

## Glass-ceramics prepared by waste fluorescent glass

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### Abstract

To prepare glass-ceramics reinforced by  $\beta$ -wollastonite, fluorescent glass and calcium carbonate were used as starting materials.  $\beta$ -Wollastonite, gehlenite and sodium calcium silicate were observed by X-ray diffraction analysis, and surface morphology and chemical composition were evaluated by field emission-scanning electron microscopy and energy dispersive X-ray spectrometer. © 2002 Published by Elsevier Science Ltd and Techna S.r.l.

**Keywords:** Fluorescent glass;  $\beta$ -Wollastonite; Glass-ceramic

### 1. Introduction

So-called synthetic wollastonite, known in art, is made by thermal interaction of CaO and SiO<sub>2</sub> and comprises either predominantly a glass phase consisting of cristobalite and quartz [1–3]. As known in art shaped products composed of wollastonite have excellent properties exhibiting no deterioration at high temperatures of above 1000 °C. Therefore, the products are expected to be useful as thermal insulating materials and refractories, and in road construction compositions, but there has been no useful and economical method of preparation [4].

Moreover, approximately, above 0.6 million ton of glass waste are generated in South Korea on an annual basis including glass containers, light bulbs, plate glass and automobile glass [5]. Thus, a huge opportunity exists to convert this glass waste from an environmental and economic burden to a profitable, value-added

resource. In this paper, to utilize waste fluorescent glass and resolve environmental problems, we prepared glass-ceramics reinforced by  $\beta$ -wollastonite.

### 2. Experimental

Fluorescent glass and calcium carbonate (CaCO<sub>3</sub>) were used as starting materials. Waste fluorescent glass cullet was carefully washed, and dried at 110 °C for 24 h. The composition of the mother glasses of the glass-ceramics used in this work were fixed at glass cullet: CaCO<sub>3</sub>=4:1 in weight ratio. About 30 g powder mixtures of these compositions were put in an alumina crucible and melted at 1300 °C for 1 h. To quench, the melts were rapidly poured into a water bath and dried at 110 °C for 24 h. The quenched glass was ground and pressed into disks 0.5 cm thick.

The formed glass disks were heated up to 800, 900 and 1000 °C at a rate of 5 °C min<sup>-1</sup> for 1 h, respectively, and allowed to cool inside the furnace. The heat-treated glass-ceramic specimens were cleaned with ethyl alcohol in an ultrasonic cleaner and dried at 110 °C for 24 h in air.

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Crystallinity of the specimens was analyzed by X-ray diffraction (XRD, D-Max-1200, Rigaku Co., Jpn.). The morphology and composition of the surface of the specimens were evaluated using field emission-scanning electron microscopy (FE-SEM, S-4700, Hitachi Co., Jpn.) equipped with an energy dispersive X-ray spectrometer (EDX).

### 3. Results and discussion

The XRD results on the glass-ceramics showed that a mixture of phases, such as  $\beta$ -wollastonite ( $\text{CaSiO}_3$ ), gehlenite ( $\text{Ca}_2\text{Al}_2\text{SiO}_7$ ) and sodium calcium silicate (SCS,  $\text{Na}_2\text{Ca}_3\text{Si}_6\text{O}_{16}$ ) appeared. Peak intensities corresponding to the SCS ( $2\theta=18\sim19^\circ$  and  $32^\circ$ ) and the gehlenite ( $2\theta=31^\circ$ ) decreased with increase of heat-treatment temperature, as clearly shown in Fig. 1. On the contrary, with increase in heat-treatment temperature, peak intensity at  $2\theta=30^\circ$ , corresponding to  $\beta$ -wollastonite, increased. At  $1000^\circ\text{C}$ , we confirmed the highest crystallized  $\beta$ -wollastonite reinforced glass-ceramic appeared.

Fig. 2 shows the polished surface morphology of the glass-ceramics at various heat treatment temperatures.

