

# Sintering and reactive crystal growth of diopside–albite glass–ceramics from waste glass

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## Abstract

Diopside–albite glass–ceramics were fabricated by sintering the powder mixtures of crystallization promoters and waste glass. Two kinds of promoters were synthesized using kaolin clay, talc and chemical reagents. The crystalline phases were formed by a reactive crystallization between promoters and glass during sintering. The effect of promoter components, additions and sintering temperatures on the crystallizing and densifying behavior, microstructures and mechanical properties of glass–ceramics was investigated. The results showed that the higher densities and better mechanical properties were obtained for the glass–ceramics with 12–15% crystallization promoters sintered at 950 °C for 2 h.

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**Keywords:** Crystallization promoter; Reactive crystallization; Diopside–albite glass–ceramics; Waste glass

## 1. Introduction

Pyroxene glass–ceramics based on the CaO–MgO–Al<sub>2</sub>O<sub>3</sub>–SiO<sub>2</sub> or Na<sub>2</sub>O–CaO–MgO–Al<sub>2</sub>O<sub>3</sub>–SiO<sub>2</sub> systems have considerable mechanical properties and chemical durability and are attractive materials for construction and architectural applications, such as floor- or wall-tile materials, to replace the natural marble and granite.<sup>1–4</sup> In addition, they can also be applied to the field of abrasion-resistant materials, *i.e.*, industrial floor coverings, wall facings, abrasion-resistant linings, and high-temperature insulators.<sup>5,6</sup> Usually, the raw materials used in the production of glass–ceramics are the glass industry raw materials, such as clay, quartz sand, dolomite, limestone, feldspar, calcite, natural diopside mineral and other raw materials.<sup>2,7–9</sup> In the recent decades, large amount of industrial and domestic wastes, including various fly ashes generated from coal- and oil-fired electric power stations, or from urban solid waste and

sewage sludge incinerators, kaolin clay refining waste (Kira), granite waste, slags from steel making, etc.<sup>6,10–12</sup> have been successfully applied to the preparation of glass–ceramics for the sake of environment protection and saving resource. However, it is not a very economical industrial process due to the consumption of large amounts of thermal energy during the vitrification of these wastes.

On the other hand, large amounts of waste glasses cannot be directly utilized in the preparation of glass–ceramics because of their compositional limitation and low crystallizing tendency. At present, a general way of utilization of waste glasses in the area is to remelt it together with other components into a particular formulated base glass with high crystallization tendency, and then the base glass was converted into glass–ceramics by controlled crystallization or sinter-crystallization.<sup>3,5,13–17</sup> Obviously, this route is also a high energy consuming technique. Therefore, it is more attractive to directly recycle waste glasses into glass–ceramics without remelting, as have been reported in a few of works,<sup>18–20</sup> such as the machinable fluoramphibole glass–ceramics by sintering the powder mixtures of soda-lime glass and fluormica and the β-wollastonite glass–ceramics by sintering the powder mixtures of automobile waste glass and waste shell. In this study, a possibility was attempted to prepare diopside–albite glass–ceramics by directly adding some

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Table 1  
Compositions of kaolin and talc (wt%).

Compositions	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	CaO	MgO	K <sub>2</sub> O	Fe <sub>2</sub> O <sub>3</sub>	L.O.I
Kaolin clay	47	38.3	0.47	0.14	0.7	0.39	13
Talc	62.6	1.6	0.35	30.5	0.25	–	4.7

crystallization promoters into waste glass powder and then sintering. The effects of compositions of crystallization promoters, their additions and processing parameters on the reactive crystallizing behavior, sintering behavior and mechanical properties of diopside–albite glass–ceramics were investigated.

## 2. Experimental procedure

### 2.1. Synthesis of crystallization promoters

Kaolin (Jinan Jiyuan Corporation, China), talc (Dalian Gongrong Corporation, China) and chemical reagents were used as starting materials for the synthesis of crystallization promoters, the compositions of kaolin and talc are shown in Table 1. Two types of crystallization promoters were synthesized: promoter D1 consisting of (wt%) 54 kaolin and 46 talc and promoter D2 consisting of 44 kaolin, 36 talc, 10 BaCO<sub>3</sub>, and 10 ZnO, respectively. Some water was added to the homogenized raw materials to make a green body, dried at room temperature and fired at 1100 °C for 2 h. The obtained body of crystallization promoters was crushed, ball-milled in a ceramic jar mill with alumina media and sieved to powders <100 μm.

