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Dense Zirconia–SiC Platelet Composites made by Pressureless Sintering and Hot Pressing

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Abstract

2·6 mol% Y-TZP composites with up to 30 vol% SiC platelet additions were fabricated by hot pressing and pressureless sintering to densities above 93%. Borosilicate glass additions aided densification, reduced the thermal mismatch stress between the matrix and the platelets, and reduced grain growth rates in the mildly reducing atmosphere employed. Strength was reduced (up to 50% in hot-pressed material and 60% in pressureless-sintered composites) by the addition of up to 20 vol% platelets as a result of increased flaw size and tensile thermal mismatch stresses in the matrix. With a matrix grain size of 0·71 μm, platelets increased toughness (from 7·0 to 8·3 MPa √m for a 30 vol% addition) while the amount of transformation decreased from 33 to 22%, demonstrating that mechanisms such as crack deflection were present. Conversely, where the composite was processed to give a matrix grain size which optimised transformation toughening (≥0·85 μm), the addition of the platelets led to a reduction in toughness (from 14·0 to 8·8 MPa √m for a 30 vol% addition). The volume fraction tetragonal which transformed during fracture to monoclinic zirconia decreased with platelet addition, for all processing conditions. The composite toughness was always lower than the maximum in the monolith arising from transformation toughening alone, due to a low fracture stress in the composite and modulus load transfer, both of which reduce the transformation zone size. © 1997 Elsevier Science Limited.

1 Introduction

Transformation toughening is ineffective above the stress-induced martensite start temperature, but the addition of platelets or whiskers to a zirconia matrix can increase high-temperature toughness and strength through mechanisms such as crack deflection. For example, Claussen *et al.*[1] obtained twice the strength in a 30 vol% whisker-TZP (tetragonal zirconia polycrystals) composite compared to the monolith at 1000°C.

The effect of a reinforcement on strength and toughness of zirconia-based composites depends strongly on the matrix grain size. In the flaw size controlled regime, platelet or whisker additions can increase toughness.[1,2] However, composite strength can be severely reduced, attributed to an increase in flaw size, particularly if agglomerated reinforcements are present. In contrast, the toughest monolithic zirconias have strengths which are limited by transformation-induced microcracking. For these ceramics, toughness may decrease with reinforcement addition, although strength may be increased.[2]

A number of toughening mechanisms have been identified in ceramic–ceramic composites, including crack deflection,[3–6] crack bridging and pull-out,[6–11] stress-induced microcracking[12] and modulus load transfer at the crack tip.[2] Of these, crack deflection and modulus load transfer are believed to operate in platelet composites,[2,13] although crack bridging and pull-out may also occur.[14] Toughening due to the platelets depends on the bonding between reinforcement and matrix,[14] the aspect ratio[13] and the presence of preferred orientation.[13,15] The contribution arising from modulus load transfer is not clear. Load transfer dissipates the crack tip stresses by spreading the stress over a larger volume[2] and this may also increase the transformation zone size and therefore further increase toughness.

Key factors determining composite properties are the sign, magnitude and location of residual stresses induced by thermal mismatch between matrix and reinforcement. Zirconia has a high thermal expansion coefficient ($\alpha ZrO_2 = 10 \times 10^{-6}°C^{-1}$ from 0 to 1000°C) compared to SiC ($\alpha = 4{\cdot}7 \times 10^{-6}°C^{-1}$, 0–1500°C) so that the matrix is always under tensile stress. These stresses alter the matrix transformability and the extent of reinforcement debonding. In the present work, borosilicate glass

was added in order to reduce the magnitude of the tensile stresses in the matrix. Borosilicate glass has a low thermal expansion coefficient (α = 2·5–3·5 × $10^{-6}°C^{-1}$ for 0–1000°C) which will partly offset the high thermal expansion coefficient of the zirconia. Moreover, the elastic strain will only start to form below the T_g of the glass on cooling from sintering. While borosilicate glass additions do not leach yttria stabiliser from the zirconia,[16–18] they may lead to formation of $ZrSiO_4$. Although this $ZrSiO_4$ will reduce the ZrO_2 fraction available for transformation, it has a lower thermal expansion coefficient than ZrO_2 ($\alpha ZrSiO_4$ = 6 × $10^{-6}°C^{-1}$ for 0–1500°C) and will therefore further reduce matrix tensile stresses.

The current work provides an investigation into the zirconia–SiC platelet system in order to investigate whether platelets can increase toughness to the same extent as whiskers and to establish whether the additive effect of transformation toughening and crack deflection toughening processes can result in a ceramic with a higher toughness than achievable from transformation toughening alone.