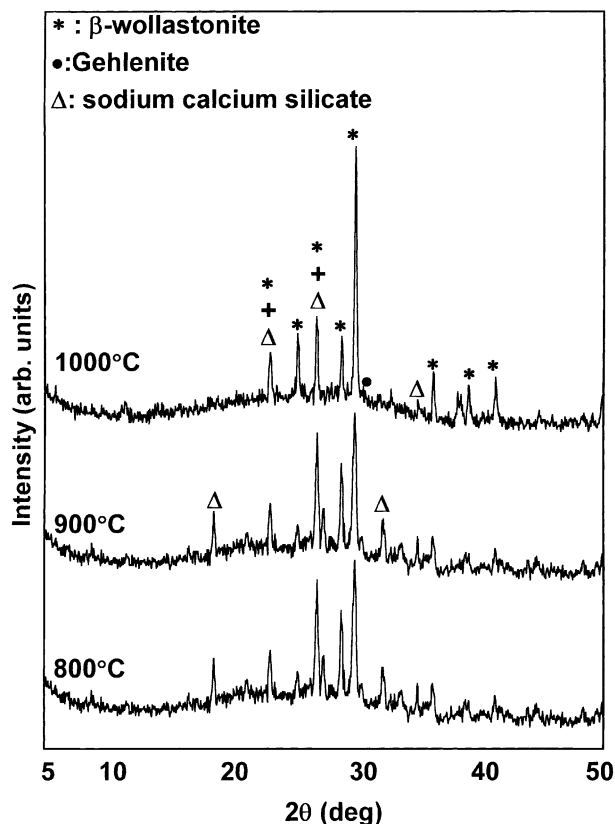


Fig. 1. XRD patterns for the glass-ceramics heat treated at various temperatures.

Morphological analysis of the glass-ceramics heat-treated at 800 and  $900^\circ\text{C}$  shows some well-crystallized round-shaped grains in the matrix. However, as the heat treatment temperature was increased to  $1000^\circ\text{C}$ , the round-shaped crystal decreased from about  $30\sim35\ \mu\text{m}$  to about  $15\sim20\ \mu\text{m}$  in size. To more clearly investigate crystal composition, we performed EDX analysis for the same area used in morphological analysis. EDX analysis of the breakage-part (denoted B in Fig. 2) of the round-shaped crystals indicated the presence of gehlenite in glass-ceramics, whereas the hollow-part (denoted H in

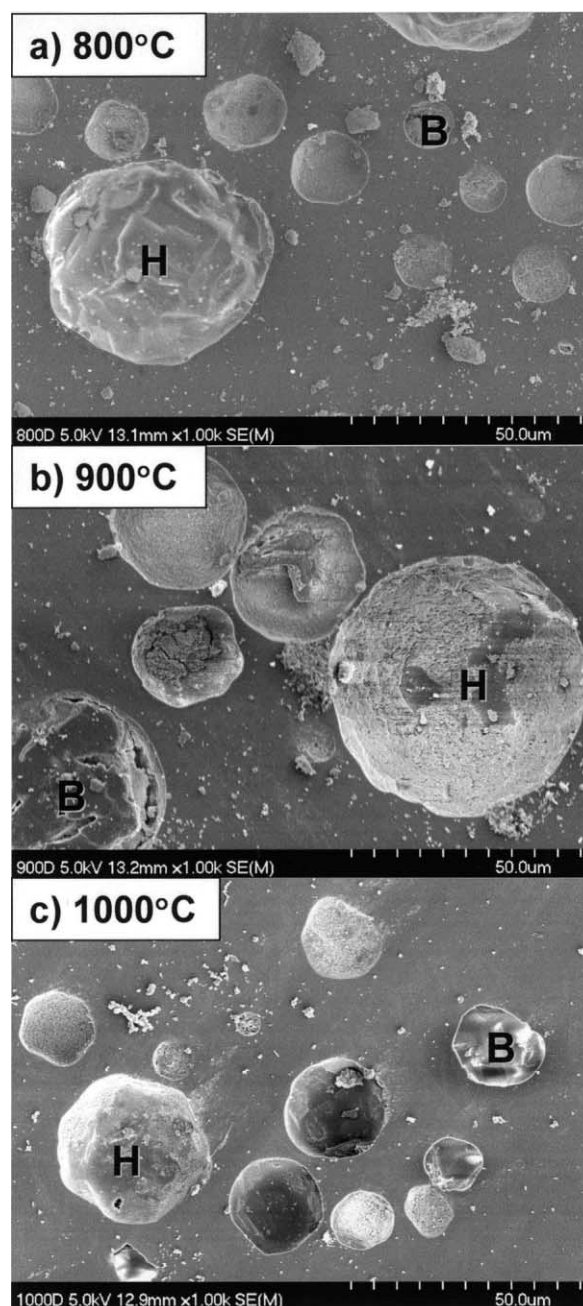


Fig. 2. Surface morphology of the glass-ceramics heat treated at various temperatures.

