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Sintering behaviour and mechanical properties of Al_2O_3 platelet-reinforced glass matrix composites obtained by powder technology

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Abstract

A cold-pressing and pressure-less viscous flow sintering treatment for the manufacturing of dense alumina platelet-reinforced glass matrix composites was investigated. The sintering behaviour of mixtures of powdered borosilicate glass cullet, coming from the pharmaceutical industry, and Al_2O_3 platelets was studied respecting the retardation in sintering caused by the introduction in glass of rigid, non-sintering, inclusions. Despite the relatively low Al_2O_3 platelet addition significant increases in bending strength, microhardness and, above all, fracture toughness were achieved, due to the homogeneity of mechanical dispersion of platelets and the development of residual stresses, caused by the thermo-elastic mismatch between the constituent phases. A crack deflection toughening mechanism, due to the specific reinforcement, was found to be effective.

Keywords: D. Glass; Ceramic matrix composites

1. Introduction

Composite materials result from the joining of two (or more) different materials. While in the polymeric matrix composites the reinforcement is needed to improve the mechanical strength and stiffness of the un-reinforced matrix, in the glass matrix composites the reinforcement is needed mainly to modify the fracture behaviour, causing the composite to be less brittle than the parent glasses, with secondary effects such as improvements in hardness, wear resistance, or mechanical strength at high temperatures [1,2]. The enhancement of fracture toughness, in relation to the un-reinforced matrix, is due to a complex of matrix/reinforcement interactions, which causes cracks to deviate or branch, with a certain fracture energy absorption. Fibre-reinforced glasses, in which matrix/reinforcement interactions cause the maximum energy absorption, show excellent bending strength and fracture toughness, but they are available mainly by expensive hot-pressing techniques [3]; in particulate-reinforced composites the toughness enhancement is undoubtedly less substantial, but their manufacture is notably easier. Significant experiences have been conducted on the reinforcement of glass (and glass-ceramics) with ceramic particles [4]. In this case, the improvement in mechanical properties is mainly related to crack deflection, caused by the introduction of compressive residual stresses in the matrix, due to the thermal expansion mismatch between the phases. Crack deflection, as a toughening effect, is strongly dependent on the aspect ratio (ratio of length to width) of the reinforcing phase [2]. Ceramic whiskers, characterised by a huge aspect ratio, show excellent toughening contributions. The use of whiskers is currently limited because of high costs, toxicity by inhalation and difficulties in homogenising within the matrix. Ceramic platelets (SiC [5,6] and Al_2O_3 [7,8]) have been used successfully to reinforce glass and glass-matrix composites for the last 10 years, with extensive applications in ceramic matrices such as mullite, zirconia and alumina [9–11]. The toughening effect, even if inferior than that of whiskers, is much more substantial than that corresponding to equiaxed par-

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ticles. Such reinforcements, besides environmental safety, are of low-cost and are commercially available as abrasive materials for polishing applications, since the polishing operation is highly effective with angular particles [12]; extensive application as fillers in polymers, in order to improve the thermal conductivity, are also reported, since platelet-shaped particles provide a great deal of surface contact, thus forming chains along which heat may be efficiently transferred [13]. Some work has been performed on manufacturing platelet-reinforced glass-matrix composites by cold-pressing and pressure-less viscous flow sintering, thus yielding a cost effective processing route [14].

Since the 1960s several studies have been conducted in manufacturing innovative glass-based materials from the treatment of silicate wastes. Glass-ceramics [15,16], glass fibres [17], foam glasses [18,19] and, mainly in the last 10 years, glass or glass-ceramic matrix composites [14,20–22] were developed with this aim. Any acceptable process should be characterised by significant production economics. As a consequence expensive hot-pressing techniques cannot be employed. The main objective of this work was to optimise the sintering behaviour of Al_2O_3 platelet-reinforced recycled glass, in order to reach high density products, with suitable mechanical properties, especially in relation to fracture toughness, and with a short-time procedure.

