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Mechanical properties of Al_2O_3 and Al_2O_3 + ZrO_2 ceramics reinforced by SiC whiskers

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The effect of the SiC whisker content on the mechanical properties of Al_2O_3 and Al_2O_3 + 20 vol % ZrO_2 (2 mol % Y_2O_3) ceramic composites has been investigated. It is shown that the strength and fracture toughness of the composites are increased by the addition of 0–30 vol % SiC whiskers with only one exception that 30 vol % SiC whisker leads to a decrease in the flexure strength. The addition of 20 vol % ZrO_2 (2 mol % Y_2O_3) significantly improves the mechanical properties of the Al_2O_3 + SiC whisker (SiC_w) composites and the t–m phase transformation of ZrO_2 is enhanced by the residual stresses caused by the thermal incompatibility between the SiC_w and the matrix. The toughening effect of both SiC whiskers and the t–m phase transformation of ZrO_2 (2 mol % Y_2O_3) is shown to be additive, but the addition of ZrO_2 decreases the strengthening effect of the SiC whiskers.

1. Introduction

Recently, it has been shown that whisker reinforcement and the addition of a phase-transforming component of partially stabilized zirconia are two effective measures for improving the fracture toughness of Al_2O_3. However, the experimental results show serious variations due to the differences in preparation of the material or in the testing procedures. For example, Becher and co-workers [1–3] have shown that an addition of 20 vol % SiC whiskers (SiC_w) to Al_2O_3 can increase the flexure strength and the fracture toughness from 400 MPa and 4.5 MPa $m^{1/2}$ of the matrix to 650 MPa and 8.5 MPa $m^{1/2}$ of the composite. A further addition of 20 vol % ZrO_2 to the Al_2O_3 + 20 vol % SiC_w increases the flexure strength to 750 MPa but decreases the fracture toughness slightly to 7.8 MPa $m^{1/2}$. Claussen and co-workers [4, 5] have shown that the composite of Al_2O_3 + 15 vol % (t-ZrO_2) + 20 vol % SiC_w aged at 1500 °C for 24 h, has a flexure strength of 700 MPa and a fracture toughness of 13.5 MPa $m^{1/2}$, but a hot-pressed composite of Al_2O_3 + 32 vol % (m-ZrO_2) + 20 vol % SiC_w has a flexure strength of 673 MPa and a fracture toughness of 6.3 MPa $m^{1/2}$. Such variations in results have also been obtained by other authors [6–8]. Therefore, a systematic study of the strengthening and toughening effects of both SiC whiskers and the ZrO_2 component to the Al_2O_3 matrix and, especially, to examine the combined effect of these two factors appears to be necessary.

2. Experimental procedure

The starting materials selected for investigation were α-Al_2O_3 powders with a grain size of 0.05 μm, ZrO_2 (2 mol % Y_2O_3) powders with a particle size of 0.65 μm (having 34 % t-ZrO_2 and 66 % m-ZrO_2), and β-SiC whiskers of 0.1–1 μm diameter and 30–100 μm length. The powders and the whiskers were mixed according to the compositions shown in Table 1, then inserted into plastic bottles together with ZrO_2 balls and absolute alcohol. The mixtures were then ground for 24 h and after drying were cold pressed at 200 MPa. The cold-pressed billets were then hot pressed under nitrogen at 1650 °C and 25 MPa for 1 h to a size of 60 mm × 60 mm × 6 mm.

The hot-pressed billets were cut into specimens 3 mm × 4 mm × 36 mm for three-point bending tests to determine the flexure strength with a span of 30 mm, and a crosshead speed of 0.5 mm min^{-1}. Specimens for single-edge notched beam (SENB) tests, 2.5 mm × 5 mm × 25 mm, were cut for fracture toughness measurement with $S/W = 4$. $a/W = 0.5$. The width of the notch was 0.24–0.26 mm and a crosshead speed of 0.05 mm min^{-1} was used. Average values for six specimens were taken for both flexure strength and fracture toughness tests. All tests were performed on an Instron-1186 machine. The elastic modulus was determined by measuring the strains in the three-point bending tests and the hardness values obtained by using a Vicker's hardness tester. The density of the specimens was determined by the Archimede's method. The Hitachi S-570 type of scanning electron microscope was used for examining the fracture morphology and the crack paths and a D/max-rB type X-ray diffractometer was used for the crystallographic analysis of the ZrO_2 component.