### 2.2. Preparation of glass–ceramics

Recycled window glass was used as the base glass whose composition was (wt%) 72.0 SiO<sub>2</sub>, 8.2 CaO, 14.3 Na<sub>2</sub>O, 1.3 Al<sub>2</sub>O<sub>3</sub>, 3.5 MgO, and 0.3 K<sub>2</sub>O. The glass was cleaned, crushed, ball-milled and sieved to powders <150 μm.

Crystallization promoters were added to the glass powder, the proportions of promoters were tabulated in Table 2. The powder mixtures were homogenized in a ball-mill for 4 h, then a 6 wt% PVA water solution as a binder was added to the mixtures, and finally were uniaxially pressed into cylindrical compacts in a hardened steel die at 30 MPa. After heating at 400 °C for 2 h to remove the binder, the compacts were heated up to the temperature range of 900–1000 °C at a heating rate of 4 °C/min for 2 h and allowed to cool inside the furnace to obtain the final glass–ceramics.

Table 2  
Components of glass–ceramics (wt%).

Sample code	Components
GD1-10	10D1 + 90glass
GD1-15	15D1 + 85glass
GD1-20	20D1 + 80glass
GD2-10	10D2 + 90glass
GD2-12	12D2 + 88glass
GD2-15	15D2 + 85glass
GD2-20	20D2 + 80glass

### 2.3. Characterization

The bulk densities of the glass–ceramics were measured by the Archimedes method and three samples were estimated to get the mean value. A Vickers hardness tester (HA-10A) was used to measure hardness, the load was 2 kg and loading time was 30 s. Each value is the mean value of five measurements at each sample. Compression strength and three-point bending strength were measured by using cylindrical samples (15 mm diameter × 14 mm height) and rectangular bars (5 mm × 6 mm × 40 mm), respectively, in an Instron-type apparatus with a crosshead speed of 0.02 mm/s, and five samples were measured to get the mean value for compressive strength and six bars for bending strength.

Scanning electron microscope (JSM-6360) was employed for microstructure observations of the glass–ceramics at fracture surfaces coated with thin film of gold and energy dispersive spectroscopy (Oxford-INCA) was employed for determination of chemical compositions of the crystals. The powders (<40 μm) were applied for phase identification via X-ray diffraction (XRD) using a diffractometer (Philips-PW1710) with CuKα radiation under the operating conditions of 40 kV and 30 mA, and a sampling interval of 0.02° 2θ.

## 3. Results and discussion

### 3.1. XRD of crystallization promoters

Crystallization promoter D1 consisted of enstatite (MgSiO<sub>3</sub>, JCPDS, 19-768), mullite (3Al<sub>2</sub>O<sub>3</sub> 2SiO<sub>2</sub>, JCPDS, 15-0776) and quartz (SiO<sub>2</sub>, JCPDS, 65-0466) (Fig. 1a), enstatite and mullite are the decomposition products of talc and kaolin at high temperature, respectively,<sup>21,22</sup> and quartz may be a residue of clay,<sup>23</sup> indicating that almost no reaction between talc and kaolin occurs during firing. However, promoter D2 contains enstatite and cordierite (2MgO 2Al<sub>2</sub>O<sub>3</sub> 5SiO<sub>2</sub>, JCPDS, 02-0646), which suggests that the addition of BaCO<sub>3</sub> and ZnO facilitates a reaction between talc and kaolin to form cordierite and hinders the formation of mullite, as shown in Fig. 1b.