2. Experimental

Borosilicate recycled glass cullet (Kimble/Kontes, Vineland, NJ) was employed as the matrix. This type of glass is currently intended for pharmaceutical applications. Chemical composition and physical properties of the investigated glass are shown in Table 1.

A dilatometric analysis was performed in order to determine the dilatometric softening point, essential for the study of the sintering behaviour. From the dilatometric plot, which is shown in Fig. 1, we can infer that the dilatometric softening point is at about 650 °C. Differential thermal analysis was used to study the crystallisation behaviour of borosilicate glass; a DTA plot, as illustrated in Fig. 2, shows a moderate crystallisation exothermic peak at 785 °C.

Table 1
Chemical composition and physical properties of the investigated borosilicate glass

Chemical composition (wt.%)	
SiO_2	72
B_2O_3	12
Al_2O_3	7
CaO	1
Na_2O	6
K_2O	2
BaO	<0.1
Physical properties	
Working point (°C)	1140
Annealing point (°C)	570
Strain point (°C)	530
Softening point (°C)	785
Thermal expansion coefficient ($°C^{-1}$)	5.5×10^{-6}
Density (g/cm^3)	2.33

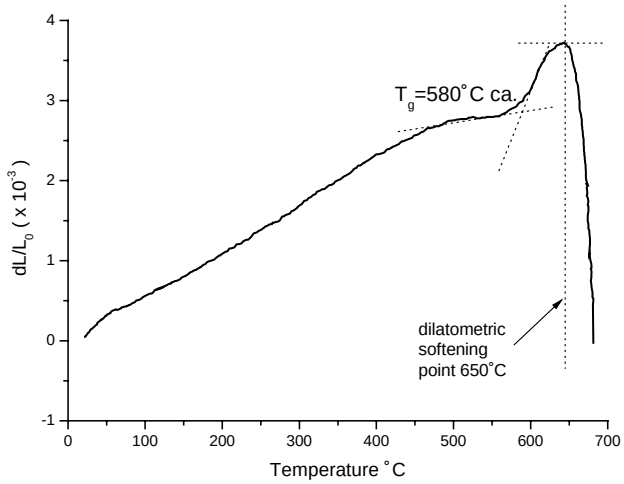

Fig. 1. Dilatometric plot of the investigated borosilicate glass.

Glass cullet was first dry ball milled and sized in order to obtain grains <37 μm, then it was ground in a tungsten carbide vibratory mill for 15 min, producing fine glass particles <20 μm.

α-Alumina platelets were chosen as the reinforcement (Advanced Nano Technology Pty. Ltd., Welshpool, WA, Australia). The platelets are hexagonal-shaped, with major axes between 2 and 5 mm and thickness <0.2 μm (axial ratio < 0.1). The extraordinary thinness of the reinforcement is due to the mechano-chemical process adopted by the manufacturers (platelets are synthesised by high energy milling of suitable precursors), instead of the extensively reported [13] synthesis by calcination of alumina fluoride precursor, obtained by chemical reaction between boehmite (aluminium hydroxide) and hydrofluoric acid. The typical platelet morphology is shown in Fig. 3.

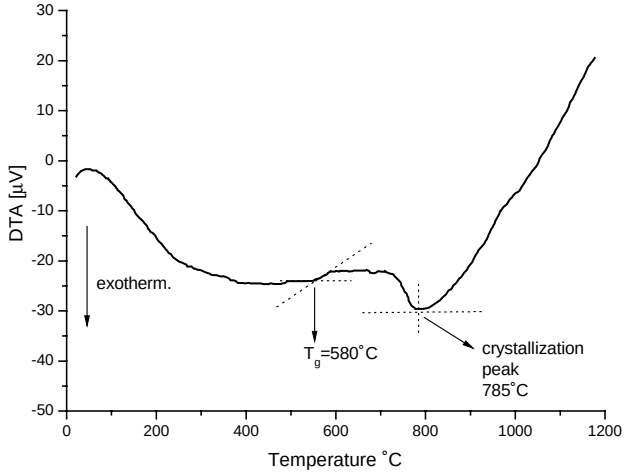

Fig. 2. DTA plot of the investigated borosilicate glass, showing a slight crystallization peak at 785 °C.