3. Results and discussion

3.1. Relative density

The relative density (measured density divided by calculated theoretical density) values of the AS and

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TABLE I Compositions of the Al_2O_3 based composites

	Composite series AS(Al_2O_3 + SiC_w)				AZS(Al_2O_3 + ZrO_2 + SiC_w)			
Content	AS0	AS1	AS2	AS3	AZS0	AZS1	AZS2	AZS3
Al_2O_3(vol %)	100	90	80	70	80	70	60	50
SiC_w(vol %)	0	10	20	30	0	10	20	30
ZrO_2(vol %)	0	0	0	0	20	20	20	20

AZS series of specimens are shown in Fig. 1. It is seen that the addition of SiC_w slightly decreases the relative density of the Al_2O_3 matrix due to the geometric incompatibility of these two particles. However, due to the relatively long time of mixing the agglomeration of the Al_2O_3 grains and the networks of SiC_w are satisfactorily avoided so that the relative density is at least 98.8 % for Al_2O_3 + 30 vol % SiC_w. The addition of SiC_w to the Al_2O_3 + 20 vol % ZrO_2 (2 mol % Y_2O_3) matrix has no significant effect on the relative density which is higher than 99.4 % for all cases.

3.2. Hardness and elastic modulus

The Vicker's hardness and elastic modulus values of the composites are shown in Fig. 2. The two parameters are clearly increased by the addition of SiC_w. For the AS series, a 30 vol % SiC_w increases the hardness and elastic modulus from 14.5 and 401 GPa for the matrix to 18.6 and 454 GPa, respectively. The original hardness of the Al_2O_3 + 20 vol % ZrO_2 (2 mol % Y_2O_3) is 16.1 GPa and the AZS with 30 vol % SiC_w has a hardness of 19.6 GPa. The elastic modulus of the Al_2O_3 + 20 vol % ZrO_2 (2 mol % Y_2O_3) is 380 GPa, lower than that of the Al_2O_3 matrix because of the addition of the ZrO_2 component with a much lower modulus value (220 GPa). Nevertheless, the modulus of the AZS series is also obviously increased by SiC_w addition, as for the AS series.

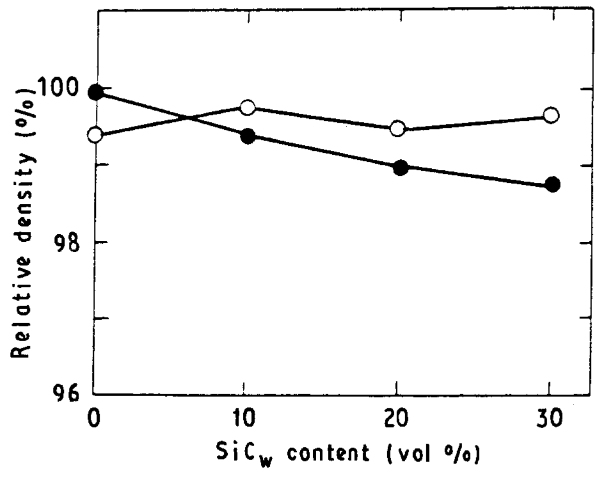

Figure 1 Relative density of the (●) AS and (○) AZS series of composites as a function SiC_w content.

