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A low cost route for fabrication of wollastonite glass–ceramics directly using soda-lime waste glass by reactive crystallization–sintering

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Abstract

Wollastonite glass–ceramics have been fabricated by directly mixing soda-lime waste glass powders with a synthetic crystallization promoter and then sintering, the wollastonite being formed by a reaction between crystallization promoter and glass during sintering. Effects of the crystallization promoter contents and processing parameters on the reactive crystallization behavior, sinterability and properties of glass–ceramics were investigated. The results showed that the level of reactive crystallization was predominantly controlled by temperature, and the sinterability of glass–ceramics deteriorated with increasing content of promoter and temperature. Raising temperature had a considerable influence on strength but less on hardness of glass–ceramics. The best mechanical properties were obtained for the glass–ceramic with 15% crystallization promoter sintered at 850 °C for 2 h.

Keywords: A. Sintering; Crystallization promoter; Reactive crystallization; Soda-lime waste glass

1. Introduction

Due to its aesthetic marble like appearance, outstanding mechanical properties and high chemical durability, wollastonite glass–ceramics are attracting more applications in construction and architecture used as floor– or wall–tile materials to replace the natural marble and granite [1]. Usually, in the conventional sinter–crystallization process, a particularly formulated parent glass must be used to ensure that the wollastonite precipitates from the glass during sintering [1–5]. The parent glass belongs to the $CaO–Al_2O_3–SiO_2$ ternary system, its specific composition is 8–30% CaO, 5–12% Al_2O_3 and 50–60% SiO_2 by weight together with a small amount of other oxides such as R_2O and MO et al. [1–3,6–8]. However, soda-lime waste glass cannot be directly used in the preparation of wollastonite glass–ceramics because of their unsuitable composition and low crystallizing tendency. At present, a common solution to the problem is to remelt it together with other components into a parent glass cullet with suitable composition and high crystallizing tendency for sinter–crystallization [2,6,9–11]. Obviously, compared with the high energy consuming route mentioned above, a route that recycles soda-lime waste glasses directly into wollastonite glass–ceramics without remelting is more attractive, as reported in our previous works [12–14], such as machinable fluoramphibole glass–ceramics obtained by sintering the powder mixtures of soda-lime waste glass and fluormica and diopside–albite glass–ceramics obtained by sintering the powder mixtures of soda-lime waste glass and crystallization promoter. The main purpose of this study is to attempt the possibility to prepare wollastonite glass–ceramics by directly adding synthetic crystallization promoter into soda-lime waste glass powder and then sintering. The effects of crystallization promoter content and processing parameters on the reactive crystallizing behavior, sinterability and properties of wollastonite glass–ceramics were investigated.

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2. Experimental procedure

2.1. Synthesis of crystallization promoter

Kaolin (Jinan Jiyuan Corporation, China) and chemical reagents were used as starting materials for the synthesis of crystallization promoter, the compositions of kaolin were (wt%): 47.0 SiO_2, 38.3 Al_2O_3, 0.47 CaO, 0.14 MgO, 0.7 K_2O, 0.39 Fe_2O_3, and 13 LOI. The promoter consisted of (wt%) 30 kaolin, 50 $CaCO_3$, 10 $BaCO_3$ and 10 ZnO, the addition of BaO and ZnO was to promote crystallization and improve sinterability of glass–ceramics [15,16]. A green body was made by adding some water into the homogenized raw materials, dried at room temperature and fired at 1100 °C for 2 h to synthesize a crystallization promoter. The obtained promoter was crushed, ball-milled in a ceramic jar mill with alumina media and sieved to powders $<10\ \mu m$.

2.2. Preparation of glass–ceramics

Waste window glass was used as the base glass whose chemical composition was (wt%) 72.0 SiO_2, 8.2 CaO, 14.3 Na_2O, 1.3 Al_2O_3, 3.5 MgO, and 0.3 K_2O. The glass was cleaned, crushed, ball-milled and sieved to powders $<150\ \mu m$.