### 3.2. Reactive crystallization in glass–ceramics

Two crystalline phases, *i.e.*, aluminum-containing diopside (Ca(Mg,Al)(Si,Al)<sub>2</sub>O<sub>6</sub>, JCPDS, 41-1370) as the major phase and albite (NaAlSi<sub>3</sub>O<sub>8</sub>, JCPDS, 19-1184) as the minor phase, were developed but the crystallization promoter D1 disappeared completely in the sintered glass–ceramic GD1 series (Fig. 2), so that a reaction between promoter D1 and the glass occurred to form both of the new phases. The intensity of diopside and albite diffraction peaks in GD1-20 increased slightly as being compared with GD1-15, meaning that the crystallinity increased with promoter amount and sintering temperature. Typical morphologies of these crystals are shown in Fig. 3, diopside crystals were rod-like and albite ones were granular. The EDS analysis demonstrates that both diopside and albite contained Ca and Na elements (Fig. 4), the Na content of albite was more than one of diopside, whereas the Ca content was just the reverse. These

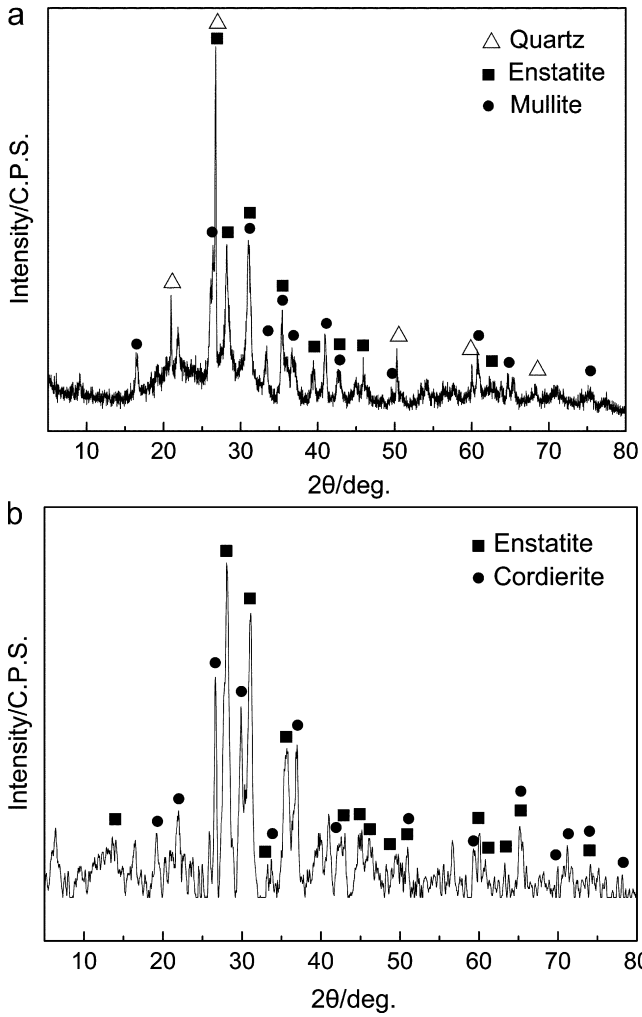


Fig. 1. XRD patterns of crystallization promoters, (a) D1, (b) D2.

elements diffused from the glass to the promoter because the promoter D1 contained no Ca and Na. The same results occurred in the glass–ceramics GD1-10 and GD1-20 too.

According to Fig. 5, the crystallization promoter D2 could also react with the glass to form diopside and albite in the sin-

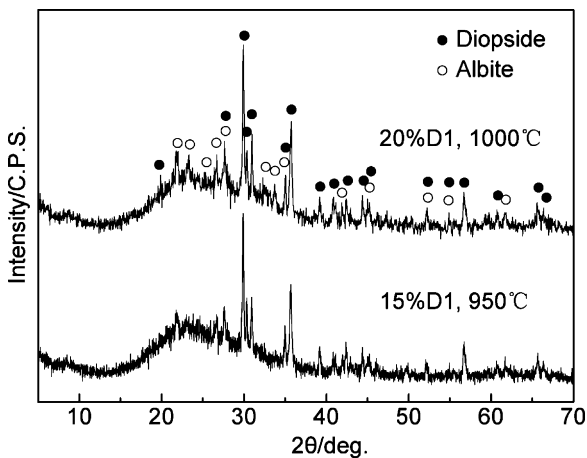


Fig. 2. XRD patterns of glass–ceramics with different D1 contents sintered at different temperatures.