2 Experimental

2·6 mol% yttria-TZP (Y-TZP) powders (Dai-ichi, Japan) with an average surface area of 4·0 m^2/g (measured by BET nitrogen absorption) were used as the matrix materials throughout. α-SiC platelets supplied by Newmet Ltd were used. Coarse and agglomerated particles were removed by sedimentation to obtain platelets of 5–70 μm wide by 0·5–5 μm thick. Amorphous borosilicate (denoted BS) glass powder was obtained by milling Pyrex™ glass (with a composition (wt%) of 80 SiO_2, 13·4 B_2O_3, Na_2O, 0·5 K_2O and 2·5 Al_2O_3) to give a mean particle size after sedimentation of 0·64 ± 0·14 μm (one standard deviation), determined using a laser particle size analyser (Coulter LS130 laser interferometer). TZP ceramics were processed in the as-received state and with a 1·5 wt% glass addition made to the TZP powder. In addition, amorphous colloidal silica (mean particle size 130 ± 25 nm) was added to a TZP powder to examine the effect of the silica on grain growth kinetics. The mechanical properties and microstructure of silica-containing materials are not reported here.

All starting materials resisted sedimentation and agglomeration when suspended in distilled water with an NaOH addition to raise the pH to 10. Mixing of suspensions containing individual components was undertaken with vigorous stirring for 24 h using a magnetic stirrer. The resulting slip was then filter pressed under uniaxial loading at 200 MPa. The 30 mm diameter by 8 mm thick green compacts of approximately 62% theoretical density were pressureless sintered at 1500°C for up to 6 h using a heating and cooling rate of 2 K/min. To prevent oxidation of the SiC platelets, mildly reducing conditions were induced by packing the green compact in a controlled mixture of carbon granules and coarse calcia-stabilised zirconia powder. No evidence of calcia contamination could be found by energy dispersive spectroscopy (EDS), but all testing and characterisation of the composites was conducted after removal of ~2 mm of the sintered surface. Monoliths were also sintered in air under otherwise identical conditions in order to evaluate the effect of the mild reducing conditions. Green compacts were hot pressed in a graphite die at 1500°C for 1 to 2 h and a pressure of 25 MPa to produce discs of 30 mm diameter and 5 mm thick. The press was operated in air although the atmosphere can be considered reducing due to the graphite around the sample.

The density of green compacts and sintered samples was measured by Archimedes' mercury displacement. Fracture toughness was evaluated using the indentation technique with a load of 30 kg on a plane perpendicular to the pressing direction. A crack length/indentation load analysis suggested that indentation crack length did not increase for the composites above a load of 20 kg. Loads above 30 kg gave unacceptable levels of spalling. The average toughness and one standard deviation in toughness is based on 15 indentations on each of five specimens. The Young's modulus for composites was calculated assuming a rule of mixtures, taking E = 200 GPa for the monolith and E = 400 GPa for SiC platelets.

Fracture strength was determined using three-point bending at a crosshead speed of 0·1 mm/min. Bars were cut to 30 × 5 × 4 mm and the stress surface, which was perpendicular to the hot pressing axis (i.e. the same orientation as for the toughness measurements), was polished to a 0·25-μm diamond finish. Edges of the bars were chamfered at 45° to a depth of approximately 40 μm in order to prevent preferential edge crack initiation. The average and range of data are presented for five tests for each material.

Phase contents were determined by X-ray diffraction (XRD) and transmission electron microscopy (TEM). Quantification of the monoclinic content from XRD traces was achieved using the Toraya[19] modification to the technique of Garvie and Nicholson.[20] Average grain size measurements and an assessment of platelet distribution were undertaken on polished, thermally

etched surfaces (1500°C for 20 min), both perpendicular and parallel to the filter-pressing direction, using scanning electron microscopy (SEM). The tetragonal zirconia grain size was measured using the linear intercept method taking approximately 100 grains for each datum. Large cubic zirconia grains and SiC platelets were not included in the measurement. On each specimen two areas were measured on each of two perpendicular sections. The average and range of values from five specimens are presented. Detailed microstructural analysis was undertaken using a TEM (Jeol 200CX and Jeol 3010UHR) equipped with EDS. TEM samples were prepared using conventional ultrasonic drilling, mechanical grinding and dimpling followed by argon ion milling to perforation.

3 Results

3.1 Densification

The as-received Y-TZP powder sintered to near theoretical density after 4 h at 1500°C in air. Adding borosilicate glass did not alter the densification characteristics appreciably, although the use of mild reducing conditions did impair sinterability, with a theoretical density of 97·3–97·6% being achieved compared to 98·4–99·5% for identical sintering conditions in air.

