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Toughening mechanisms and properties of mullite matrix composites reinforced by the addition of SiC particles and Y-TZP

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Mullite matrix composites reinforced by SiC particles and Y-TZP, were fabricated by hot-pressing. The effects of adding SiC particles and Y-TZP to mullite or mullite-based materials on properties and toughening mechanisms in the composites were investigated. Crack deflection is proposed as the principal toughening mechanism, produced by the addition of SiC particles. Transformation and microcrack toughening are the two main toughening mechanisms caused by Y-TZP addition. However, the magnitude of their contribution varied with increasing Y-TZP addition. With low Y-TZP addition, the transformation toughening dominated, while at a higher Y-TZP content, the microcrack toughening was dominant. The simultaneous addition of SiC particles and Y-TZP to mullite resulted in higher increases in both flexural strength and fracture toughness, than the simple sum of those obtained by the separate processes. It appears that the two toughening processes were coupled, thereby leading to synergistic toughening and strengthening effects in the mullite composites.

1. Introduction

Mullite has stimulated intensive interest in the field of high-temperature application because of its low thermal expansion coefficient, low thermal conductivity, excellent creep resistance, phase stability and retention of fracture strength at high temperatures [1–6]. However, relatively low fracture strength and toughness of this material at room temperature, as compared to other ceramics such as stabilized-tetragonal zirconia (TZP), partially-stabilized zirconia (PSZ) and Si_3N_4, have kept it from being applied. It is critical to improve fracture strength and toughness of mullite ceramics so that they can be widely used for high-temperature applications. It is desirable to fabricate mullite matrix composites with the incorporation of reinforcing agents to improve their poor strength and toughness. Two main techniques have been attempted so far to toughen mullite, which involve adding ZrO_2 and SiC whiskers [7, 8]. In these composites, the principal toughening mechanisms were considered to be whisker pull-out, crack deflection, crack bridging and crack pinning introduced by the addition of SiC whiskers, and microcracking or phase-transformation toughening caused by the addition of ZrO_2.

It is generally believed that only one toughening mechanism occurs during fracture; but many toughening mechanisms may be observed in the same composite which often reveal additive or synergistic toughening effects. Recently, attention has been paid to utilizing these multiple toughening effects to enhance properties in ceramic matrix composites. These effects can be produced by simultaneous addition of multiple reinforcing agents in the same composite. Some encouraging results have been achieved for the mullite matrix composites with the simultaneous addition of SiC whiskers and ZrO_2 [9–11]. For instance [10], the fracture toughness increased from 2.2 MPa $m^{1/2}$ in pure mullite to 4.8 MPa $m^{1/2}$ due to SiC whisker addition and to 4 MPa $m^{1/2}$ due to PSZ addition. However, when both SiC whiskers and PSZ were added, the toughness reached a higher value of 6.2 MPa $m^{1/2}$. The increase in the fracture toughness due to SiC whiskers and ZrO_2 appears to be nearly additive. Analysis was also carried out for the combined toughening effects introduced by a simultaneous addition of SiC whiskers and ZrO_2 to mullite matrix, which can result in an additive or synergistic toughening effect [11, 12].

The use of multiple reinforcing agents in mullite matrix composites seems to be an effective method for enhancing mechanical properties significantly. In this work, SiC particles (p) and Y-TZP were selected as two reinforcing agents for mullite matrix composites. The mechanical properties of mullite matrix composite formed by the incorporation of such two reinforcing agents were compared with those of the composites with either SiC particle or Y-TZP addition alone. Toughening mechanisms caused by SiC particle or Y-TZP addition, and multiple toughening effects and mechanisms introduced by their simultaneous addition, were also explored.

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2. Experimental procedure

Starting powder for the matrix material was a commercially available mullite with an average particle diameter of 2 μm and a composition as follows: 75.92 wt % Al_2O_3, 23.34 wt% SiO_2 and 0.74 wt% of other oxide impurities (CaO, Fe_2O_3, Na_2O, etc.). SiC particles, as one of the reinforcing agents, were the commercially available α-type SiC with a mean particle size of 0.93 μm and an average surface area of 7.53 $m^2 g^{-1}$, which was composed of 27.0 wt % total C, 0.41 wt % free C, 68.5 wt % total Si, 1.08 wt % free Si, 2.15 wt % O and 0.33 wt % of other oxides such as Fe_2O_3, Al_2O_3 and CaO. Y-TZP containing 2.8 mol % Y_2O_3 as the other reinforcing agent was prepared by the chemical co-precipitation method.