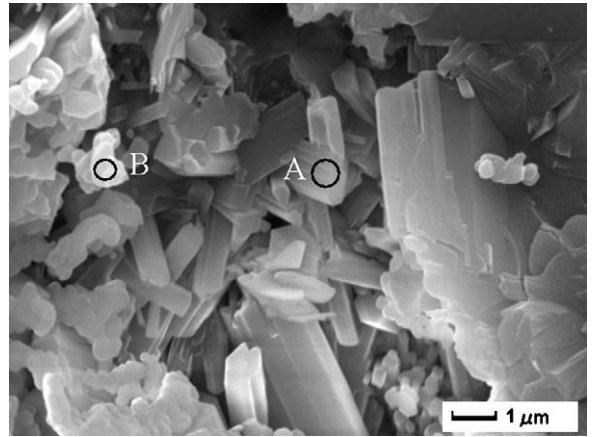


Fig. 3. SEM micrograph of glass–ceramic GD1-15 sintered at 950 °C.

tered glass–ceramic GD2 series. Unlike GD1-15, the albite was the major crystalline phase together with the minor diopside in GD2-12. However, the diopside became the majority in GD2-20, and its crystallinity increased markedly with D2 amount and

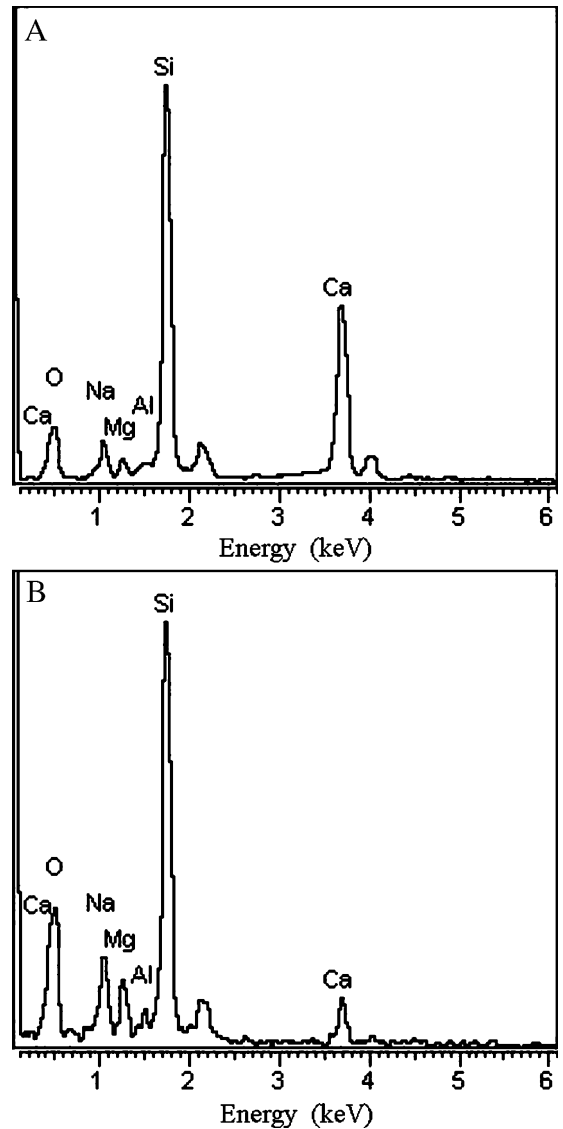


Fig. 4. EDS spectra of selected zones in Fig. 3: (a) diopside, (b) albite.

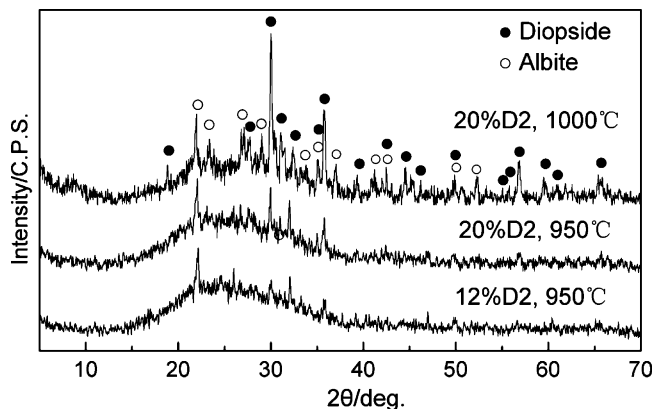


Fig. 5. XRD patterns of glass–ceramics with different D2 contents sintered at different temperatures.

sintered temperature. It should be noted that these crystalline phases in the glass–ceramic GD2-20 had Ba and Zn elements besides Ca and Na (Fig. 6), which differed from ones in the glass–ceramic GD1-15 because the promoter D2 contained Ba and Zn, indicating again that both diopside and albite were the products of reaction between the promoters and the glass and the diffusion of Ca and Na from glass to the promoters truly occurred during sintering.