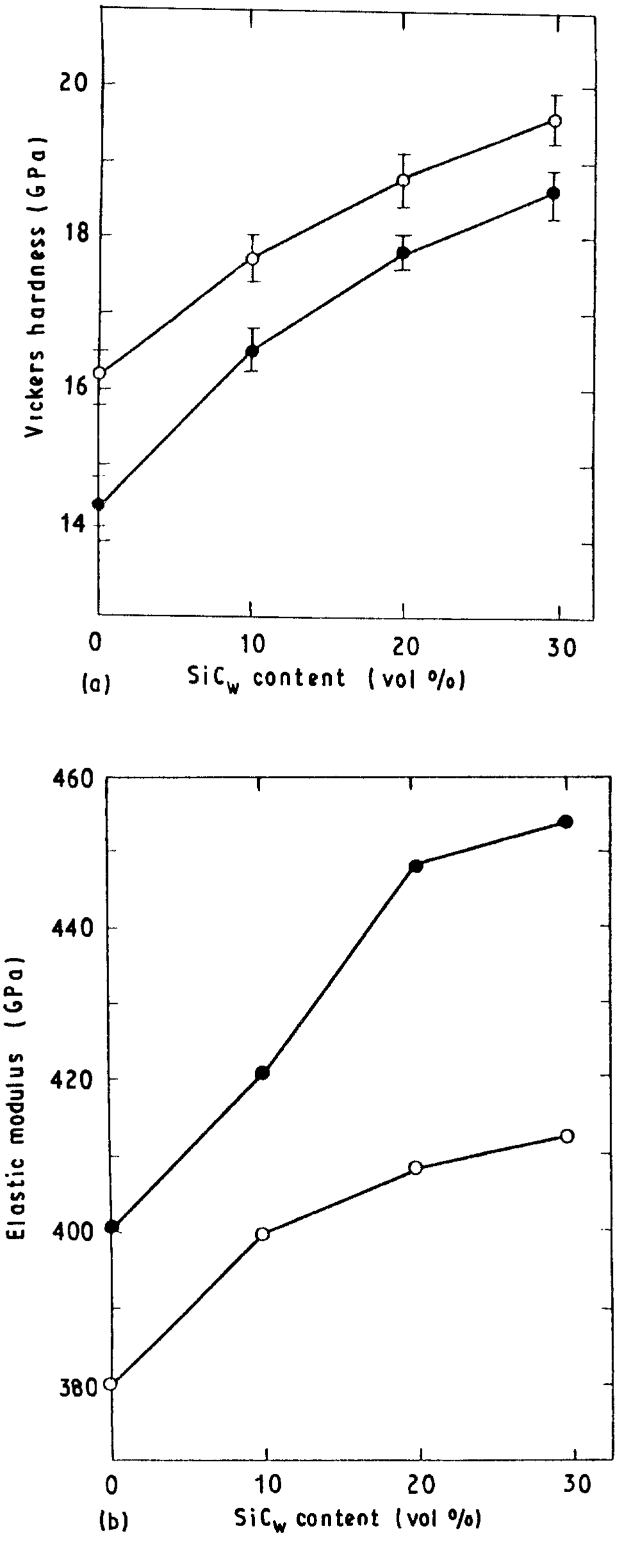

Figure 2 (a) Vicker's hardness and (b) elastic modulus of the (○) As and (●) AZS series of composites as a function of SiC_w content.

3.3. Flexural strength and fracture toughness

The flexural strength and fracture toughness values of the two series of composites are shown in Fig. 3. The strength data of the AS series shown in Fig. 3a are very close to those obtained by Tiegs and Becher in 1987 [3] and Yang and Stevens in 1991 [9]. The first 10 vol % SiC_w has very high strengthening effect and increases the flexural strength from 235 MPa for the matrix to 535 MPa. Further increment of the SiC_w content leads to a much lower strengthening and the strength of Al_2O_3 + 30 vol % SiC_w is only 634 MPa. The addition of 20 vol % ZrO_2 (2 mol % Y_2O_3) to the Al_2O_3 matrix enormously increases the flexural strength to a value of 659 MPa, while the addition of SiC_w increases only slightly the flexural strength of the AZS series of composites with a maximum value of 784 MPa at 20 vol % SiC_w. The further increase of the SiC_w content leads, on the other hand, to a decrease in the strength (720 MPa at 30 vol % SiC_w). The decrease in strength can be explained as a result of the formation of microcracks due to thermal incompatibility between the Al_2O_3 and ZrO_2 powders with the SiC whiskers.

It can be seen from Fig. 3b that the addition of 20 vol % ZrO_2 (2 mol % Y_2O_3) increases the fracture toughness of Al_2O_3 from its original value of 4.4 MPa $m^{1/2}$ to 6.8 MPa $m^{1/2}$. The addition of SiC_w monotonically increases the fracture toughness of both AS and AZS series of composites. This is in good agreement with the results obtained by Yang and Stevens [9]. A 30 vol % SiC_w can increase the toughness of Al_2O_3 to 7.5 MPa $m^{1/2}$ and the toughness of Al_2O_3 + 20 vol % ZrO_2 (2 mol % Y_2O_3) to 10.4 MPa $m^{1/2}$. Obviously, additivity exists in the toughening effect of both SiC_w and the ZrO_2 component for the Al_2O_3 matrix, but there is no additivity with respect to the strengthening effect.

3.4. Strengthening and toughening mechanisms

The scanning electron micrographs of the fracture surfaces of toughness specimens are shown in Fig. 4. It can be seen that the addition of SiC_w, significantly refines the grain size of the Al_2O_3 matrix and changes the fracture mode from intergranular to preferentially transgranular. The microholes are left due to the pull-out of whiskers, and the roughness of the fractured surfaces is increased by the addition of whiskers. These facts are clear evidence of crack deflection and that more energy is absorbed during fracture of the SiC_w containing material. In some cases, the SiC_w may be ruptured causing more energy consumption, as shown in Fig. 4d.

SEM observations of the crack path at the corners of indents during the hardness tests are shown in Fig. 5 for the material AZS3 (Al_2O_3 + 20 vol % ZrO_2 (2 mol % Y_2O_3) + 30 vol % SiC_w). The crack deflection (Fig. 5a), SiC_w rupture (Fig. 5b) and SiC_w pull-out and bridging (Fig. 5b–d) are clearly seen. The ZrO_2 particles can effectively enhance the crack deflection and shield the main crack from further propagation (Fig. 5d).