Density as a function of SiC platelet addition for all materials containing the BS addition is given in Fig. 1. Near full densification was achieved in hot-pressed samples in less than 1 h, similar densities occurring in samples hot-pressed for 1 or 2 h. The addition of platelets reduced the density of hot-pressed samples, but only by a maximum of 2% theoretical for 30 vol% platelets. This reduction occurred primarily as a result of platelet agglomeration with associated porosity, but also from interfacial cracking between platelet and matrix. In contrast, substantial reductions in matrix density were found in pressureless-sintered samples, with platelet loading above 20 vol% resulting in extensive macrocracking on cooling from the sintering temperature.

3.2 Microstructure

The mild reducing conditions used to produce the pressureless-sintered samples promoted larger tetragonal zirconia grain sizes than for equivalent sintering conditions in air, Table 1. This effect was less pronounced with the borosilicate addition.

The monolithic zirconia with borosilicate hot pressed for 1 h had a finer tetragonal zirconia grain size (0·71 μm) than that hot-pressed for 2 h (0·82 μm). The pressureless sintered monolith with the borosilicate addition had a mean tetragonal zirconia grain size of 0·85 μm. With the exception of the grain size, the microstructure of hot-pressed samples was similar to that of the pressureless-sintered monoliths. The tetragonal zirconia grain size showed a small but reproducible increase with platelet addition ≥20 vol% for equivalent sintering times for the borosilicate-containing materials, Fig. 2. All samples contained a uniform distribution of large cubic zirconia grains and a thin (5–20 nm) film of glassy phase along most grain boundaries, which had the effect of rounding the boundary at triple junctions. In addition, a small

Table 1. Tetragonal zirconia grain size after sintering in air and mild reducing conditions

Sintered at 1500°C, 4 h	*2·6 Y-TZP*	*+ Borosilicate*	*+ Silica*
In air	0·51 μm	0·58 μm	0·55 μm
In C/ZrO_2	0·92 μm	0·73 μm	0·86 μm

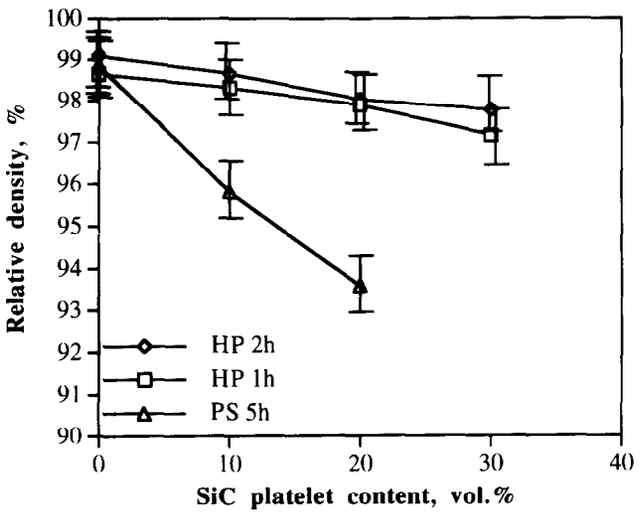


Fig. 1. Relative density as a function of SiC platelet content for hot-pressed (HP) and pressureless-sintered (PS) materials containing borosilicate additions.

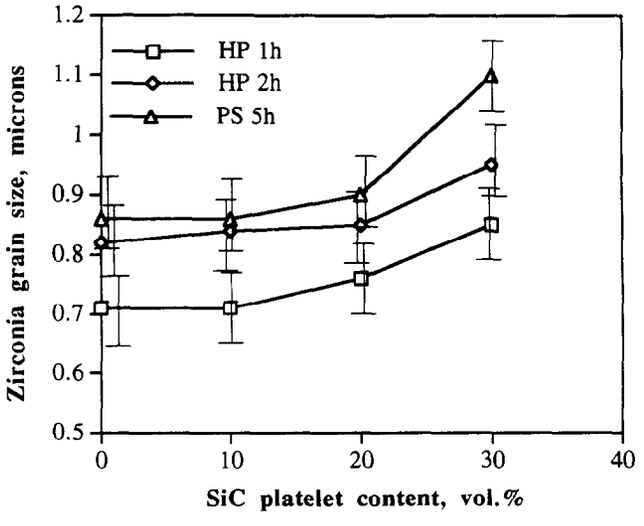


Fig. 2. Zirconia (t + m) grain size as a function of SiC platelet content for pressureless-sintered and hot-pressed materials, all with borosilicate additions.

fraction of $ZrSiO_4$ was present in all samples (confirmed by EDS and electron diffraction studies), promoted by the addition of the borosilicate glass. The level of yttria in the glassy phase was below detectable limits for EDS, suggesting that yttria had not been leached from the tetragonal zirconia. A full description of this microstructure and that formed with different compositions of glass-forming additions is given in an earlier paper.[17]

The SiC platelet size and distribution in the composites were quite uniform in both pressureless-sintered and hot-pressed materials, Fig. 3. Occasional cracking was detected at platelet/matrix interfaces on polished sections. This was more prevalent in the pressureless-sintered materials. The cracks ran along the SiC–glass interface rather than through the glassy phase which covered the majority of interface regions, Fig. 4. Phase separation was observed in the glassy phase adjacent to the platelets, Fig. 4, believed to be due to incorporation of silica from the platelet surface into the borosilicate glass. Some turbostratic carbon was present within the platelets, believed to have originated from SiC platelet synthesis.