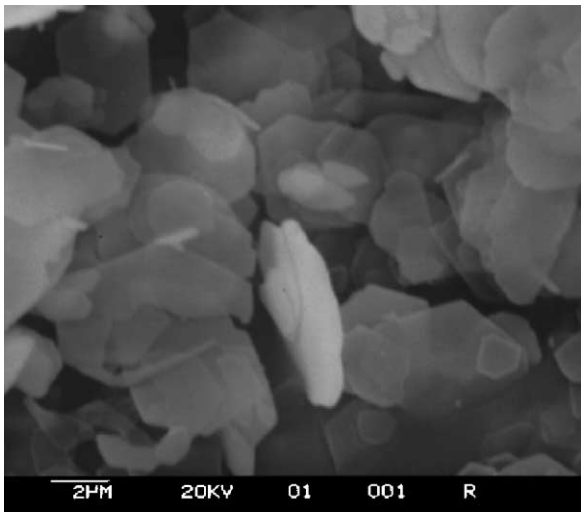

Fig. 3. SEM micrograph of Al_2O_3 platelets used as the reinforcing phase.

Fine glass powders were blended with alumina platelets in 5, 10 and 15% concentrations by volume, and mixed in a dry ball mill for 1 h. Powders of pure matrix were also studied. The powders were treated with a small amount of *n*-butyl-acetate and uniaxially pressed in a steel die of rectangular section (50 mm × 34 mm) at room temperature, by using an hydraulic press operating at 40 MPa. Green tiles were sintered in air at temperatures varying from 700 to 850 °C. The heating rate was 5 °C/min and the sintering time 1 h for all samples. The use of a small quantity of *n*-butyl-acetate is essential for binding the powders and for minimising the formation of cracks in the green tiles, which could be detrimental to the mechanical properties of sintered products. The use of similar organic binders has been reported in the literature [23,24].

The thermal expansion coefficient of alumina platelets ($\alpha_p = 8.9 \times 10^{-6}\,^{\circ}C^{-1}$) is higher than that of borosilicate glass matrix ($\alpha_m = 5.5 \times 10^{-6}\,^{\circ}C^{-1}$), thus causing the development of tangential compressive and radial tensile stresses in the matrix around the reinforcement upon cooling from the sintering temperature. A certain crack deflection effect is expected.

The density of the sintered compacts was measured by the Archimedes' principle. At least 10 fragments were analysed for each sample. The theoretical densities of composite materials were calculated from their compositions and from the density of the constituents, following the rule of mixtures. The ratio measured density/theoretical density was chosen as a reference parameter.

Beam samples of about 3 mm × 2 mm × 42 mm, for bending strength (modulus of rupture) determinations were cut from sintered samples. All samples were carefully polished to a 6 μm finish, by using abrasive papers and diamond paste. The edges of the bars were bevelled by using fine abrasive papers and diamond paste. Four point bending tests (32 mm outer span, 8 mm inner span) were performed by using an Instron 1121 UTS (Instron Danvers, MA), with a crosshead speed of 0.1 mm/min. Each data point represents the average of 5–10 individual tests.

Polished samples were employed for Vickers indentation tests, which yielded the hardness (H_v), at low load (500*g*) and the indentation fracture toughness (K_{IC}), at high load (2000*g*), of the investigated materials. The fracture toughness was calculated by using the well known equation of Anstis et al. [25], starting from the measured length of cracks emanating from the corners of the Vickers indents. The reference Young's modulus for fracture toughness determination by using the equation of Anstis et al. was the Young's modulus of the glass matrix, for all samples, thus neglecting the gain in elastic modulus provided by the inclusions (the Young's modulus of alumina is much larger than that of glass), due to relatively low volume fraction of reinforcement (maximum at 15%, significant elastic modulus enhancements being reported in literature for 30% concentration, with composites manufactured by hot-pressing [7,8]); the K_{IC} calculations for glass matrix composites were consequently underestimated.