The toughening effect of the ZrO_2 particles due to their t–m phase transformation can be revealed by crystallographic analysis. The results of the X-ray diffraction analysis of polished and fractured surfaces

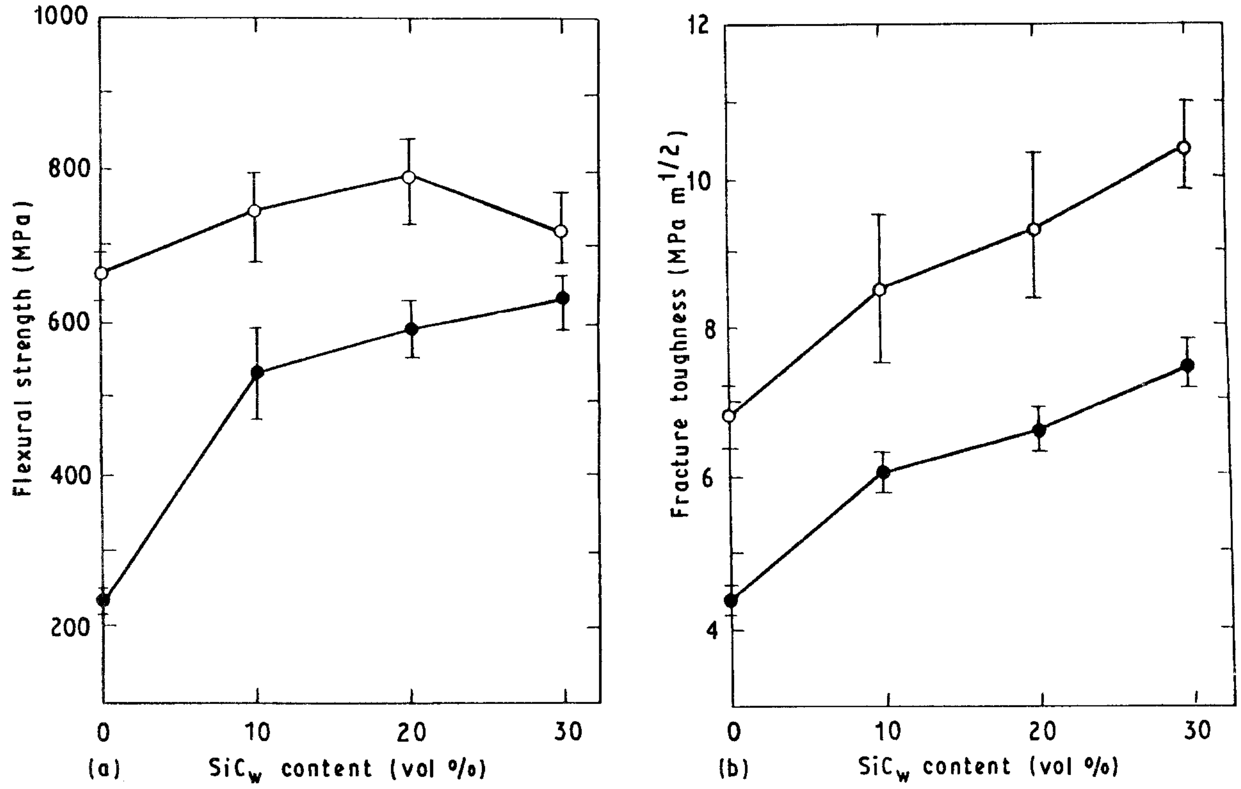

Figure 3 (a) Flexural strength and (b) fracture toughness of the (○) AS and (●) AZS series of composites as a function of SiC_w content.