While the preparation of glass–ceramics by sinter-crystallization, no crystalline phases existed in the base glass before sintering, but the glass itself was prone to devitrification, and the crystalline phases were developed directly from the base glass during sintering. In contrast, while the preparation of glass–ceramics by reactive crystallization, the crystallization promoters (crystals) had been already added to the soda-lime glass before sintering, but the glass itself was less prone to devitrification, since no crystalline phases were formed in the pure soda-lime glass powder compacts after sintering.<sup>19</sup> Obviously, diopside and albite cannot be developed directly from the soda-lime glass, they were only formed via the reaction between the promoters and the glass during sintering.

The crystallizing ability of glass is related to the linkage degree of glass network, the more breaking the glass network is, the easier the crystallization of glass. Although Ni and Ti metal crystal powders can provide the heterogeneous nuclei for the devitrification of the soda-lime glass, they cannot induce the crystallization of the glass because they are not in favor of the breakdown of glass network.<sup>24,25</sup> Mg<sup>2+</sup> cation is a modified species,<sup>8,26</sup> while it diffuses from the promoters to the glass during sintering, the local Mg concentration in the glass around the promoters was richer, which would decrease the viscosity of glass, lead to the breakdown of glass network and enhance the tendency of glass crystallization.<sup>26,27</sup> At same time, Ca<sup>2+</sup> and Na<sup>+</sup> cations diffuse also from the glass to the promoters, which would make the promoters transform into the nuclei of diopside and albite according to the following schematic equations:

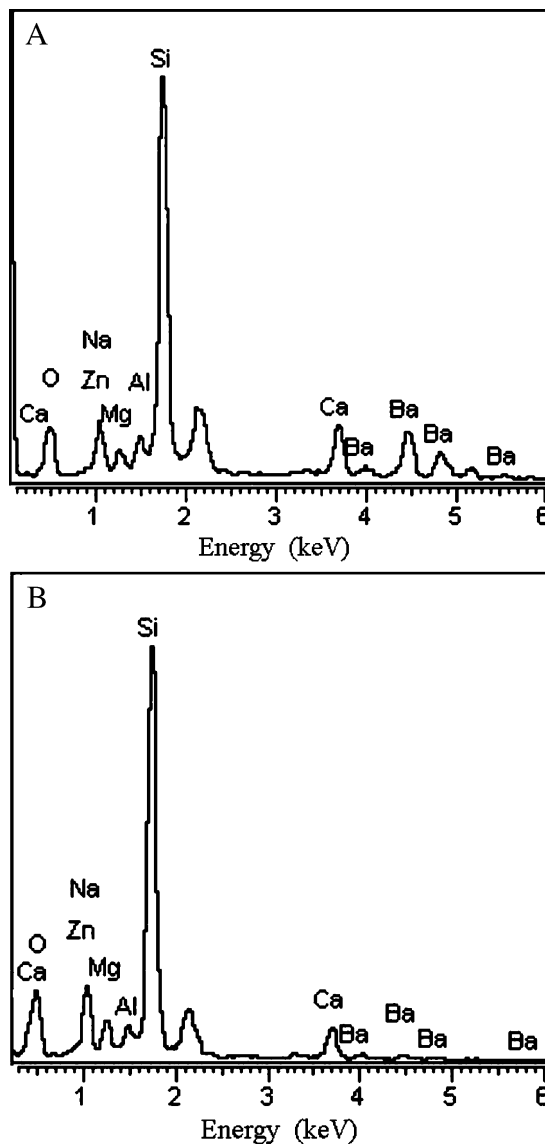
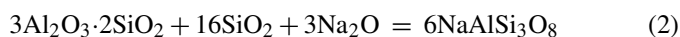
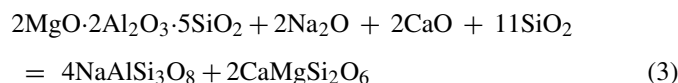


Fig. 6. EDS spectra of (a) diopside and (b) albite in glass–ceramic GD2-20.