The glass powder combined with 12%, 15%, 20% and 25% crystallization promoter by weight, denoting to GW12, GW15, GW20 and GW25, respectively. The powdered 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 850 °C–1000 °C at a rate of 4 °C/min for different dwell time and then allowed to cool inside the furnace to the ambient temperature to obtain the final glass–ceramics.

2.3. Characterization

The bulk densities of the glass–ceramics were measured by the Archimedes method and five 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 three measurements at each sample. Compression strength and three–point bending strength were measured by using cylindrical compacts (10 mm diameter × 10 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 (JEOL, 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 ($<40\ \mu m$) of the glass–ceramics were applied for phase identification via X–ray diffraction (XRD) using a diffractometer (Philips, PW–1710) 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 promoter

As clearly seen in Fig. 1, only one crystalline phase, gehlenite ($Ca_2Al_2SiO_7$, JCPDS, 35–0755), was present in the synthetic crystallization promoter. Obviously, a reaction between kaolin and $CaCO_3$ occurred during the firing process to form the gehlenite, which was consistent with the result of the mixture of kaolin and calcite fired at 1100 °C [17]. The reaction paths may be as following:

$$CaCO_3 \rightarrow CaO + CO_2\uparrow$$

$$Al_2Si_2O_7 + 2CaO \rightarrow Ca_2Al_2SiO_7 + SiO_2$$

3.2. Reactive crystallization in glass–ceramics

Fig. 2 shows the evolution of crystallization in the glass–ceramic GW12 during isothermal treatment at 850 °C. Upon heating up to 850 °C, wollastonite ($CaSiO_3$, JCPDS, 42–0550) had started to form but gehlenite still existed, and the peaks corresponding to wollastonite became more prominent while the ones corresponding to gehlenite ($2\theta = 31.42°$ and 37°) became weaker with increasing dwelling until vanished completely after 12 h. Consequently, a reaction between the promoter and the glass occurred, resulting in the decomposition of gehlenite and the formation of wollastonite at the expense of gehlenite. Both wollastonite and gehlenite coexisted in the glass–ceramics with different additions of the promoter sintered at 850 °C for 2 h. The peaks corresponding to wollastonite in all of glass–ceramics were not significantly different but the ones corresponding to gehlenite gradually became stronger with

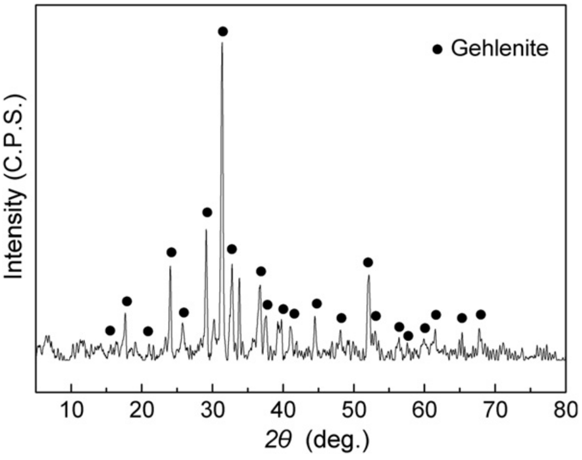

Fig. 1. XRD pattern of crystallization promoter.

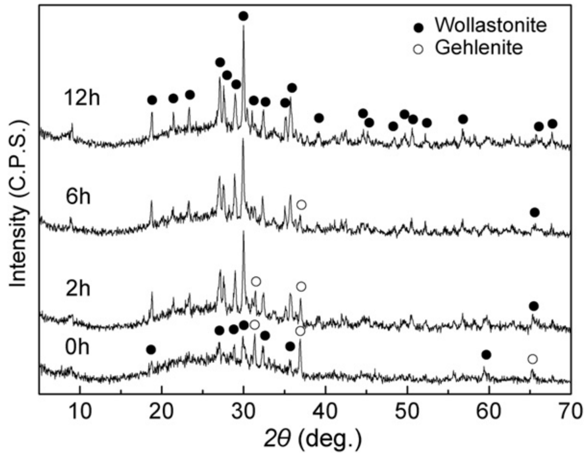

Fig. 2. Effect of isothermal time on crystallization of the glass–ceramic GW12 at 850 °C for 2 h.