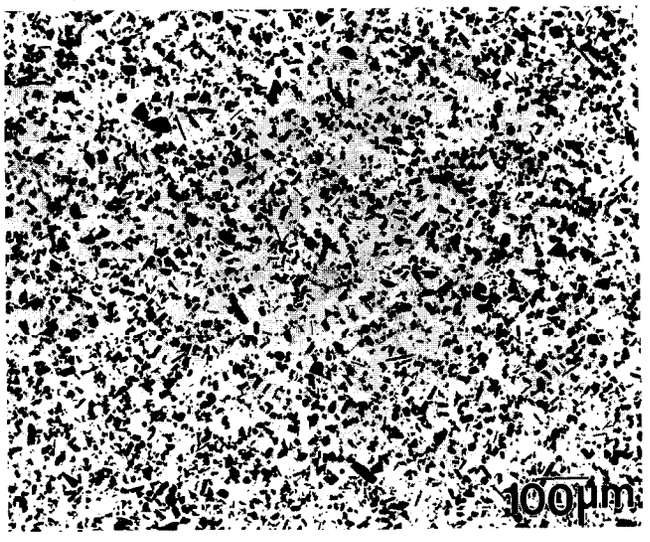


Fig. 3. Scanning electron micrograph of the composite with 30 vol% SiC, hot-pressed at 1500°C for 1 h.

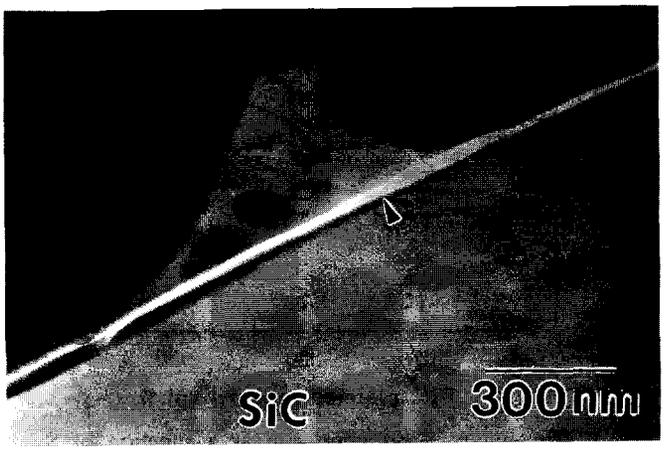


Fig. 4. Bright field TEM image showing cracking (arrowed) at the platelet/matrix interface.

Sections removed parallel to the filter-pressing axis (which was also parallel to the hot-pressing axis) indicated that reorientation of the platelets had occurred, with the long axis of the platelet preferentially perpendicular to the pressing axis. This effect was more marked with hot pressing than pressureless sintering. However, a significant proportion of randomly orientated platelets was present in all samples.

Local clusters of monoclinic zirconia grains were found in the composites, but not the monoliths, frequently located adjacent to platelets. X-ray diffraction indicated that the polished surface monoclinic volume fraction increased with SiC platelets volume fraction for samples containing borosilicate glass for all sintering conditions, Fig. 5.

3.3 Mechanical properties

The fracture toughness as a function of sintering time is given in Fig. 6(a) for a Y-TZP monolith with and without borosilicate addition. For short sintering times, toughness was reduced by borosilicate addition, but not the maximum toughness (~14 MPa $\sqrt{m}$ after 5 h) which remained the same. The borosilicate addition appears to lead to better toughness retention at longer sintering times. A similar trend was found for hot-pressed materials, Fig. 6(b), although maximum toughness was increased by the borosilicate addition. The effect of the platelets on the toughness/sintering time relationship is given in Fig. 7 which indicates that composites with the borosilicate addition were tougher than those without.

