Journal of
Ceramic Science
and Technology

J. Ceram. Sci. Tech., 04 [04] 193-196 (2013)
DOI: 10.4416/JCST2013-00019
available online at: http://www.ceramic-science.com

Investigation of the Mechanical Properties and Machinability of Fluorphlogopite-Gehlenite Glass-Ceramics

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received July 23, 2013; received in revised form November 10, 2013; accepted November 20, 2013

Abstract

The gradual addition of CaO to a SiO_2-Al_2O_3-B_2O_3-MgO-K_2O-F glass-ceramic composition led to the precipitation of gehlenite crystals and the formation of a potassium-calcium solid solution of fine mica crystals. The above additions almost tripled the bending strength and led to a ~ 1.5-fold increase in the fracture toughness of the glass-ceramic specimens. When the amount of CaO reached 30 weight parts, the microhardness of glass-ceramic specimens was considerably reduced while their machinability was improved.

Keywords: Mechanical properties, glasses

I. Introduction

Fluorphlogopite glass-ceramics are usually considered as the best candidates among machinable glass-ceramics [1–4]. The good machinability, besides other properties like suitable bioactivity and a high dielectric constant, makes these mica-based glass-ceramics suitable for various applications like dental restorations, electrical insulators, in welding technology as holders for welding components, etc. [5,6].

The improvement of the mechanical properties of these materials has also been considered by researchers, who have tried to modify the glass composition [7–11] and the glass-forming methods in order to improve the glass-ceramic microstructures [12–15].

In the present work the authors have tried to improve the mechanical properties of a fluorphlogopite-based glass-ceramic by developing new crystalline phases, through the addition of various amounts of CaO to an initial glass composition. The machinability of the resulting glasses and glass-ceramics has also been investigated.

It should be noted that the sinterability and crystallization behavior of the above-mentioned glasses have been investigated previously [16]. It has been shown that the addition of more than 15 weight parts of CaO to the base glass leads to the appearance of gehlenite as a new crystalline phase [16]. Furthermore, the sinterability of the above-mentioned glasses has shown a significant connection between densification and crystallization temperatures of glasses, i.e. the 5-parts-weight-CaO-containing glass (C5), which had the lowest DTA crystallization peak temperature, required the highest sintering temperature, i.e. 1180 °C, and the 30-parts-weight-CaO-containing glass (C30) that had the highest DTA crystallization temperature, required the lowest sintering temperature, i.e. 1000 °C. Besides, the XRD patterns of the glasses after firing at their optimum sintering temperatures showed that gehlenite was only retained in the densified specimen C30, which had the lowest densification temperature.

II. Experimental Procedures

Table 1 shows the chemical composition of various glasses. Details of the preparation of the glass frits and glass-ceramics have been provided elsewhere [16]. However, the bulk glasses needed for the microhardness analysis were obtained by melt-casting the prepared glasses into a preheated steel die.

Table 1: Chemical composition of glasses (parts by weight).

Oxides	C0	C5	C10	C15	C20	C30
SiO_2	37.96	37.96	37.96	37.96	37.96	37.96
Al_2O_3	15.36	15.36	15.36	15.36	15.36	15.36
B_2O_3	6.32	6.32	6.32	6.32	6.32	6.32
MgO	18.10	18.10	18.10	18.10	18.10	18.10
CaO	–	30.00	20.00	15.00	10.00	5.00
K_2O	7.56	7.56	7.56	7.56	7.56	7.56
F	9.00	9.00	9.00	9.00	9.00	9.00
TiO_2	5.60	5.60	5.60	5.60	5.60	5.60

The three-point bending strength of the resulting glass-ceramics was measured with an Instron universal testing machine (Model 1196, USA). Five polished rectangular specimens (50 × 10 × 5 mm) chosen from each com-

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position were tested. The fracture toughness of the prepared glass-ceramics was measured with the Chantikul method [17]. Five polished rectangular specimens with the same dimensions as mentioned above were also used for this test. A Vickers microhardness tester with a diamond pyramid indenter (Buehler, Micromet 1) was used to measure the microhardness. The load was 4.9 N and the loading time was 30 s.

The machinability of glass-ceramics was evaluated by naked eye observation of the drilled specimens and two other criteria, that is brittleness (B) and n factor [18, 19]. The glass-ceramics were drilled with a 2 mm conventional drill, at a drilling rate of 3.8 cm/min at a drilling speed of 300 rpm. The result of this examination was compared with the calculated amounts for brittleness and the n factor.