The fracture surfaces of sintered samples were characterised by scanning electron microscopy. Powdered samples were investigated by X-ray diffraction.

3. Results and discussion

The initial phase of this work was related to the optimisation of viscous flow pressure-less sintering of glass and glass matrix composites. Recent works reported that minimum temperature for optimum viscous flow sintering of glass is the dilatometric softening temperature, at which the contraction of a sample due to viscous flow is exactly counterbalanced by the thermal expansion, while the maximum temperature is the Littleton's softening temperature, at which gross viscous flow appears. An optimum sintering temperature could be estimated as 50 °C above the dilatometric softening temperature [26]. From Fig. 1 we can infer that the dilatometric sintering temperature is about 650 °C, so that sintering of glass, with an high densification, had to be performed at least at 700 °C. It must be remembered that viscous flow may be hindered by crystallisation, when the sintering temperature approaches the temperature of crystallization exothermic peak (which is at about 785 °C for the investigated glass) and is maintained for a suitable time. Borosilicate glasses may show significant crystallization, so that short time thermal processes are needed; Banuprakash et al. [27] reported rapid hot-pressing cycles for the densification of Cu-reinforced Pyrex glass in order to avoid large cristobalite precipitation. The investigated borosilicate glass is characterised by a slight crystallization exothermic peak, and the chosen firing time is relatively short. Preliminary tests on un-reinforced glass demonstrated that complete densification could be obtained at 700 °C; sintered glass, as shown in Fig. 4, is X-ray amorphous. The absence of devitrification of the investigated glass is thought to be due to the

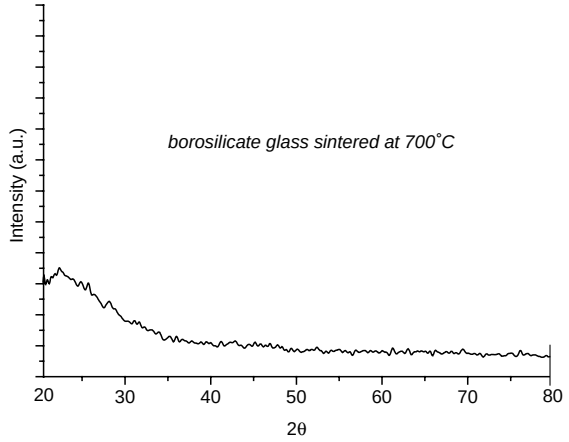

Fig. 4. XRD spectrum of sintered borosilicate glass (700 °C).

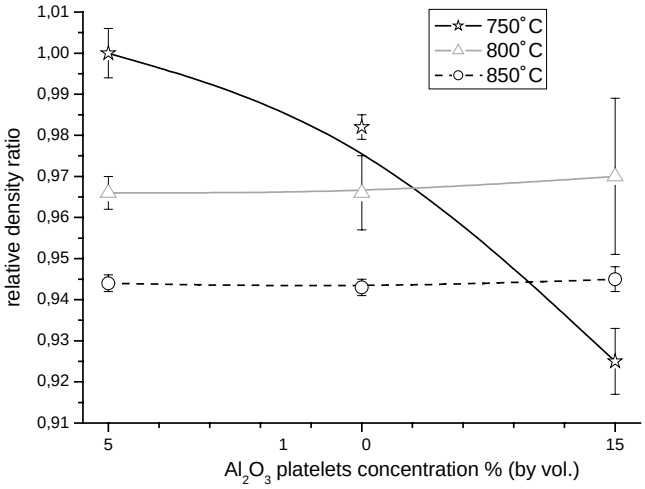

Fig. 5. Densification behaviour of Al_2O_3 platelet-reinforced glass matrix composites at different sintering temperatures.

relevant Al_2O_3 content in the composition; recent studies have illustrated the role of alumina (or zirconia) as inhibitor of crystallization during sintering of borosilicate glass [28].