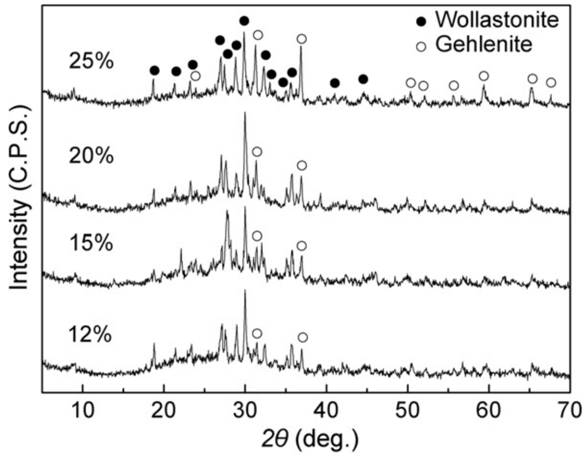

Fig. 3. Effect of promoter content on crystallization of glass–ceramics sintered at 850 °C for 2 h.

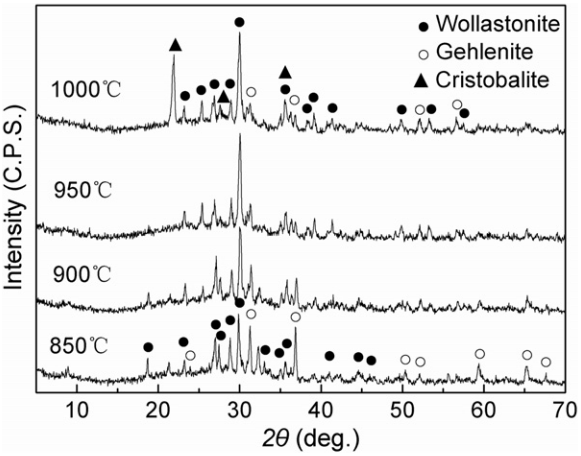

Fig. 4. Effect of temperature on crystallization of the glass–ceramic GW25 sintered at various temperatures for 2 h.