The microstructure was investigated with a scanning electron microscope (SEM, Cambridge Instruments, Cambridge, UK).

III. Results and Discussion

(1) *Mechanical properties*

Fig. 1 shows the relationship between the bending strength of various compositions and their CaO content. It is obvious that the bending strength rises with the increase of CaO content, so that it almost tripled in specimen C30 in comparison with specimen C0. Several factors can be responsible for this behavior, as follows: a) reduction in the size of the plate-like fluorphlogopite particles, b) the formation of gehlenite as a new crystalline phase with high mechanical strength in specimens C20 and C30, and c) the formation of a calcium fluorphlogopite crystalline phase along with the potassium fluorphlogopite and/or the formation of a partial solid solution of calcium-potassium fluorphlogopite [20].

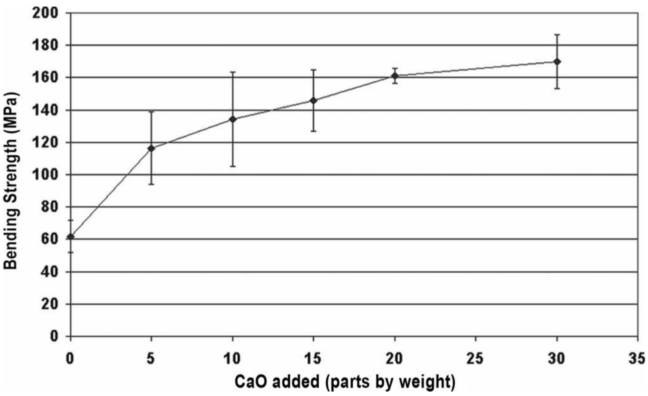

Fig. 1: Three-point bending strength of specimens as a function of CaO.

Figs. 2a – 2c show the randomly distributed plate-like mica particles in different specimens C0, C10 and C30. The statistical assessment of micrographs showed that the mean length of plate-like particles decreased gradually with CaO addition from ~ 5 μm in specimen C0 to ~ 2.5 μm in specimen C30.