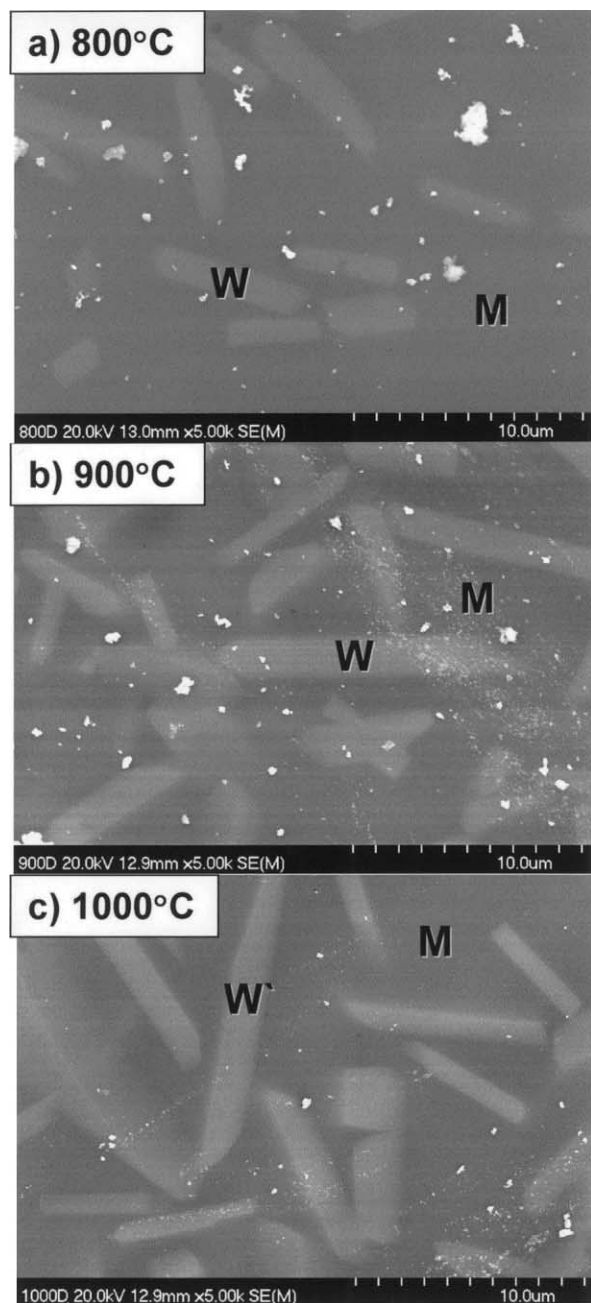


Fig. 3. Surface morphology at higher magnification of the glass-ceramics heat treated at various temperatures.

Fig. 2), probably due to the detachment of gehlenite grains in the matrix, was composed of a mixture of gehlnite and SCS.

However, there is no morphological data of the  $\beta$ -wollastonite for our specimens, although previous XRD exhibited the presence of the  $\beta$ -wollastonite. Thus, to detect the  $\beta$ -wollastonite, we performed FE-SEM analysis at higher magnification. As shown in Fig. 3, whisker-type  $\beta$ -wollastonite (denoted W) in  $\text{SiO}_2$  glass matrix (denoted M) confirmed by EDX, not shown here, were observable. Similar to previous XRD, heat-treatment temperature increased from 800 to 900 and 1000 °C, the  $\beta$ -wollastonite significantly increased.

Further experimental study is needed to investigate the relationship between the mechanical strength and the chemical durability, in order to apply for practical usage.

#### 4. Conclusion

To utilize waste fluorescent glass and to resolve environmental problems, we have prepared  $\beta$ -wollastonite glass-ceramics. With increase of the heat treatment temperature, the round-shaped crystals composed gehlenite and SCS in the matrix decreased. Whisker-type  $\beta$ -wollastonite in  $\text{SiO}_2$  glass matrix significantly increased, with the increase of the heat-treatment temperature.

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#### References

- [1] M. Kume, T. Mizumn, US patent 4443550, 1984.
- [2] K. Kubo, US patent 3928054, 1975.
- [3] V. Swamy, L.S. Dubrovinsky, F. Tutti, J. Am. Ceram. Soc. 80 (1997) 2237–2247.
- [4] K.C. Rieger, Am. Ceram. Soc. Bull. 74 (1995) 160–161.
- [5] An Environmental White Paper, The Ministry of Environment, Republic of Korea, 2000.