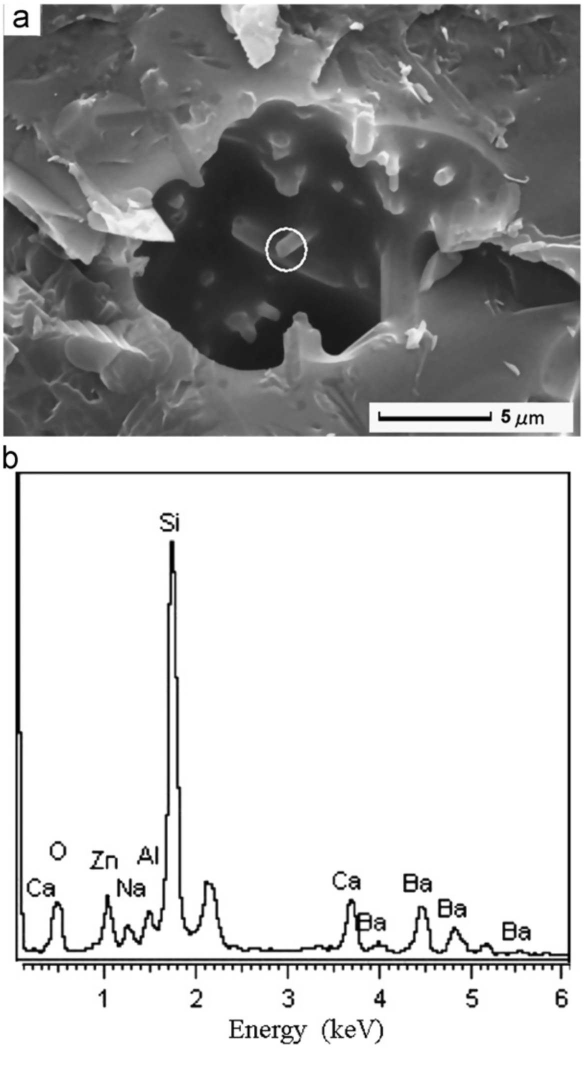

Fig. 5. (a) SEM micrograph of the glass–ceramic GW15 sintered at 850 °C and (b) EDS spectra of selected wollastonite crystal in (a).

the increase in the promoter addition, as shown in Fig. 3, indicating that the level of the reactive crystallization may be temperature–dependent. This hypothesis can be further proved by the XRD results of GW25 sintered at various temperatures, as shown in Fig. 4. It can be seen that there was considerable amount of gehlenite at 850 °C, however, the peaks corresponding to gehlenite gradually became weaker while the ones corresponding to wollastonite became stronger with increasing temperature, suggesting that raising temperature facilitated the wollastonite formation through the reactive crystallization. Heating up to 950 °C, gehlenite almost disappeared, in addition, cristobalite developed when further heated up to 1000 °C.

The morphologies of wollastonite crystals in glass–ceramic GW15 are shown in Fig. 5 a, they were rod–like and embedded in the glass matrix, the ones in other glass–ceramics were also similar. The EDS analysis shows that these rod–like crystals contained Al, Ba, Zn and Na besides Ca, Si and O elements (Fig. 5 b), therefore, the wollastonite formed by the reactive crystallization was a solid solution containing Al, Ba, Zn and Na [7,18]. Al, Ba and Zn came from the promoter and Na was from the glass, demonstrating that the wollastonite was the product of the reactive crystallization between promoter and glass again.

The wollastonite cannot be developed directly from the soda-lime glass due to its unsuitable composition and poor crystallizing ability, so that it is only formed by the reactive crystallization between promoter and glass. However, only few studies have been carried out on the aspect. Most glass–ceramics in the CAS glass systems could yield three kinds of crystalline phases: wollastonite, gehlenite and anorthite, mainly depending on the level of alumina [1,7,19]. In order to obtain wollastonite, the alumina should not exceed 12% in the glass composition, if not, gehlenite or anorthite would form [1,7,8,20–23]. For example, wollastonite developed in the CAS glass with 6.9% Al_2O_3 while gehlenite in the glass with 12.6–15.2% Al_2O_3 [1,7,21]. Barbieri et al. reported that with the increase in alumina content from 10.4% to 16.2%, the crystalline phases in the glasses evolved as following: wollastonite, anorthite+wollastonite and gehlenite+anorthite [8]. Müller et al. stated that while sintering of powder mixtures of calcium–alumo–borosilicate glass and 4.7% Al_2O_3, wollastonite firstly developed between 850 °C and 900 °C and then anorthite formed above 900 °C owing to the interaction among alumina, wollastonite and residual glass [24]. Gehlenite crystallized in the CAS glass powder with 12.6% Al_2O_3 sintered at 850 °C, further heating up to the temperatures of 950–1050 °C the content of gehlenite decreased and some wollastonite formed [21], Yun et al. also reported a similar result that the gehlenite reacted with a glass containing low alumina to form wollastonite [11]. Therefore, it may be a reasonable hypothesis based on the above analyses that gehlenite would become unstable in the glass with low alumina and easily transform into wollastonite. In the present work, the level of Al_2O_3 is very low in the soda-lime glass, a diffusion of aluminum from the promoter to the glass would occur during sintering [11], and resulted in the decomposition of gehlenite and the formation of wollastonite.