Powder mixtures of mullite, SiC and Y-TZP were mixed in the desired proportions using distilled water with Al_2O_3 balls for 6 h. In addition, an extra 0.5 wt % Y_2O_3 as a sintering aid was added to those powder mixtures which contained the addition of SiC particles. After drying in air, the powder mixtures were sieved through a 120 mesh sieve and moulded in a BN-coated graphite die for hot pressing, which was carried out in an atmosphere of nitrogen at 1600 °C for 40 min under a pressure of 25 MPa. As a comparison, monolithic mullite was also fabricated under the same conditions.

All the hot-pressed samples were sliced and ground to dimensions of 2.5 mm × 5 mm × 28 mm for strength measurements and to dimensions of 5 mm × 2.5 mm × 28 mm with a notch of 0.22–0.25 mm width and 2.5 mm depth for toughness measurements. The flexural strength and fracture toughness of the hot-pressed samples were measured in three-point bending with a span of 20 mm at a constant crosshead speed of 0.5 and 0.05 mm min^{-1}, respectively.

The microstructures were studied by transmission electron microscopy (TEM). The foils for TEM observation were prepared by grinding and polishing the sample to a 1 μm diamond grit finish, and then ion-beam thinning followed by coating with amorphous carbon. X-ray diffraction (XRD) quantitative analyses were accomplished on the polished surfaces and their fracture surfaces for the specimens containing ZrO_2 in order to determine the variation of the fraction of monoclinic ZrO_2 (m-ZrO_2) or tetragonal ZrO_2 (t-ZrO_2) before and after fracture. The fraction of monoclinic zirconia phase was calculated from the reflection–intensity ratio of the monoclinic peaks, $(1\,1\,\bar{1})$ and (1 1 1), to the tetragonal peak (1 1 1) according to Garvie and Nicholson's equation [13].

3. Results and discussion

3.1. Mullite composites reinforced by SiC particles alone

Fig. 1 shows the change of flexural strength and fracture toughness as a function of increasing volume fraction of SiC particles for the mullite composites reinforced by SiC particles alone. Flexural strength of the composites was improved by the addition of 10 vol % SiC particles. At 20 vol %, the flexural strength appears to be levelled off. Also, it is seen that there is a trend toward increasing fracture toughness with increasing particle content. It is noted that the addition of SiC particles produced a significant toughening of mullite. At 30 vol % SiC particles, the flexural strength and fracture toughness of the composites reached higher values of 360 MPa and 4.6 MPa $m^{1/2}$, respectively, as compared to the values of 240 MPa and 2.9 MPa $m^{1/2}$, respectively, for the monolithic mullite. The toughness increases in the composite with increasing SiC particle content are in agreement with those predicted from the crack deflection model for spherical particles (second phase) with a uniform spacing by Fable and Evans [14]. SEM observation of the mullite composites with SiC particle addition having Vickers' indentations, showed evidence of crack deflection [15, 16].

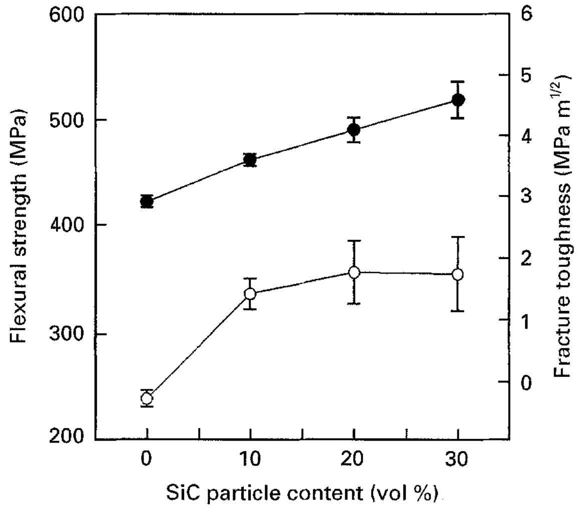

Figure 1 (○) Flexural strength and (●) fracture toughness of the mullite composites reinforced only by SiC particles as a function of SiC particle content.