Sintering behaviour of glass matrix composites may be affected, in addition to crystallization of glass, by the reinforcement. The presence of rigid inclusions causes the densification to be retarded, as the "sintering mass" is lowered (the non-sintering reinforcement "dilutes" glass) and the effective viscosity of the system is much superior than that of the matrix alone [29–31]. The reinforcement decreases the viscous flow ability of glass deeply depending on its aspect ratio [32]. The maximum reinforcement concentration (15%) in this work is thought to be the upper limit for satisfactory densification of platelet-reinforced glass matrix composites by pressure-less sintering. Higher reinforcement volume fractions or higher aspect ratios (e.g. operating with whiskers) would lead to the need for hot-pressing devices. In order to balance the retardation in sintering caused by the reinforcements the sintering temperatures for glass matrix composites were higher than that of un-reinforced matrix, varying from 750 to 850 °C. As reported before, the ratio measured density/theoretical density (i.e., relative sintered density) was chosen as a reference parameter. The theoretical densities of composite materials were calculated from the rule of mixtures. The densification behaviour is summarised in Fig. 5.

Composites obtained by sintering at 750 °C resulted in high compaction degree for low concentrations (5 and 10 vol.%), while for upper concentrations (15 vol.%) the porosity content is relevant; this is consistent with the assumption of a decreasing viscous flow ability of glass with increasing concentration of inclusions. Composites obtained at 800 and 850 °C showed an anomalous sintering behaviour. Composites obtained at 800 °C were all characterised by high relative density, corresponding to a porosity content of about 3%. In this case even with relatively high reinforcement content the densification is found to be satisfactory. For all concentrations of the composites obtained at 850 °C the relative density is lower than 0.95 (corresponding to a porosity >5%). The sintering behaviour at 800 and 850 °C contrasts with the decreasing viscosity of the system, which should cause higher densification (for a 5% alumina content a full densification is achieved at 750 °C, while at higher temperatures the densification is not complete). The fact that for every concentration the porosity content (<3% at 800 °C, >5% at 850 °C) is almost constant suggests that the sintering behaviour is influenced not only by the amount of non-sintering inclusions but also by secondary effects, which are independent from the presence of inclusions, and depend on the manufacturing process. One likely secondary effect is that, in composites obtained at 800 and 850 °C, a more rapid sintering occurs at the surface (nearest to the heating elements) with entrapment of gas, for example due to the decomposition of the binder, in the core. In the composites obtained at 750 °C, the sintering of

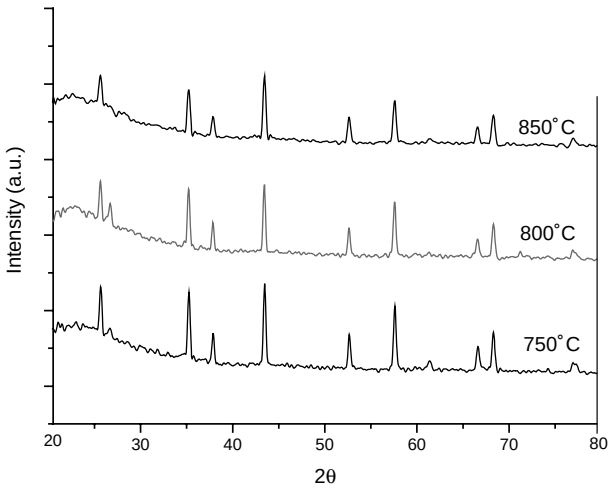

Fig. 6. XRD spectra of sintered Al_2O_3 platelet-reinforced glass matrix composites (15% Al_2O_3 content) showing α-Al_2O_3 as the only crystalline phase.