The effect of platelet volume fraction on toughness for the borosilicate-containing materials

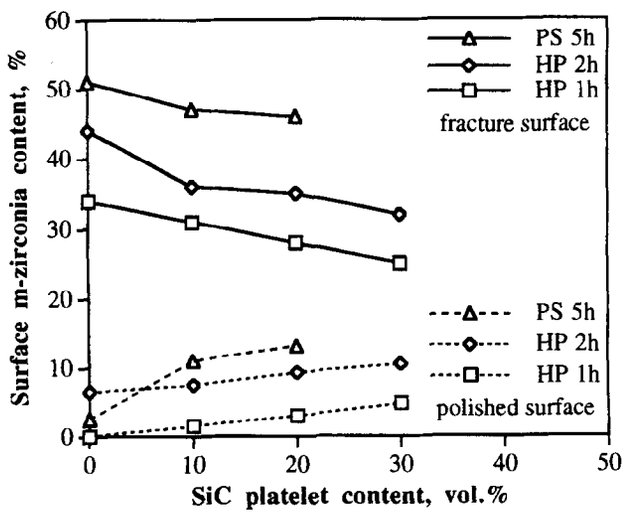
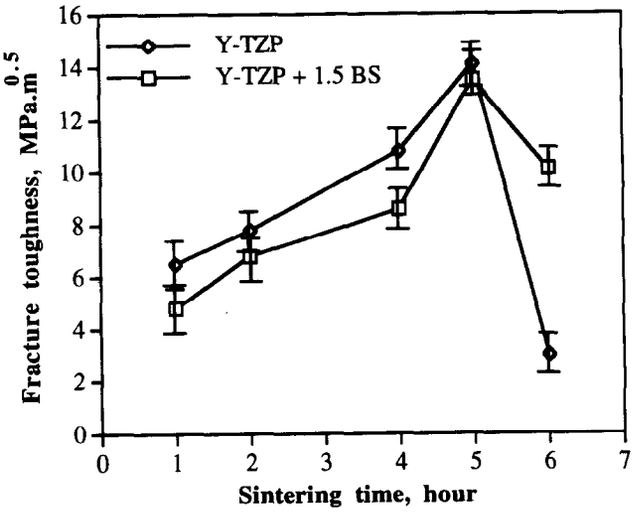


Fig. 5. Fracture and polished surface monoclinic content as a function of SiC platelet content for pressureless-sintered and hot-pressed materials containing borosilicate additions. The surface monoclinic content is expressed as a percentage of the (t + m + c), not of the whole sample.

depended sensitively on the matrix starting toughness, Fig. 8. Where sintering conditions were chosen which optimised the toughness of the matrix (monolith grain size of 0·85 μm), platelet addition reduced overall toughness. The behaviour of the pressureless-sintered sample and the sample hot pressed for 2 h were similar, despite significant differences in porosity and platelet alignment between the two samples. In contrast, where a low matrix toughness was obtained by short sintering times, which promoted a finer tetragonal grain size (monolith grain size of 0·71 μm), a moderate increase in toughness was obtained with platelet addition. Interestingly, the toughness at high platelet loadings converged for all sintering conditions.

Adding platelets decreased the bend strength of both pressureless-sintered and hot-pressed materials, Fig. 9. The bend strength of material hot pressed for 1 h decreased from 800 MPa in the monolith to 600 MPa for a 30 vol% SiC addition.



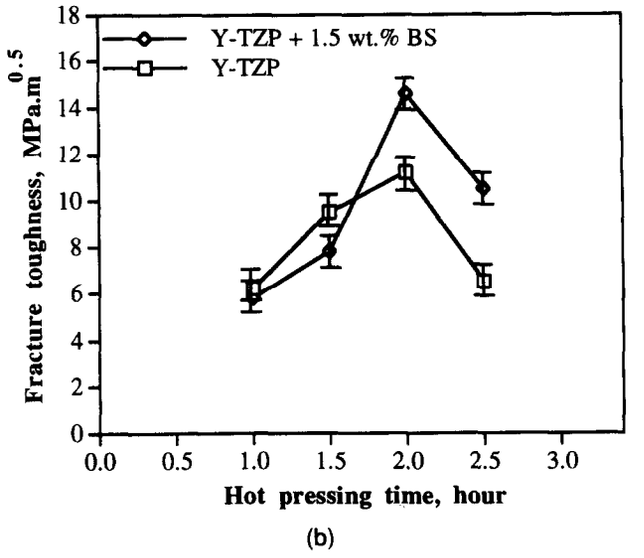


Fig. 6. Fracture toughness of the monolith with and without the borosilicate addition as a function of (a) pressureless sintering time at 1500°C; (b) hot-pressing time at 1500°C.