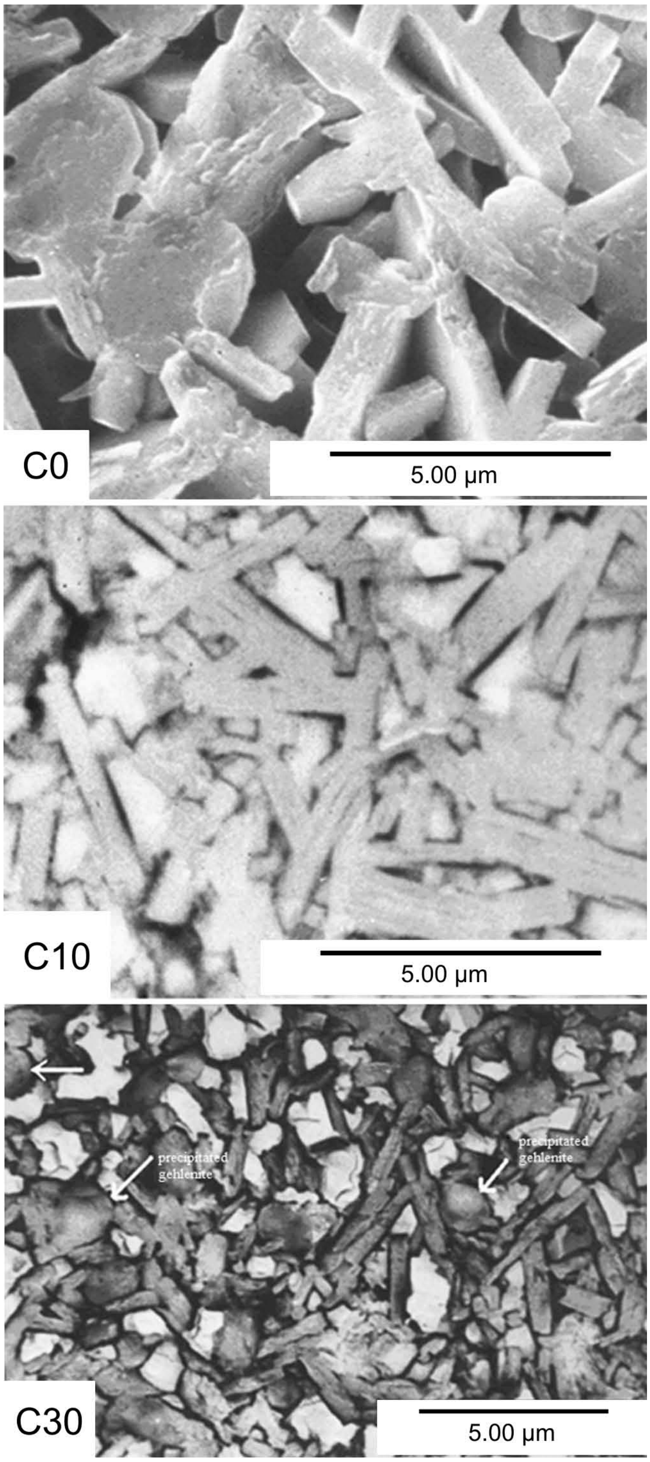

Fig. 2: Microstructures of specimens C0, C10 and C30 after sintering at their optimum temperatures for 2 h.

It has been proven by means of energy-dispersive X-ray analysis (EDX) that a small amount of calcium could be present in the fluorphlogopite particles [19]. It seems that Ca ions can be substituted for K ions in the potassium fluorphlogopite host structure, forming a solid solution. Ca ions owing to their smaller size and higher charge (larger ionic field strength) in comparison with K^+ ions could strengthen the cleavage bonding of mica structure. Hence, this substitution apparently improved the bending strength of glass-ceramic specimens [20]. In addition, the particles of gehlenite were precipitated in the fully densified specimens C20 and C30. Some of these particles are marked by arrows in Fig. 2, C30. Gehlenite has a higher elastic modulus than fluorphlogopite and it can therefore contribute to the strengthening process of specimens C20 and C30, through the well-known mechanisms such as crack bowing, crack pinning [21, 22] and crack deflection [23]. Unfortunately, experimental verification of the exact strengthening mechanism was not possible owing to the flexibility of specimens as this did not allow the detection of the trace of induced advancing cracks initiated by the Vickers indenter. Concerning the fracture toughness of the glass-ceramics, a rapid increase in its value from 1.7 to 2.2 MPa·m$^{1/2}$ was observed when the CaO reached 20 weight parts (Fig. 3). This behaviour is completely in accord with the appearance of gehlenite in specimen C20 and shows that bending strength and fracture toughness exhibit parallel trends as expected from the Griffith relationship. This confirms that the crack bowing, pinning and/or crack deflection mechanisms are actually operative in the gehlenite-containing specimens.

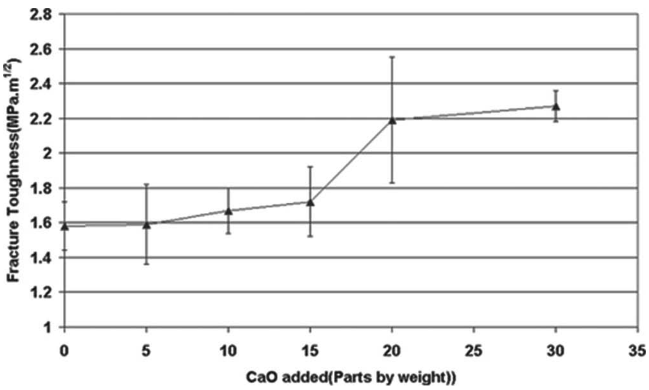

Fig. 3: Fracture toughness of specimens as a function of CaO.

Fig. 4 shows the Vickers microhardness of various glasses and glass-ceramics as a function of their CaO content. It can be seen that the variation of hardness values in the glass specimens is almost negligible compared with the glass-ceramic specimens. In the glass-ceramics, specimen C0 shows the lowest hardness value. With the gradual increase of CaO to 20 weight parts, the hardness value reaches its maximum value of 7.8 GPa. With further addition of CaO, i.e. 30 weight parts, the microhardness value falls to 2.5 GPa. In addition, the hardness of glass-ceramics, with exception of specimen C20, is noticeably lower than that of their corresponding glasses. It is well-known that contrary to other glass-ceramic systems, precipitation of fluorphlogopite decreases the hardness of specimens [24]. Thus, higher amounts of fluorphlogopite in the specimen should lead to the reduction of its hardness and vice versa. As mentioned previously, the amount of fluorphlogopite in the present glass-ceramics was decreased with the addition of CaO [16]. Accordingly, it was expected that the Vickers microhardness values of the glass-ceramics would gradually rise with the increase of their CaO content. Apart from specimen C30, containing the lowest amount of mica and the highest amount of gehlenite, the other glass-ceramics followed the expected trend. It seems that the presence of a CaO-rich, low-strength residual glass phase in specimen C30 is perhaps responsible for this behavior.