Therefore, it may be concluded that crack deflection toughening is mainly responsible for the increased fracture toughness and flexural strength of the composites. The levelling off in strength at and above 20 vol % SiC particles seems to be caused by the agglomeration and poor sinterability of SiC.

3.2. Mullite composites reinforced by Y-TZP addition alone

Fig. 2 shows the variation of flexural strength and fracture toughness as a function of increasing volume fraction of Y-TZP for the mullite composites reinforced by Y-TZP addition alone. It is noted that the Y-TZP addition produced a significant toughening of mullite. The more the Y-TZP addition, the greater was the toughness of the composite. At 30 vol %, the fracture toughness reached a high value of 6.0 MPa $m^{1/2}$. On the other hand, flexural strength of the composites increased with increasing Y-TZP content up to 20 vol %. However, the flexural strength of the composite did not show further improvement as the Y-TZP content was raised further.

In order to understand whether transformation toughening acted in the composites reinforced by the addition of Y-TZP only, the relative fraction of

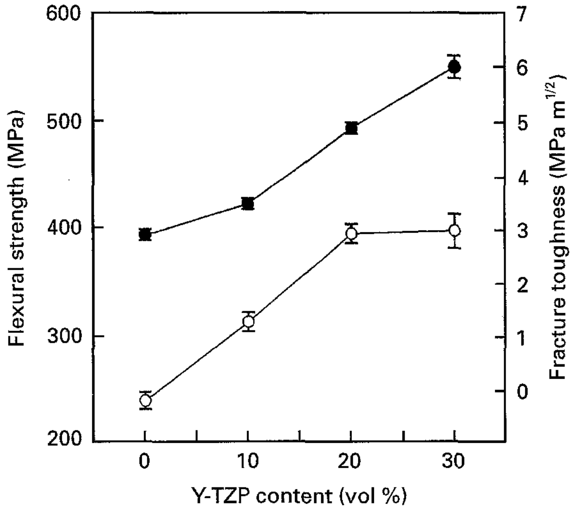

Figure 2 (○) Flexural strength and (●) fracture toughness of the mullite composites reinforced only by Y-TZP addition as a function of Y-TZP content.

m-ZrO_2 in the composites was determined on the polished surfaces and the fractured surfaces, and the results are given in Table I. Although this is somewhat arbitrary, the increase in the monoclinic portion of ZrO_2 after fracture reveals some trend of transformation toughening contribution. As shown here, a larger amount of t-ZrO_2 transformed to its monoclinic form during fracture, except for the 30 vol % Y-TZP addition. Therefore, the transformation toughening should be responsible for the increased strength and toughness in the composites. However, the amount of newly produced m-ZrO_2 during fracture fell with increasing Y-TZP content, and particularly at 30 vol % Y-TZP addition, there is no m-ZrO_2 created during fracture and, conversely, some m-ZrO_2 phase transformed back to its tetragonal form (this is not yet clearly understood and needs further study), but the fracture toughness of these composites continued to increase with increasing Y-TZP content. This indicates that the toughening is not due to transformation toughening alone. On the other hand, it can be seen that a considerable fraction of t-ZrO_2 grains had undergone transformation during cooling from the hot-pressing temperature. Moreover, the amount of m-ZrO_2 in the as-sintered samples increased with increasing Y-TZP addition. This can be attributed to three main factors as follows: a decrease in the yttrium content in Y-TZP particles as a result of Y_2O_3 dissolution into glassy phase during sintering, an increase of ZrO_2 particle size due to the increased Y-TZP content, and the large tensile stress around the ZrO_2 particle as a result of the thermal expansion mismatch between the mullite and ZrO_2. This transformation during cooling can result in microcrack formation [17]. Interaction of these microcracks with the tip of the main crack would increase the fracture energy, therefore increasing toughness. As the microcrack level increases, the toughness is also raised. However, if these microcracks are developed as links between ZrO_2 particles, the matrix will be weakened, and the overall toughness and strength can also be decreased. In our case, direct evidence for the existence of microcracks in this composite is revealed in the transmission electron micrographs in Fig. 3. The microcracking is observed at a m-ZrO_2 mullite interphase interface, and a typical twinned structure is seen to appear in the m-ZrO_2 particle, which is also consistent with Dravid *et al.*'s observation [18]. It is apparent that the microcracking and twinning in this composite are entirely due to the thermal transformation of zirconia during cooling. Thus the increased toughness at 30 vol % Y-TZP addition can be explained by microcrack toughening. The flexural strength result shows no decrease at 30 vol % Y-TZP addition, which implies that the adjacent microcracks are still isolated in the composite. Similar results have been obtained in Al_2O_3/ZrO_2 composite [19].