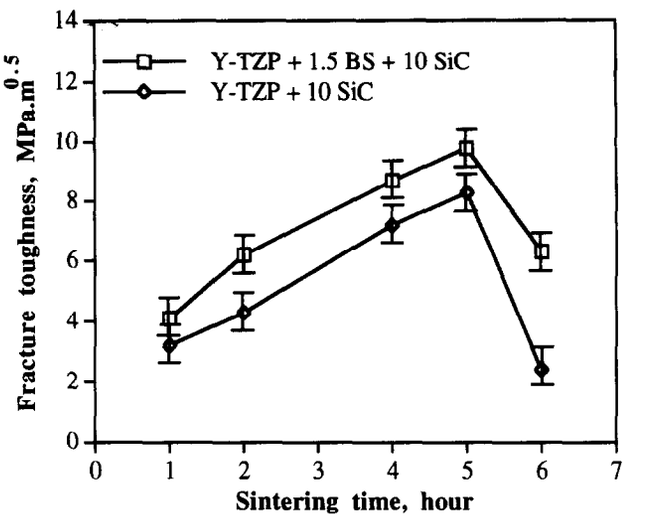


Fig. 7. Fracture toughness as a function of pressureless sintering time for a 10 vol% platelet addition, with and without borosilicate addition.

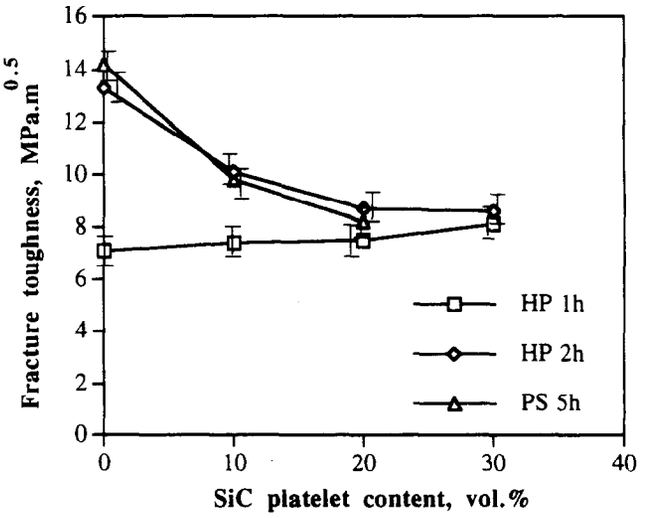


Fig. 8. Fracture toughness as a function of the SiC platelet content for pressureless-sintered and hot-pressed materials, all containing the borosilicate addition.

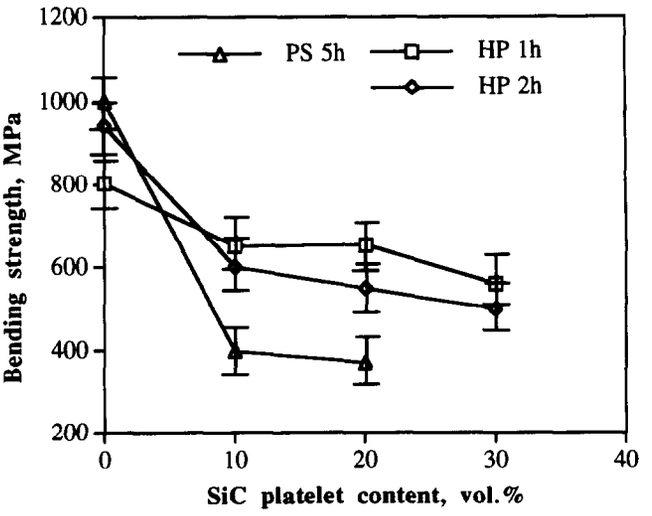


Fig. 9. Bend strength as a function of SiC platelet content for pressureless-sintered and hot-pressed materials, all containing the borosilicate addition.

The pressureless-sintered material processed for optimum matrix toughness exhibited a drop from 1000 to 400 MPa for a 10 vol% addition.

SEM studies indicated that the composite fracture surfaces were considerably rougher than those of the monolith. Numerous examples of crack deflection were identified on the fracture surfaces and on cracks induced by hardness indents (e.g. Fig. 10). Fracture surfaces revealed fine porosity present in SiC agglomerates caused by constrained densification.

The hardness increased as a function of platelet addition for hot-pressed materials, Fig. 11. This increase was greater for the composites pressed for 2 h compared to 1 h, reflecting the marginal increase in density of the material sintered for the longer time. The pressureless-sintered composites showed a similar increase in hardness for a 10 vol% platelet addition, but a dramatic decrease for the 20 vol% addition. This was considered to be a result of the reduced density of this material and increased monoclinic content.