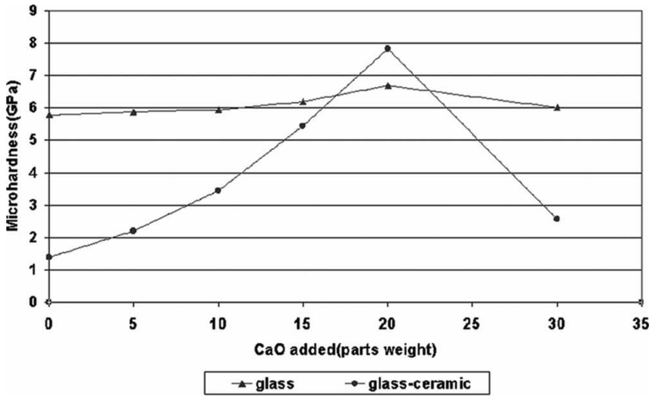

Fig. 4: Microhardness of glass and sintered glass-ceramics as a function of CaO.

(2) Machinability

Machinability is a much maligned term which has many different meanings but generally refers to the ease with which a material can be machined to an acceptable surface finish. The principal definitions of the term are entirely different: the first based on material properties, the second based on tool life, and the third based on cutting speed. Neglecting the tool life and considering a constant cutting speed, in the present work machinability refers only to material properties like microhardness, fracture toughness and associated parameters like brittleness and n factors. In general, the higher the microhardness is, the more difficult the material is to machine, requiring greater forces and lower speeds. A high applied force can lead to cracking, chipping of the drilled parts or an unacceptable surface finish.

Table 2 lists different parameters that affect machinability. A high hardness (H_v) value means a lower rate in the displacement of material during machining [19]. According to this criterion, the best machinability should be exhibited by specimen C0 as this exhibits the lowest microhardness value.

Table 2: Selected machinability criteria for the glass-ceramics.

Sample	C0	C5	C10	C15	C20	C30
H_V (GPa)	1.4	2.2	3.4	5.3	7.8	2.5
B [$(1/\mu m)^{1/2}$]	0.88	1.38	2.06	3.16	3.57	1.13
n	0.47	0.37	0.22	-0.02	-0.3	0.33

Lawn and Marshall [18] have also introduced a quantitative brittleness factor (B), which can be obtained experimentally by dividing the microhardness value by the critical stress intensity factor (K_{IC}) of the specimen:

$$B = H_v/K_{IC}$$

A lower brittleness factor means lower probability of catastrophic crack propagation in a specimen during machining. Based on this criterion (Table 2), specimen C0 is again the best machinable glass-ceramic and specimen C30 occupies the second position in the ranking.

Another parameter introduced for quantitative measurement of machinability is the n factor. It is defined by the following equation [19]:

$$n = 0.643 - 0.122H_V$$

It is claimed that a specimen can be easily machined if it has a positive n value and cannot be easily machined if it has a negative n. Accordingly, the results of Table 2 show that specimens C0 and C20 are the best and the worst machinable glass-ceramics, respectively, and specimen C30 can be considered as a good specimen in this regard.

Based on the above-mentioned results, it seems more convenient to take just the hardness value as the most suitable parameter for assessment of machinability.

According to the results of machining experiments, specimen C30, which had the highest bending strength and fracture toughness values, unlike specimen C15 does not show any signs of edge damage in its drilled surface; hence, it can be considered as possessing better machinability than specimen C15. It should be mentioned that under the selected drilling conditions, specimen C20 was broken during machining.

IV. Conclusions

According to the results of this work it can be concluded that the addition of CaO led to the gradual enhancement of both the bending strength and fracture toughness of sintered glass-ceramics. The reduction of mica crystal sizes and precipitation of gehlenite as a new crystalline phase possessing high mechanical strength were found to be responsible for this observation.

Among the various criteria, the microhardness of the specimens was more compatible with the naked eye observation of machinability. According to the results, while the addition of 15 and 20 weight parts of CaO led to an enhancement in microhardness, which is not preferable regarding machinability, the addition of 30 weight parts of the latter oxide resulted in good machinability as well as the highest bending strength and fracture toughness values.

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