TABLE I Relative fraction of m-ZrO_2 in the hot-pressed mullite composites with Y-TZP addition alone, determined on the polished surfaces and the fractured surfaces (M, mullite)

Composition (vol %)	Relative fraction of monoclinic phase in ZrO_2		
	Polished surface	Fractured surface	Increase
M + 10 Y-TZP	25	76	51
M + 20 Y-TZP	59	83	24
M + 30 Y-TZP	74	47	–

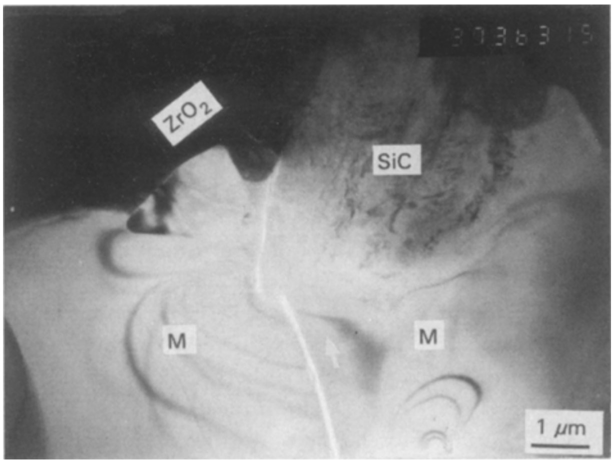

Figure 3 Transmission electron micrograph of the mullite composite containing 30 vol % Y-TZP, showing a microcrack (arrowed) at the m-ZrO_2/mullite (M) interface.

It is noted that both transformation toughening and microcrack toughening caused by Y-TZP addition resulted in a higher value of fracture toughness. The magnitude of their contribution varied with the volume fraction of Y-TZP addition. At low amounts of Y-TZP from 10–20 vol %, transformation toughening dominates the increased toughness and strength, and at higher Y-TZP content (30 vol %), the microcrack toughening determines the higher toughness in the mullite composites with Y-TZP addition alone.

3.3. Mullite composite reinforced by both SiC particles and Y-TZP

As shown in Fig. 1, the 30 vol % SiC particle addition had optimal toughening effect for the composite reinforced only by SiC particles. So did the 20 vol % Y-TZP addition for the composite with Y-TZP addition alone. Thus the composites with 30 vol % SiC particles and 20 vol % Y-TZP addition were selected as two base materials to investigate the effects of Y-TZP and SiC particles, respectively. Fig. 4 shows the effects of increasing SiC particle content on flexural strength and fracture toughness of the composites with a constant Y-TZP content of 20 vol %. It was found that both flexural strength and fracture toughness increased with increasing SiC particle content up to a maximum values at 30 vol % SiC particles. When added concurrently with Y-TZP, a higher than 10 vol % SiC particle addition can produce more marked toughening and strengthening effects in the composites, compared with the composites reinforced by SiC particles alone, as seen in Fig. 1. The effects of increasing Y-TZP content on flexural strength and fracture toughness of the composites with a constant SiC particle content of 30 vol % are shown in Fig. 5. Both flexural strength and fracture toughness had an increasing trend with increasing Y-TZP content, having the optimal values at 20 vol % Y-TPZ addition. Although this trend was also followed to some degree by the composites reinforced only by Y-TZP addition, as seen in Fig. 2, these strength increments produced by Y-TZP plus SiC particle addition appear to be greater than those produced by Y-TZP alone. It is seen that an optimum flexural strength of 610 MPa and fracture toughness of 7.1 MPa $m^{1/2}$ of the composites were obtained by the addition of both 30 vol % SiC particles and 20 vol % Y-TZP. However, for the composite with 30 vol % SiC particle addition alone, flexural strength and fracture toughness were 357 MPa and 4.6 MPa $m^{1/2}$, respectively, and for the composite with 20 vol % Y-TZP addition alone, 400 MPa and 4.9 MPa $m^{1/2}$, respectively. Thus, the data seem to exhibit synergistic toughening and strengthening effects of the combination of SiC particles and Y-TZP.