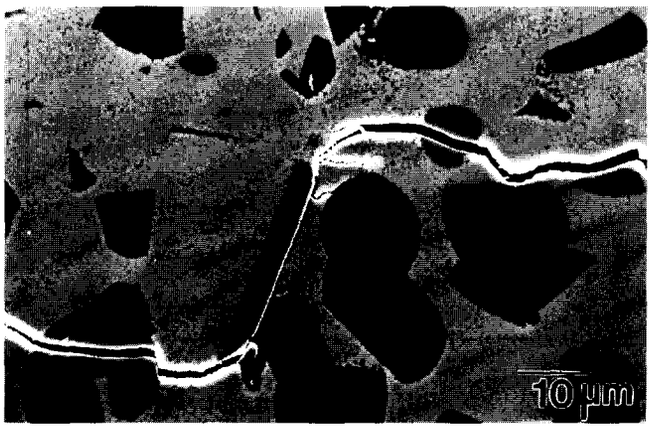


Fig. 10. SEM micrograph of a crack induced by a Vickers indent. Note the deflection of the crack by the SiC platelet and the points at which the crack has passed directly through the reinforcement.

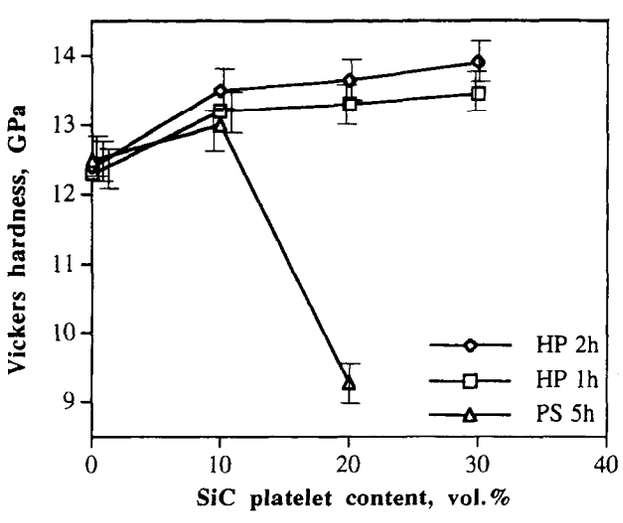


Fig. 11. Vickers hardness as a function of SiC platelet content for pressureless-sintered and hot-pressed materials containing borosilicate additions.

4 Discussion

Becher[14] has considered the toughening provided by crack deflection, bridging and pull-out and considers that a synergistic toughening effect of transformation and crack bridging should be possible in whisker- and particle-reinforced systems. Indeed, in earlier studies, Becher[21] showed that the combined addition of SiC whiskers and tetragonal zirconia to a mullite matrix gave a five-fold increase in toughness, greater than the increase from individual additions of SiC and zirconia added together. The toughness of the composite is described by:[21]

$$K^c = K^m = \Delta K^T \quad (1)$$

Where K^m is the toughness of the matrix and ΔK^T is given by:

$$\Delta K^T = A\varepsilon^T E^c V^T (r^{1/2}{}_T) \quad (2)$$

where A is a constant related to the stress state (0·45–0·5 for shear stresses, 0·2–0·25 for principal stresses), ε^T is the transformation strain, E^c is the stiffness of the composite, V^T is the volume of tetragonal which actually transforms during fracture and $(r^{1/2}{}_T)$ is the transformation zone size.

Crack-deflection toughening has been shown to occur in the present work by the increase in toughness with platelet addition for the composites hot pressed for 1 h (Fig. 2), despite a decrease in the volume fraction of tetragonal zirconia transformed to monoclinic symmetry on fracture (Fig. 5). However, no synergistic effect of transformation toughening and crack deflection was found. The composites were processed to give a range of grain sizes and therefore a range of driving forces for transformation toughening. However, the composites' toughness was always well below the highest value of 15 MPa $m^{0.5}$ obtained in the monolith. The current results suggest that it is not possible to obtain additive toughening mechanisms of transformation and crack deflection in platelet systems to the extent that the maximum system toughness exceeds the maximum from transformation alone.

The reasons for this are complex, but reflect a significant decrease in the contribution from transformation toughening with the addition of platelets as shown by a decrease in the (fracture surface – polished surface) monoclinic value for all materials, Fig. 5. This is consistent with the findings of Huang and Nicholson,[13] Heussner and Claussen[2] and Selcuk *et al.*[22] Heussner and Claussen suggested that elastic load transfer by platelets lowers the shear stresses at the crack tip reducing the volume tetragonal zirconia transforming to monoclinic symmetry.