3.3. Densification of glass–ceramics

Fig. 6 shows the effect of the crystallization promoter contents on the sintering behaviors of glass–ceramics, it can be seen that the density increased with more promoter content, which would be related to the increase of residual gehlenite amount (Fig. 3) because the gehlenite density of 3.05 g/cm^3 is greater than the wollastonite (2.92 g/cm^3). However, the water absorption changed slightly when the promoter content was below 20%; the absorption increased dramatically with the promoter content above 20%. This sudden increase indicates that the viscous flow of the glass was hindered severely. The densification of the glass/crystal powder compact is mainly achieved by the viscous flow of the glassy phase [25], which is related to the amount of glassy phase in the compact and its viscosity. To add a crystalline phase to the glass powder can inhabit the densification because it (i) reduces the amount of glassy phase and (ii) increases the viscosity of the glassy phase [26]. For example, Dutton and Rahaman found that adding 20 vol% Ni crystalline powder to a soda-lime glass powder made the viscosity of the glass increase 100 times compared with that of the pure glass [27]. Thus, the resistance to the sintering of the compact became greater with more addition of crystalline phase. A critical amount exists and beyond it the densification will be impeded severely, this critical value depends on the composition of glass and the kinds of added crystals, it was 15% for the Ti [28], 20% for the fluormica [13] in the soda-lime glass powder and 15% for the alumina in the aluminum silicate glass powder [29]. In the present work, obviously, the critical value of the promoter was 20%. The conclusion can be further confirmed by the microstructural observations (Fig. 7), a few pores distributed on the glassy matrix in GW12, a slight increase in GW15, however, a great increase in GW20 and GW25, suggesting a less resistance to the viscous flow of the glass while the promoter content was below 20%.

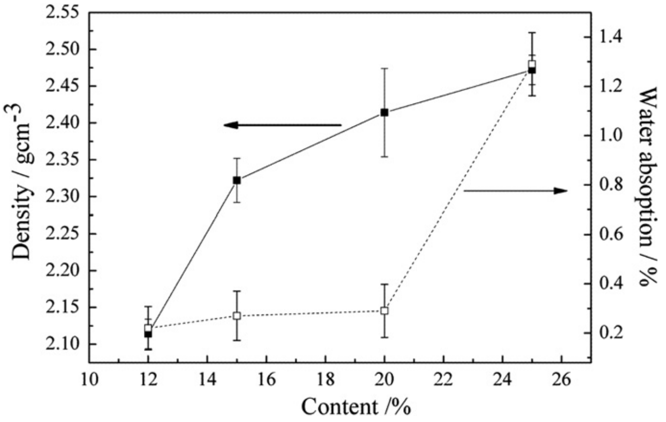

Fig. 6. Density and water absorption of glass–ceramics with different promoter content sintered at 850 °C for 2 h.

As shown in Fig. 8, both density and water absorption of GW25 decreased as temperature increases. As compared with the surface of GW25 sintered at 950 °C (Fig. 9a), more pores appeared and a lot of crystals presented within the pores in the interior of GW25, indicating that the resistance to the viscous flow of the glass is less in the surface than that in the interior. Raising temperature would reduce the viscosity of the glass which was in favor of the densification of the glass–ceramic, thus, the water absorption quickly dropped from 850 °C to 900 °C. At the same time raising temperature would also facilitate the reactive crystallization which resulted in the formation of more wollastonite, these crystals 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 and offsetting the positive effect of raising temperature [13,14]. Consequently, raising temperature could encourage the densification more effectively in the surface than in the interior, this is the reason of both density and absorption decreased simultaneously with increasing temperature. Another reason for the reduction of density should be arbitrated to the reduction of gehlenite amount and the increase of wollastonite amount with increasing temperature (Fig. 4). In addition, the decrease in density may be related to the formation of more induced crystallization porosity at higher temperature [8,30,31].