Table 2
Bending strength of Al_2O_3 platelet-reinforced glass matrix composites sintered at different temperatures

Modulus of rupture (MPa)	Al_2O_3 (%)			
	700 °C	750 °C	800 °C	850 °C
0	76.31 ± 15.70			
5		93.32 ± 4.58	77.79 ± 12.05	62.54 ± 9.83
10		96.91 ± 10.04	95.35 ± 7.41	79.89 ± 10.06
15		109.30 ± 8.30	122.19 ± 15.90	100.27 ± 13.73

glass is thought to be relatively slow, so that gasses evolve before definitive densification. In such composites, as a consequence, only the amount of non-sintering inclusions is found to be effective. In the composites obtained at 800 °C, the sintering ability of the system is elevated, so that at 15% concentration of alumina platelets the densification is satisfactory, while at 5 and 10% there is no improvement. Eight hundred degree celcius (since it causes the highest densification for the maximum concentration of alumina platelets) is thought to be a suitable temperature for the densification of the whole range of compositions: the relative density would approach 1 for all the compositions but it is shifted to a lower value due to the probable presence of organic residues. In the composites obtained at 850 °C the lacking of pore evolution in the composites causes the shifting to be more drastic.

Besides crystalline peaks due to the alumina platelet addition, the glass matrix composites are X-ray amorphous at all sintering temperatures, as shown in Fig. 6; this is consistent with findings in the literature illustrating the inhibiting ability of alumina with respect to crystallization of glass, not only in the formation of glass but also in the manufacturing of glass/alumina compacts [33]. The above reported anomalous sintering behaviour is not affected by any crystallization phenomenon.

The bending strength of composites was found to increase with increasing platelet content. With a volume fraction of 15%, platelets caused a bending strength enhancement greater than 50%. Even with smallest volume fraction, 5%, the bending strength enhancement may be notable: in the case of sintering at 750 °C, with almost complete densification, modulus of rupture was higher than 95 MPa. Bending strength data are summarised in Table 2. The influence of platelet content on mechanical resistance of glass matrix composites may become more evident from the plot bending strength versus relative sintered density, which is shown in Fig. 7. Even if glass matrix composites were rarely characterised by complete densification, these composites showed superior bending strength.

Like bending strength, Vickers' hardness of glass matrix composites, illustrated in Fig. 8, showed a certain dependence on residual porosity. As expected, hardness in composites increases with increasing platelet content (alumina is much harder than glass), but the enhancement may be reduced by porosity; samples sintered at 850 °C showed a slight increase, while samples with almost complete densification (5 and 10% at 750 °C and 15% at 800 °C) showed the highest values, which exceed those reported in literature, especially for cold-pressing and sintering. For the most highly dense composites' hardness is found to increase almost linearly with alumina content. The achieved hardness enhancement by platelets addition makes glass matrix composites suitable for applications requiring good wear resistance.

Indentation fracture toughness determinations, which are illustrated in Fig. 9, were conducted on the most dense

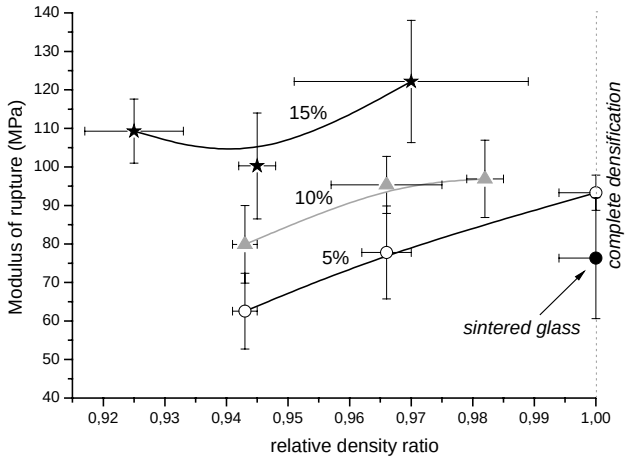

Fig. 7. Modulus of rupture of glass matrix composites in function of the relative density ratio. The lines are reported as a guide for the eye.