The increases in flexural strength and fracture toughness of the mullite matrix composites with the addition of both SiC particles and Y-TZP for several compositions, in comparison with the simple sum of increments obtained by either SiC particle or T-TZP addition alone, is presented in Table II. It is noted that not all mullite matrix composites with the simultaneous addition of both SiC particles and Y-TZP exhibit the synergistic toughening and strengthening effects, which is seen to appear only when the SiC particle content was raised to 30 vol %.

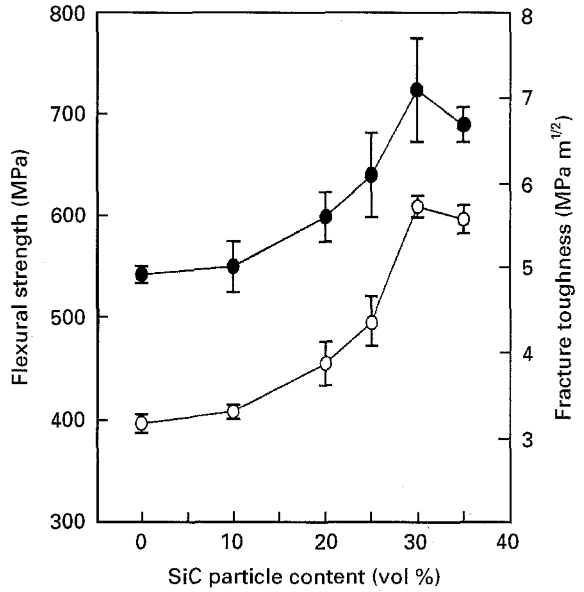

Figure 4 Effects of the volume per cent of SiC particles on (○) flexural strength and (●) fracture toughness of the mullite composites reinforced by both SiC particles and 20 vol % Y-TZP.

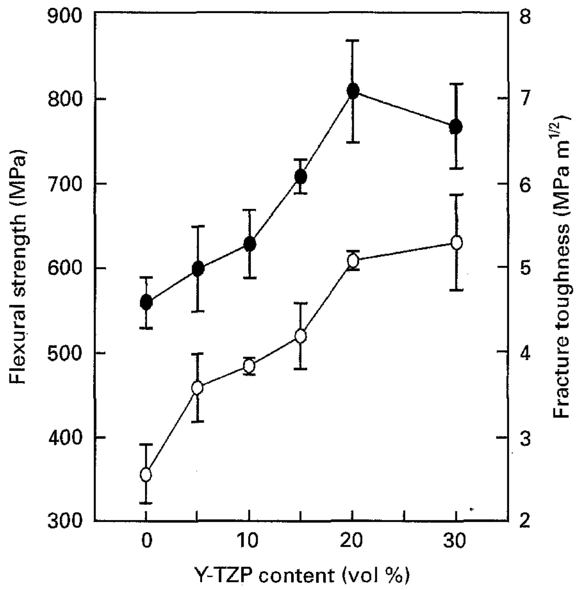

Figure 5 Effects of the volume per cent of Y-TZP on (○) flexural strength and (●) fracture toughness of the mullite composites reinforced by both 30 vol % SiC particles and 30 vol % Y-TZP.