Thus, Mg-rich glass around the promoters could crystallize epitaxially on these nuclei. As a result, the reasons that crystallization promoters induce the crystallization of the soda-lime glass are: (i) providing the heterogeneous sites for nucleating of diopside and albite and (ii) facilitating the breakdown of glass network.

In addition, the pyroxenes are a wide family of minerals with a wide chemical composition. A wide variety of ionic substitutions and an isomorphism of the various elements occurred in the members of the pyroxene group. Al<sup>3+</sup> in the promoters can diffuse into the diopside to replace some Mg<sup>2+</sup> and Si<sup>4+</sup>, therefore, the resultant diopside was aluminum-containing one.<sup>7</sup>

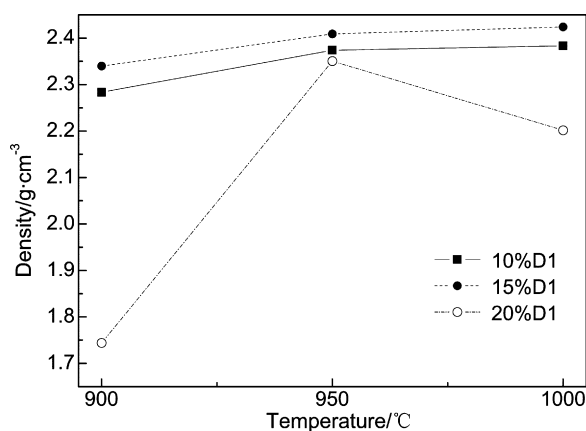


Fig. 7. Effect of D1 contents and temperatures on the density of glass-ceramics.

### 3.3. Densification

The densities of the glass-ceramics with different D1 contents sintered at various temperatures are shown in Fig. 7. While the contents were below 15%, the densities increased with temperatures. While the content was above 20%, the density reached a maximum value at 950 °C and then decreased with further increasing temperature. The glass-ceramic with 15%D1 had a maximum density among all glass-ceramics sintered at the identical temperature, suggesting the effect of D1 content on the density is not monotonic, the density initially increased then decreased with increasing the D1 content.

It can be seen from Fig. 8, the effect of D2 contents on the densities had the same tendency as D1, *i.e.*, the density initially increased and then decreased with increasing the D2, but the maximum value of density appeared in the glass-ceramic with 12% content instead of 15%. The density reached a maximum value for all glass-ceramics except the glass-ceramic GD2-20 at 950 °C.

The densification of the glass powder compact is achieved by the viscous flow of the glass,<sup>28</sup> generally, raising temperatures can improve the densification due to reducing the glass viscosity. At present work, however, the densities of compacts were related not only with the sintering temperatures but also with

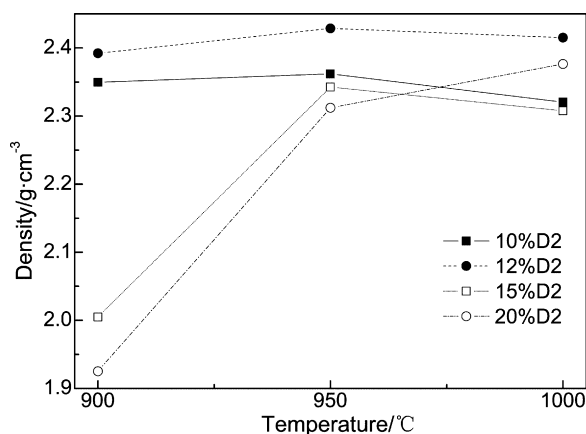


Fig. 8. Effect of D2 contents and temperatures on the densities of glass-ceramics.

Table 3

Mechanical properties of the glass-ceramics sintered at 950 °C, standard deviation values are given in parentheses.