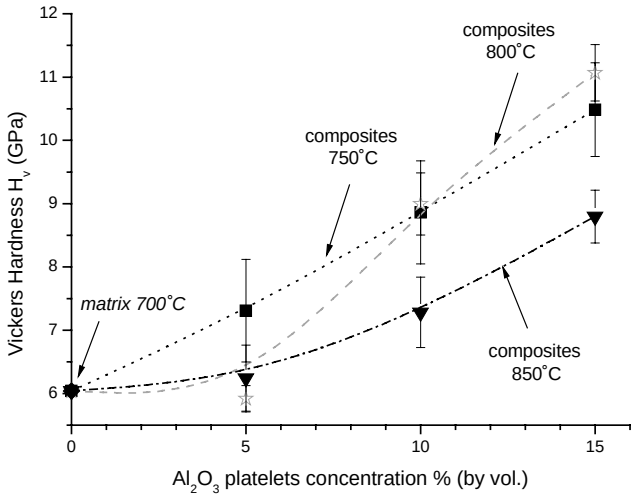

Fig. 8. Vickers hardness increase with increasing Al_2O_3 platelet concentration in the investigated glass-matrix composites.

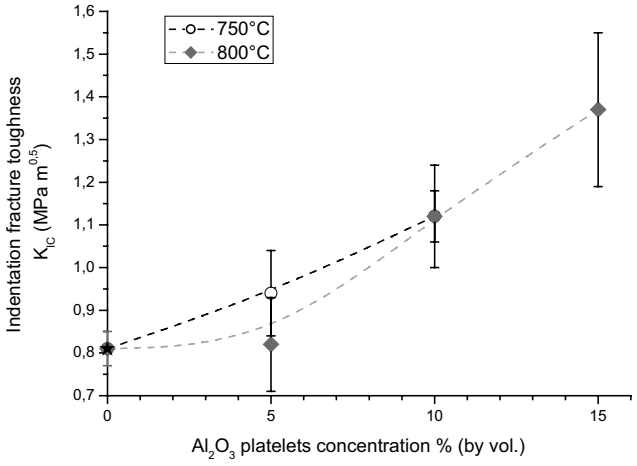

Fig. 9. Indentation fracture toughness increase with increasing Al_2O_3 platelet concentration in the investigated glass-matrix composites (samples exhibiting highest densification).

composites. Even K_{IC} exhibited an increasing trend with increasing alumina content. The obtained values agreed well with those reported for similar composites developed by hot-pressing [7,8], although, as mentioned above, they may be underestimated. The value for 15% reinforcement (at 800 °C), 1.369 MPa m$^{1/2}$, is thought to be promising. A crack deflection toughening was found to be effective; this causes the fracture surfaces to be extremely rough in relation to that of parent glass, since cracks deviate around the reinforcement, as illustrated in the fracture surfaces in Fig. 10, in good agreement with the findings in the literature [34].

In order to reveal the homogeneity of the reinforcement caused by mechanical mixing, a hydrofluoric acid etching of polished surfaces was performed (aqueous solution of HF 5 vol.%, 2 min lasting exposure). Fig. 11 illustrates that a good homogeneity of platelet dispersion was achieved, with no anisotropic orientation; as mentioned above, platelets are hexagonal-shaped and notably thin. The low aspect ratio is