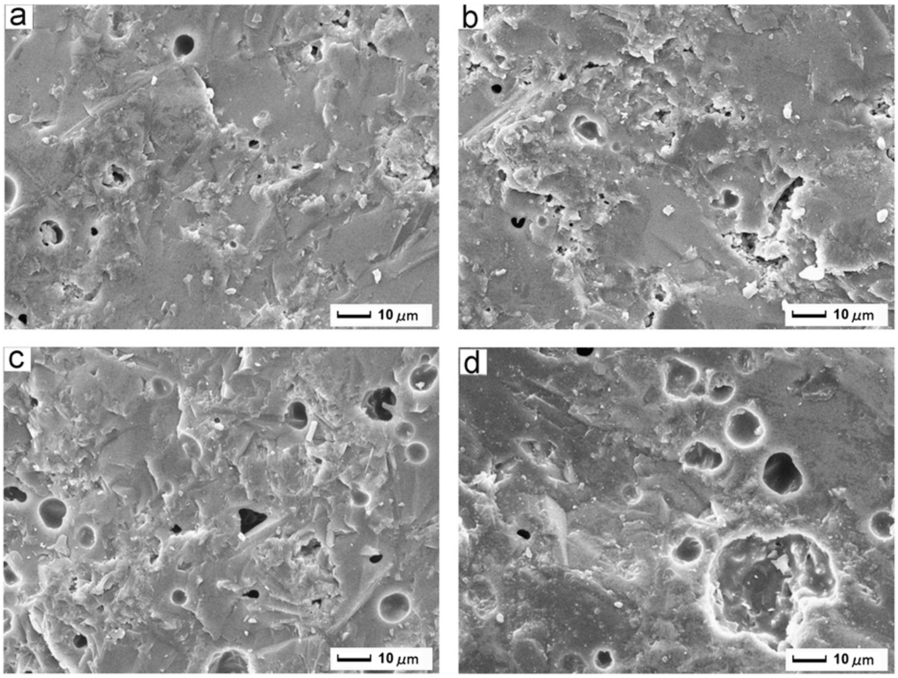

Fig. 7. Microstructures of glass–ceramics with different crystallization promoter contents sintered at 850 °C for 2 h (SEM) (a) GW12, (b) GW15, (c) GW20, and (d) GW25.

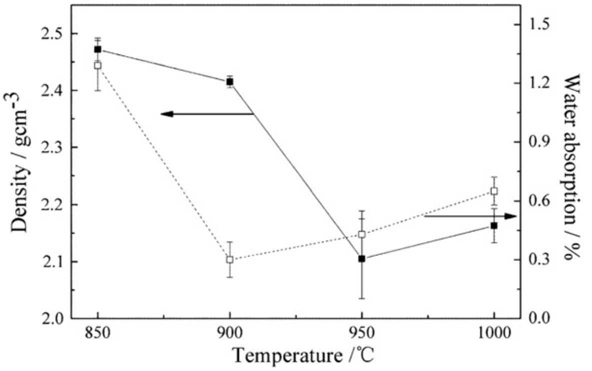

Fig. 8. Effect of temperatures on the density and water absorption of glass–ceramic GW25.

3.4. Mechanical properties

Table 1 shows the mechanical properties of the glass–ceramics with different promoter contents, the strengths initially increased and then decreased with more addition of promoter, and reached the maximum value of the GW15. The crystals in the GW15 were linked each other by the glassy phase to form a solid spatial skeleton (Fig. 5a), and its microstructure was denser (Fig. 7b), which would deflect crack and enhance the glass matrix well. Although the microstructure of the GW12 was denser too, its crystallinity was lower (Fig. 3), which could not enhance the glass matrix well. While the promoter content exceeded 20%, the densification was hindered and more