Samples	Compression strength (MPa)	Bending strength (MPa)	Hv (GPa)
GD1-10	46 (±9.9)	15 (±5.5)	5.86 (±0.09)
GD1-15	78 (±16.4)	43 (±8.5)	7.03 (±0.61)
GD1-20	63 (±12.8)	22 (±7.4)	7.19 (±0.15)
GD2-10	49 (±6.3)	18 (±3.2)	5.98 (±0.1)
GD2-12	116 (±18.8)	62 (±7.3)	7.77 (±0.19)
GD2-15	105 (±11.3)	54 (±8.8)	7.86 (±0.54)
GD2-20	83 (±8.1)	28 (±10.5)	7.27 (±0.23)

the addition of crystallization promoters because incorporation of the promoters into glass powder can hinder the viscous flow of the glass.<sup>25,29,30</sup> There was a critical value of the promoter, the density of glass-ceramic increased with temperatures if below it, but the density increased initially and then decreased with temperatures after reaching a maximum value if above it. Based on these results, the critical values were 15% for D1 and 12% for D2, respectively. The conclusion can be further illustrated by the microstructural evolution. It can be seen from Fig. 9 that most of crystals embedded in the glassy matrix except the ones in some pores, together with a handful of pores distributed on the glassy matrix in GD1-15 and GD2-12 (Fig. 9a, b, e, and f), suggesting a less resistance to the viscous flow of the glass because a less crystals were formed by the reactive crystallization and they would dispersed in the glassy matrix. At this case, the glass could flow well at higher temperatures and the density increased with temperatures. When the promoter addition was above the critical value, more crystals were formed and they would contact each other to form a denser crystal skeleton in the glassy matrix, meaning a greater resistance to the viscous flow of the glass because the viscous glass penetrated hardly to the gaps in the denser skeleton. Therefore, denser interlocked crystals presented within each of pores and more pores appeared, as shown in GD1-20 and GD2-20 (Fig. 9c, d, g, and h). At this case, raising temperature can not only reduce the glass viscosity but also encourage the formation of more crystals by reactive crystallization which, in turn, offsets the positive effect of raising temperatures. Consequently, the densification was hindered and the density declined instead of increased as the temperatures increase.<sup>25,30</sup> Moreover, the induced crystallization pores might be another reason for the decrease in the density with increasing temperatures.<sup>31</sup>

### 3.4. Mechanical properties

Table 3 shows the mechanical properties of glass-ceramics, the strength initially increased and then decreased with increasing the additions of crystallization promoters. The initial improvement of strength could be attributed to the formation of more crystals which acted as a reinforcement to the glassy matrix, however, the increase in crystallinity hindered the densification, resulting in the formation of more pores and in turn decreasing the strength of glass-ceramics. Thus the variety of strength with the gradual addition of promoters was basically

