for 2 h in air. The melt was poured into deionized water and the glass slags were comminuted by agate balls for 24 h. The glass powder was mixed with different weight fraction of 3Y-TZP (3 mol% yttria-stabilized zirconia) by ball milling with ethanol for 8 h, and then dried at about 70 °C. The blended powders whose average powders dimension was about 200 μm were put into stainless die and hot-pressed at 800 °C with 30 MPa for 1 h in vacuum, and then furnace was allowed to cool. The contents of zirconia were 0, 5 wt.%, 10 wt.%, 15 wt.% and 30 wt.%, respectively. Hence, as Table 1 showed, the composites were denoted as G1P, G1P5Z, G1P10Z, G1P15Z, G1P20Z and G1P30Z.

The crystalline phase, microstructure and mechanical properties of the composites with different zirconia contents were analyzed by XRD, SEM and mechanical properties tests. XRD analysis of the hot pressed composites was carried out on HITACHI D/max-γB. The block specimens were detected in order to prevent transformation of tetragonal zirconia by comminuting. The 2-theta step increment

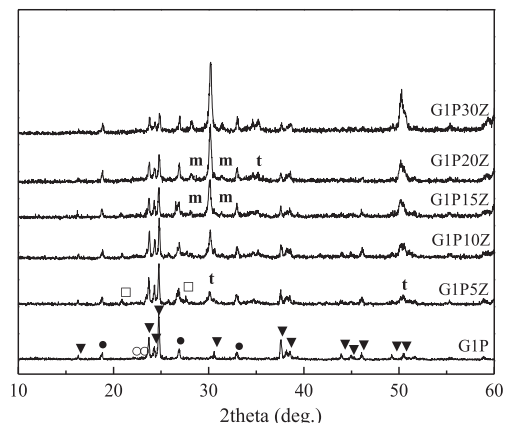


Fig. 1. XRD patterns of glass–ceramics with different content of zirconia hot-pressed at 800 °C for 1 h in vacuum ▼ Li₂Si₂O₅ ● Li₂SiO₃ ○ Li₃PO₄ □ SiO₂(Quartz) ▲ t-ZrO₂ ● m-ZrO₂.

was 10° with interval time of 2 s per step in the range of 10°–60° (λ Cu Kα = 1.5406 Å). The porosity was determined by metallographic image analysis system (VNT QuantLab-MG) combined with an OLYMPUS microscope. The ratio of the total areas of pores to the total selected area on the polished specimen surface was defined as the porosity. The crystallinity of glass is estimated by Eq. (1), which can be calculated by the diffraction peaks location, number and relative intensity according to XRD pattern.

$$X_c = \frac{\sum_i I_{ci}}{\sum_i I_{ci} + K I_a} \times 100\% \quad (1)$$

Where X_c is the crystallinity, I_a is the integrated intensity of non-crystalline phase, I_c is the integrated intensity of crystalline phase precipitated from glass phase, and K is a constant related to the measurement condition and glass compositions. According to the previous experimental results, $K = 0.963$.

The morphology of the crystalline phases developed after hot-pressing sintering was investigated using SEM (HITACHI S-4700). Polished specimens were etched with approximately 2 vol.% HF solution for 60 s. The morphologies of Li₂Si₂O₅ crystals and zirconia particles were exposed because Li₂SiO₃ and Li₃PO₄ crystalline phases were dissolved in HF solution.

The porosity was determined by an OLYMPUS microscope with the software of metallographic image analysis system (VNT QuantLab-MG). The magnification was 50X. The ratio of the total areas of pores to the total selected area on the polished specimen surface was defined as the porosity. The polished surfaces were observed along vertical direction. 3–5 polished specimens were tested in order to measure the porosity.

For the determination of the flexure strength and fracture toughness of the final hot-pressing composites, groups of bar specimens (at least 5 specimens) were tested by Instron-1186 universal testing machine. Thus, according to GB/T 4741-1999, the sections of 3 × 4 × 36 mm were cut from the specimen using a diamond saw for 3-point bending at room temperature with a span length of 30 mm and a crosshead speed of 0.5 mm min^{−1}. The five specimens of 2 × 4 × 20 mm with 2 mm open depth which were prepared by diamond grinding wheel were used to measure the fracture toughness K_{IC} at a crosshead speed of 0.05 mm min^{−1} by single edge notched beam (SENB) testing. Before testing, they initially polished using SiC sandwich of 1200#, 1500# and 2000# as abrasive, and afterward with polishing sandpaper and polishing

Table 1
Constituents of zirconia/lithium disilicate glass–ceramics composites.

Constituent (wt.%)	G1P	G1P5Z	G1P10Z	G1P15Z	G1P20Z	G1P30Z
Lithium disilicate base glass	100	95	90	85	80	70
3Y-TZP	0	5	10	15	20	30

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