The relative fraction of m-ZrO_2 was determined on polished surfaces and fractured surfaces for the mullite composites with the simultaneous addition of SiC particles and Y-TZP. The results are given in Table III and the data for the composite with 20 vol % Y-TZP addition alone is included for comparison. It is apparent that the amount of m-ZrO_2 phase determined on the polished surface in the as-sintered specimens markedly dropped due to the addition of SiC particles. It indicates that the addition of SiC particles can effectively impede the transformation of t-ZrO_2 into its monoclinic form during cooling in the mullite/Y-TZP-base material, thus keeping more zirconia content in the tetragonal phase. This may result from the very high Young's modulus of SiC. Moreover, the addition of extra Y_2O_3 in the composites, which can inhibit the dissolution of yttrium from stabilized t-ZrO_2 particles during sintering, was also responsible for the low levels of m-ZrO_2 in the as-sintered samples. However, although the amount of m-ZrO_2 phase was obviously lowered after fracture due to the addition of SiC particles for the mullite composites with the addition of both SiC particles and Y-TZP, the newly increased m-ZrO_2 phase amount during fracture (except for the mullite composite with both 30 vol % SiC particles and 30 vol % Y-TZP) continued to show some increases, compared with the

TABLE II Increases, $\Delta\sigma_f$ and ΔK_{1c}, in flexural strength and fracture toughness of the mullite matrix composites with the addition of both SiC particles and Y-TZP in comparison with the simple sum, $\Delta\sigma_{f,\,sum}$ and $\Delta K_{1c,\,sum}$, of increments obtained by the separate processes

Composition (vol %)	Flexural strength (MPa)			Fracture toughness (MPa m$^{1/2}$)		
	σ_f	$\Delta\sigma_f$	$\Delta\sigma_{f,\,sum}$	K_{1c}	ΔK_{1c}	$\Delta K_{1c,\,sum}$
M + 20 Y-TZP + 10 SiC$_p$	409	169	255	5.0	2.1	2.7
M + 20 Y-TZP + 20 SiC$_p$	456	216	275	5.6	2.7	3.2
M + 20 Y-TZP + 30 SiC$_p$	611	371	274	7.1	4.2	3.7
M + 10 Y-TZP + 30 SiC$_p$	476	236	191	5.3	2.4	2.3
M + 20 Y-TZP + 30 SiC$_p$	611	371	274	7.1	4.2	3.7
M + 30 Y-TZP + 30 SiC$_p$	633	393	277	6.7	3.8	4.1

mullite/Y-TZP composite with no SiC particle addition. Thus, in the composites with the addition of both SiC particles and Y-TZP, transformation toughening was retained as the principal toughening mechanism caused by the Y-TZP addition.

Fig. 6 shows a transmission electron micrograph of the mullite composite with 20 vol % Y-TZP and 30 vol % SiC particles. As shown here, a crack propagated through the mullite matrix, and was deflected by the SiC particle at the SiC/mullite interface. Crack deflection is considered to be the main toughening mechanism in the composites, produced by SiC particle addition.

It is well known that transformation toughening occurs because the stress field of the crack tip is relieved by the t → m transformation of ZrO_2 near the propagating crack in t-ZrO_2-toughened composites. A transformation zone is formed in front of the crack tip, greatly reducing the stress intensity of the crack tip. If the crack propagates further, increased applied stresses should be required. The contribution associated with transformation toughening, ΔK^C, can be given by [20, 21]

$$\Delta K^C = \gamma E e^T v^T w^{1/2} \qquad (1)$$

where γ is a constant related to the stress state, E is the Young's modulus of the composite, e^T is the transformation stress, v^T is the volume fraction of transformable t-ZrO_2, and w is the size of transformation zone. As shown here, ΔK^C is a function of the transformation zone size, $w^{1/2}$. However, $w^{1/2}$ is dependent on the ratio of the critical matrix toughness, K^M, to the critical transformation stress required to transform the t-ZrO_2 particles, σ^T [20]. The increased K^M will lead to the increase in $w^{1/2}$. Thus, the greater the K^M, the greater is ΔK^C. It is apparent that increasing the matrix toughness combined with transformation toughening will produce a synergistic toughening effect. In addition, the increased Young's modulus of the composites can increase ΔK^C due to the addition of SiC particles. In this case, the increased toughness of the base mullite/SiC$_p$ material was achieved by the addition of SiC particles. Therefore, a combination of ZrO_2 transformation toughening and SiC particle reinforcement should result in a toughness increase, which is higher than a simple sum of toughness increments achieved by the separate processes. A similar phenomenon has been observed in the mullite composites toughened by both SiC whiskers and ZrO_2 [11, 12].