The properties of composites are dominated by the thermal mismatch stresses. The thermal mismatch stress, σ_m can be calculated from:[23]

$$\sigma_m = \frac{(\alpha_m - \alpha_p)E_p V_p \Delta T_g}{1 + V_p(E_p/E_m - 1)} \quad (3)$$

where $\alpha_{ZrO_2}(=10 \times 10^{-6}°C^{-1})$, $\alpha_{SiC}(=4{\cdot}7 \times 10^{-6}°C^{-1})$, are the thermal expansion coefficients for 0–1000°C, E_m ($\approx$200 GPa) and E_p ($\approx$400 GPa), are the moduli of the matrix and platelets, V_p is the volume fraction of platelets and T_g is the temperature below which no interfacial relaxation takes place. The Littleton softening temperature for Pyrex™ glass is 720°C (M.C. Cable, personal communication) which yields σ_m = 139 MPa for a 10 vol% addition, 254 MPa for 20 vol% and 352 MPa for 30 vol% platelets. However, this calculation ignores the expansivity of the borosilicate addition (α = 2·3–3·5 $\times$ 10^{-6} $°C^{-1}$) which promoted a semi-continuous grain boundary glassy film and a small volume fraction of $ZrSiO_4$ ($\alpha = 6 \times 10^{-6}°C^{-1}$).

The true residual stress condition is clearly complex and eqn (3) only gives some indication of the magnitude and location of tensile and compressive stresses. Nevertheless, the stresses clearly had a significant effect as shown by the cracking at platelet/matrix interfaces and the increase in polished surface monoclinic content with platelet addition (Fig. 5). The significance of tensile stresses in the zirconia depends strongly on the tetragonal grain size. For a grain size close to the critical grain size for spontaneous transformation, the tensile stresses would be expected to promote extensive transformation to monoclinic on cooling, and an overall decrease in toughness. Conversely, for a fine tetragonal grain size, the tensile stresses might be expected to augment an externally applied stress and enhance the driving force for transformation leading to enhanced toughness. However, no evidence was found in the current study that tensile stresses in the matrix increase the total volume fraction of tetragonal zirconia transforming on fracture. They do, however, increase the proportion of monoclinic zirconia found on cooling from sintering.

The reason for the increase in grain size with platelet addition of ≥20% is not obvious. Although the change was small (Fig. 5), such small changes would be expected to have a significant effect on toughness since the average grain size was close to the critical size for spontaneous transformation on cooling.[24] This result contrasts with that of Yasudu *et al.*[25] who found a reduction in grain size in a SiC whisker-Y-PSZ composite (0·3 μm) compared to the monolith (0·5 μm), attributed to the restraint of grain coarsening imparted by the whiskers. However, for an equivalent volume fraction of reinforcement, there would be a smaller number of platelets than whiskers because of the size difference. Thus, whiskers offer a greater number of pinning points than platelets and would therefore be more effective in inhibiting grain growth.

All platelet additions decreased the strength of the material. The maximum degradation occurred for a 10 vol% addition, and further increases in platelet content only gave minor additional reductions. The reduction in strength in platelet composite materials compared to the monolith is well known[22] and is generally considered to occur because the reinforcement acts as a critical flaw, and therefore the critical flaw size is greater in the composite than in the monolith. This explains why a dramatic reduction is found for the first addition, but that further additions have a much smaller effect. However, the presence of a large tensile thermal mismatch stress in the matrix (σ_m of 352 MPa for a 30 vol% addition, if the thermal expansivity of the glassy phase is ignored) will have also played a significant role in reducing strength. Claussen *et al.*[1] observed reduced strength (around 50%) when adding 30 vol% whisker to a 3Y-TZP matrix, compared to a 25% reduction for the composite hot-pressed for 1 h in the present work and 42% reduction for the composite hot-pressed for 2 h. The smaller reduction in the present work is interesting, given that the critical flaw size will be greater in the platelet composite than the whisker composites of Claussen *et al.* The differences may therefore be explained by a reduction in the tensile thermal mismatch stresses in the present study imparted by the borosilicate glass, which improved the strength of the composite.

5 Conclusions

(1) Composites containing SiC platelets with high density levels were produced by hot-pressing. Pressureless sintering resulted in reduced density, attributed to poor matrix penetration in regions of platelet agglomeration.

(2) The addition of platelets decreased the volume fraction tetragonal zirconia transforming to monoclinic symmetry for all processing conditions.

(3) For samples hot-pressed for 1 h, toughness increased from 7·0 to 8·3 MPa $\sqrt{m}$ for a 30 vol% addition. This was associated with a decrease in fracture surface monoclinic, demonstrating the presence of crack-deflection toughening.

(4) The maximum composite toughness was less than the maximum achieved in the monolith for all processing conditions and matrix grain sizes studied (~0·7–0·95 μm). This was a result of the greatly reduced contribution from transformation toughening which was not offset by crack-deflection toughening mechanisms.

(5) Strength was reduced for all SiC platelet additions, associated with an increase in critical flaw size and residual tensile stresses in the matrix.

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