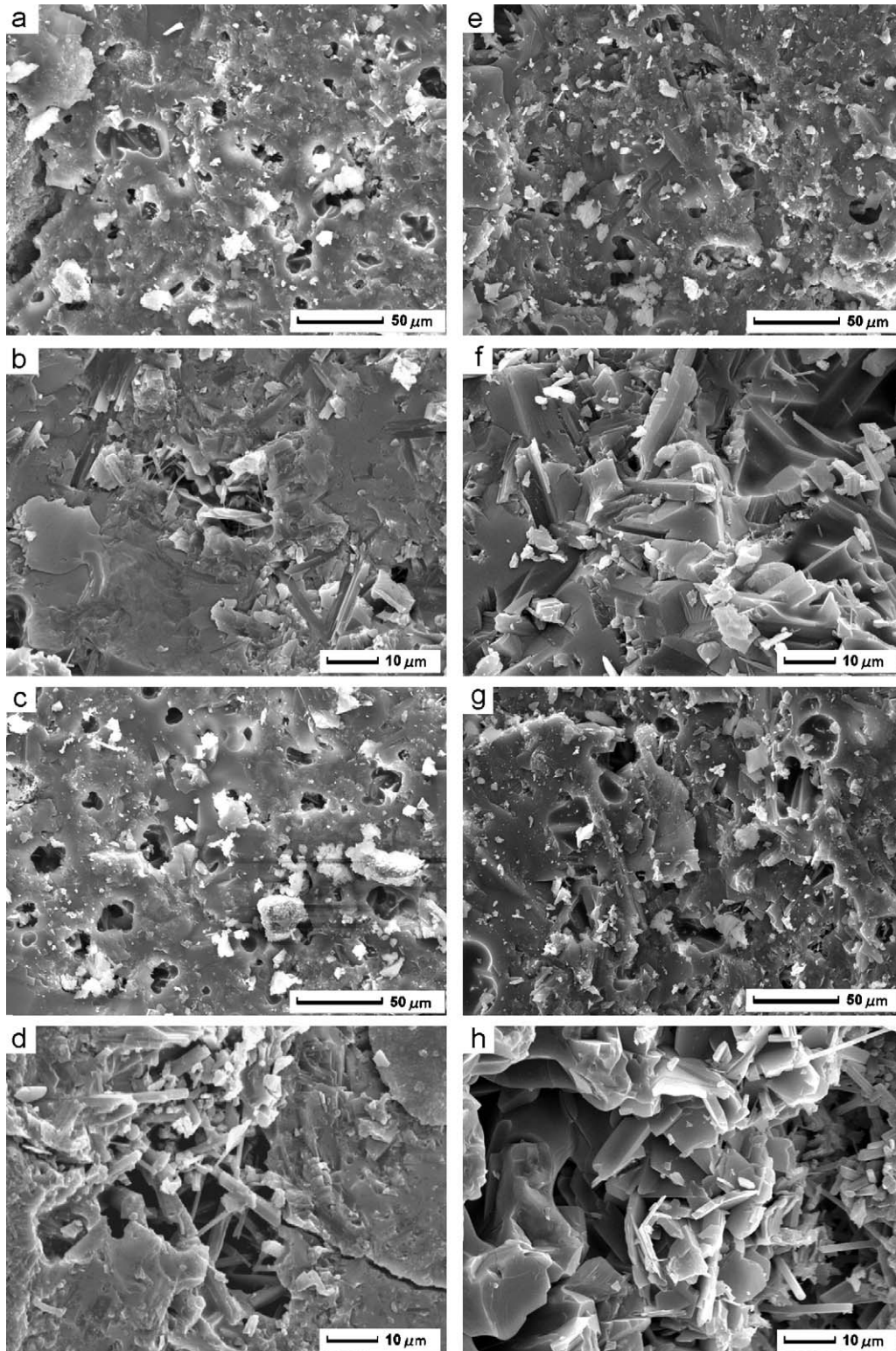


Fig. 9. Microstructures of glass–ceramics sintered at different temperatures for 2 h (SEM): (a and b) GD1-15 (950 °C); (c and d) GD1-20 (1000 °C); (e and f) GD2-12 (950 °C); (g and h) GD2-20 (1000 °C).

same as the one of density, and the strength reached to the maximum values in the glass–ceramics GD1-15 and GD2-12, respectively. It should be pointed out that the mechanical properties of the glass–ceramic GD2 series were superior to those of the GD1 series under the same addition of promoters, this was

related with their different microstructures. The fracture surfaces of the glass–ceramics GD2 were rough, indicating that crack deflection occurred by the diopside and albite crystals embedded in the glass matrix while crack propagation (Fig. 9e–h). However, the fracture surfaces of the glass–ceramics GD1 were

flat, together with more pores distributed on it, meaning that the resistance to the crack propagation was lower (Fig. 9a–d).

#### 4. Conclusions

It was feasible to fabricate diopside–albite glass–ceramics by directly adding two kinds of crystallization promoters, one consisting of enstatite, quartz and mullite and the other consisting of enstatite and cordierite, to the soda–lime glass powder and then sintering. During sintering, the glass reacted with enstatite to form diopside, with mullite and quartz to form albite, and with cordierite to form both diopside and albite, respectively. Although the preparation of promoters requires higher energy due to calcination at high temperature, the preparation of glass–ceramics needs much lower energy because they sinter at lower temperatures of  $<950^{\circ}\text{C}$ . Compared with the traditional sinter–crystallization route which requires a particular formulated base glass, the reactive crystallization route is a low energy consuming technique since only small amount of promoter ( $<20\text{ wt}\%$ ) is required and large amount of waste glasses can be directly utilized instead of remelted. The promoters acted as two roles in the reactive crystallization of glass: (i) the heterogeneous nuclei for the precipitation of crystalline phases; (ii) increasing the crystallizing ability of the soda–lime glass. The densification behaviors of glass–ceramics depended on the type and content of promoters and sintering temperatures. In order to obtain a denser glass–ceramics, a simultaneous adjustment of content of promoters and sintering temperature was necessary. The glass–ceramics with 15% D1 or 12% D2, sintered at  $950^{\circ}\text{C}$ , presented considerable densities and mechanical properties, having potential applications as building materials.

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