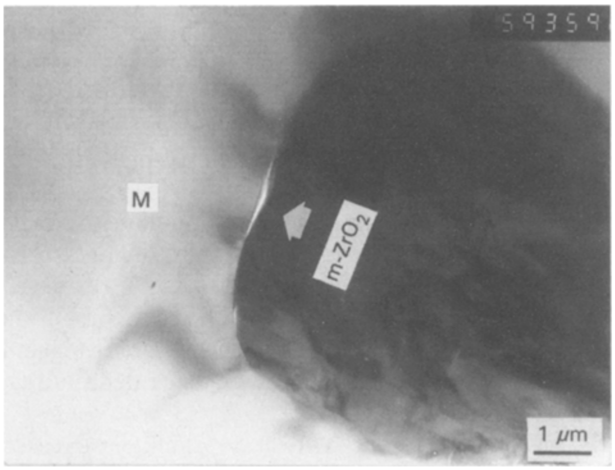

Figure 6 Transmission electron micrograph of the mullite composites containing 20 vol % Y-TZP plus 30 vol % SiC particles. The crack running from the mullite particle (M) was deflected by the SiC particle.

In ZrO_2 transformation-toughened materials, an increase in toughness, K_{1c}, may lead to a high value of strength, σ_f. Both can be related through the relationship

$$\sigma_f = K_{1c}/c^{1/2} \qquad (2)$$

where c is the critical crack length. In this case, a synergistic strengthening effect should result from the combined toughening process introduced by the simultaneous addition of Y-TZP and SiC particles in the mullite composites. However, some mullite composites, as seen in Table III, did not show the synergistic toughening or/and strengthening effects. This may be due to the fact that transformation toughening would make less contribution and perhaps another toughening mechanism, such as microcrack toughening, should be dominant in these mullite composites. However, it was pointed out that in the case of toughening achieved by the presence of dispersed microcracks, the microcrack toughening contribution will be independent of matrix toughness [11]. Therefore, the overall toughness cannot exceed the sum of the matrix toughness and the microcrack toughening contribution.

TABLE III Relative fraction of m-ZrO_2 in the mullite composites with the addition of both SiC particles and Y-TZP, determined on the polished surfaces and the fractured surfaces

Composition (vol %)	Relative fraction of monoclinic phase in ZrO_2		
	Polished surface	Fractured surface	Increase
M + 20 Y-TZP	59	83	24
M + 10 Y-TZP + 30 SiC_p	2	33	31
M + 20 Y-TZP + 20 SiC_p	21	52	31
M + 20 Y-TZP + 30 SiC_p	10	57	47
M + 30 Y-TZP + 30 SiC_p	8	20	18

4. Conclusions

Mullite matrix composites reinforced by SiC particles and Y-TZP, and were both fabricated by hot pressing. The effects of adding SiC particles, Y-TZP, and both to mullite on the mechanical properties as well as toughening mechanisms were investigated. The following results were obtained.

1. The addition of SiC particles to mullite improved the flexural strength and fracture toughness. At 30 vol % SiC particle content, the strength was raised from 240 MPa to 360 MPa, and the toughness from 2.9 MPa $m^{1/2}$ to 4.6 MPa $m^{1/2}$. The principle toughening mechanism is considered to be crack deflection in the mullite composites.

2. The addition of Y-TZP to mullite increased flexural strength and fracture toughness, which reached maximum optimal values of 400 MPa and 6.0 MPa $m^{1/2}$, respectively. Transformation toughening and microcrack toughening contributed to the increased properties. However, the magnitude of their contribution varied with increasing Y-TZP content in the composites. At low amount of Y-TZP addition, the former is dominant, but at higher Y-TZP content, a reversal occurs.

3. Adding SiC particles and Y-TZP together to mullite resulted in higher values of both flexural strength and fracture toughness than a simple sum of those achieved by the single process. The composites showed an optimal flexural strength value of 610 MPa and a fracture toughness value of 7.1 MPa $m^{1/2}$ for the addition of 20 vol % Y-TZP and 30 vol % SiC particles together.

4. In the mullite composites with the simultaneous addition of SiC particles and Y-TZP, the transformation toughening appears to be coupled to the crack-deflection toughening, caused by the addition of SiC particles, which can result in the synergistic toughening and strengthening effects.

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