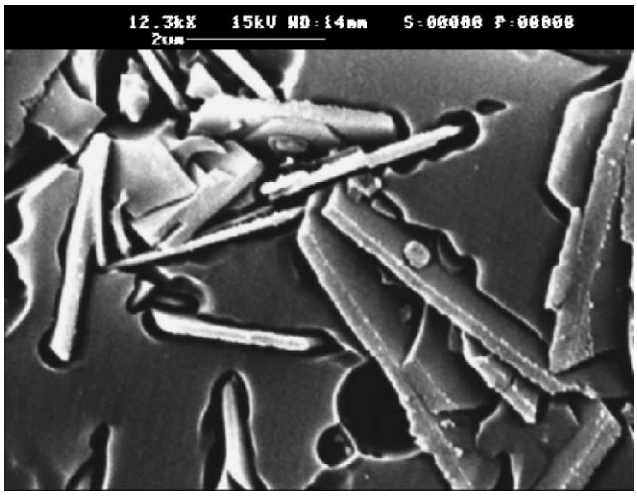

Fig. 11. SEM micrograph of the surface of a sintered Al_2O_3 platelet-reinforced glass matrix composite (15% Al_2O_3 at 800 °C) after HF etching, revealing platelets distribution.

thought to be the reason of the relevant increase in bending strength, hardness and fracture toughness, despite residual porosity typical of cold-pressing and viscous flow sintering treatment. The observed mechanical properties suggest that the investigated glass matrix composites may find technical applications in the field of building and chemical industry.

4. Conclusion

A cold-pressing and pressure-less sintering treatment for the manufacturing of highly dense alumina platelet-reinforced glass matrix composites was optimised. The sintering temperature had to be suited to the amount of platelet addition. The sintering of the un-reinforced matrix was performed at 700 °C, a temperature which is found to be suitable for high densification without gross viscous flow.

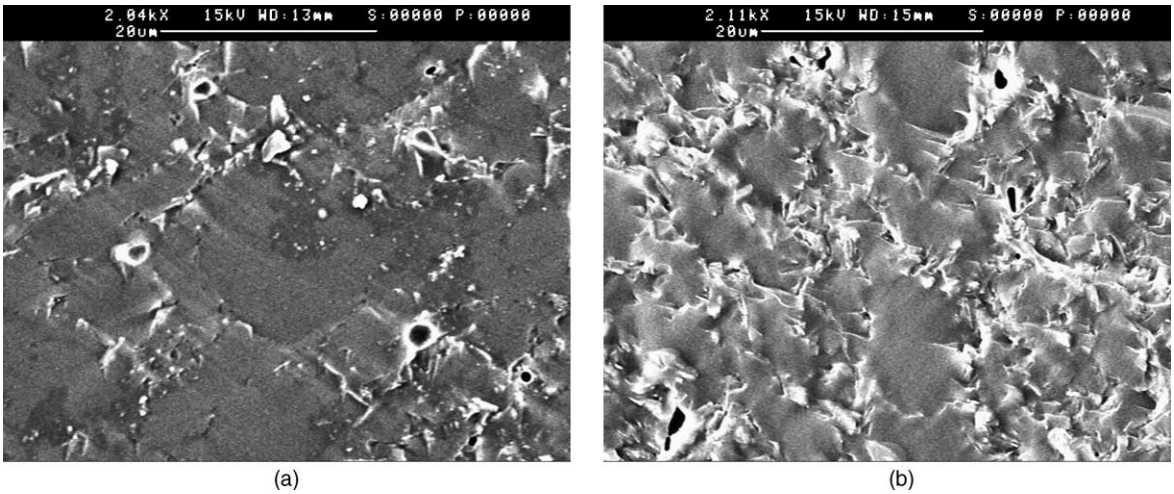

Fig. 10. SEM micrographs of the fracture surface of sintered Al_2O_3 platelet-reinforced glass matrix composites: (a) 5% Al_2O_3 at 750 °C; (b) 15% Al_2O_3 at 800 °C.

Glass matrix composites had to be developed at temperatures from 50 to 100 °C exceeding that of the un-reinforced matrix, due to the retardation in sintering caused by the introduction of rigid, non-sintering, inclusions. Good mechanical properties, consisting of bending strength, microhardness and particularly fracture toughness, were achieved in spite of the relatively low Al_2O_3 platelet addition to powdered borosilicate glass cullet and the simple and cost effective processing route; consequently, applications in the field of building and chemical industry may be suggested. The illustrated approach could be useful to the manufacturing of a number of similar glass matrix composites from several waste glasses.

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