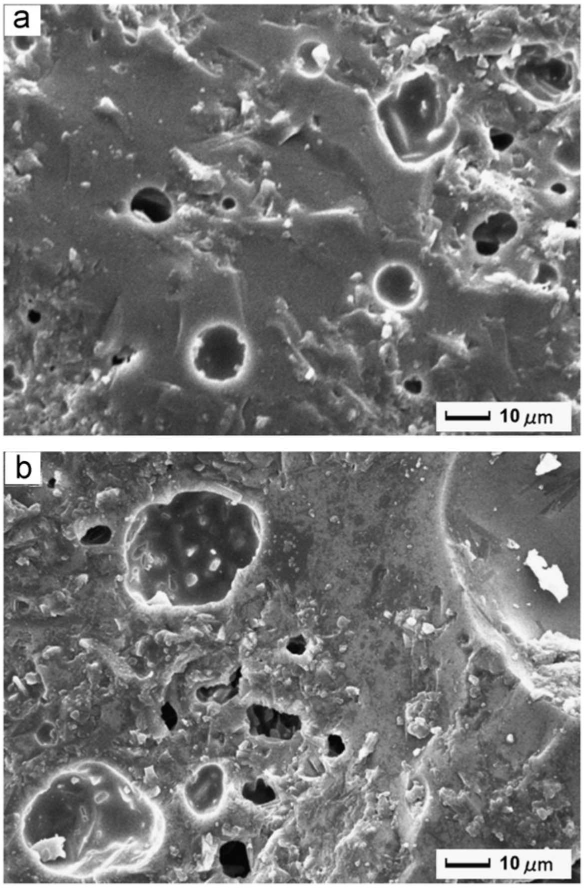

Fig. 9. Microstructures of glass–ceramic GW25 sintered at 950 °C for 2 h (SEM). (a) surface, and (b) interior.

Table 1
Effect of crystallization promoter contents on the mechanical properties of glass–ceramics sintered at 850 °C for 2 h.

Sample	GW12	GW15	GW20	GW25
Compressive strength (MPa)	157 ± 24	247 ± 21	216 ± 26	183 ± 32
Bending strength (MPa)	44 ± 12	119 ± 14	112 ± 11	75 ± 11
Vickers hardness (GPa)	4.52 ± 0.2	5.32 ± 0.1	5.08 ± 0.1	5.30 ± 0.15

Table 2
Effect of sintering temperatures on the mechanical properties of glass–ceramic GW25.

Temperature (°C)	850	900	950	1000
Compressive strength (MPa)	183 ± 32	194 ± 32	142 ± 12	109 ± 25
Bending strength (MPa)	75 ± 11	45 ± 10	41 ± 8	23 ± 3
Vickers hardness (GPa)	5.30 ± 0.15	5.51 ± 0.1	5.71 ± 0.2	5.75 ± 0.2

pores were formed (Fig. 7 c and d), resulting in the reduction of the strengths. However, the promoter content had less effect on the hardness of glass–ceramics except GW12, its lower hardness may be related to its lower crystallinity. As shown in Table 2, raising temperature deteriorated the strengths of GW25, especially the bending strength. There were several reasons to explain the phenomena. Firstly, the strengths of glass–ceramics deteriorated with decreasing density (Fig. 8) [2,32], secondly, raising temperature would lead to crystal growth, coarser microstructure and formation of more porosity (Fig. 9b) [12,21]. It should be noted, however, that the hardness increased slightly with increasing temperatures because raising temperature encouraged the reactive crystallization and increased the crystallinity of wollastonite, especially at 1000 °C, cristobalite developed and it had Hv 9.62 GPa which is higher than Hv 4.1–5.24 GPa of wollastonite. Therefore, the highest hardness was obtained at 1000 °C.

4. Conclusion

It was feasible to fabricate wollastonite glass–ceramics by directly adding crystallization promoters to the powder and then sintering. Wollastonite could be formed by the reactive crystallization between promoter and glass. As a result, this novel route is not only a low energy consuming but also an environmental friendly process. The glass–ceramic with 15% crystallization promoter sintered at 850 °C for 2 h presented the best properties: water absorption 0.27%, compressive strength 247 ± 21 MPa, bending strength 119 ± 14 MPa and hardness 5.32 ± 0.1 GPa that expects an attractive potential for building materials application.

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