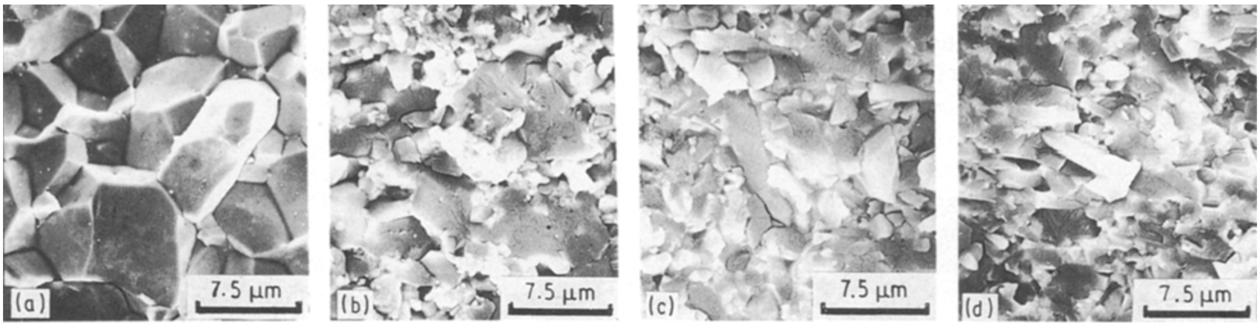

Figure 4 Scanning electron micrographs of SENB fractured surfaces of composites (a) Al_2O_3; (b) Al_2O_3 + 20 vol % ZrO_2 (2 mol % Y_2O_3); (c) Al_2O_3 + 30 vol % SiC_W; (d) as (b) + 30 vol % SiC_W.

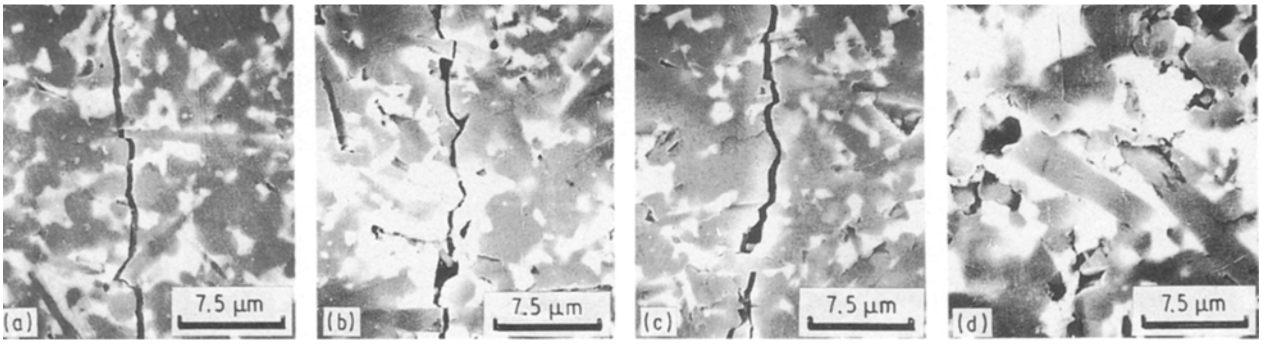

Figure 5 Scanning electron micrographs of Al_2O_3 + 20 vol % ZrO_2 (2 mol % Y_2O_3) + 30 vol % SiC_W showing the crack paths produced at the corners of indents during Vicker's hardness tests (a)–(d) see text.

TABLE II Amount of the m-phase of ZrO_2 (2 mol % Y_2O_3) on polished and fractured during SENB tests surfaces in AZS series of composites

	Material			
	AZS0	AZS1	AZS2	AZS3
Amount of m-phase on polished surface (%)	8.7	7.6	10.8	16.8
Amount of m-phase on fractured surface (%)	13.4	19.3	29.8	33.3
Amount of t–m transformation during fracture (%)	4.7	11.7	19.0	16.5

of the AZS series of composites are shown in Table II. The structural composition of the ZrO_2 (2 mol % Y_2O_3) powders before hot pressing was 34 % t + 66% m. The hot pressing was performed at 1650 °C, which lies in the single t-phase region according to the ZrO_2–Y_2O_3 phase diagram, and after cooling to room temperature part of the t-phase transforms to m-phase but to a much lower extent because of the larger constraint in the composite with a much higher elastic modulus than pure ZrO_2 (2 mol % Y_2O_3). It is seen from Table II that the Al_2O_3 matrix has a strong constraint to the ZrO_2 (2 mol % Y_2O_3) which transforms only to 8.7 % m-phase under cooling from the hot-pressing temperature. The addition of SiC_w, due to thermal stresses caused by the difference in coefficients of expansion (8.5×10^{-6} K^{-1} for Al_2O_3, 10×10^{-6} K^{-1} for ZrO_2 + 2 mol % Y_2O_3 and 4.7×10^{-6} K^{-1} for SiC_w) enhances the t–m transformation so that with increasing SiC_w the amount of m-phase on the polished surface of the material gradually increases and reaches 16.8 % for the AZS3 with 30 vol % SiC_w. An example indicating the t–m phase transformation enhanced by the tensile stresses in areas near the whiskers is shown in Fig. 6. Fig. 6 also shows that SiC whiskers and the matrix are bonded tightly with no obvious second phase or intermediate layers at the SiC_w/matrix interface. The amount of m-phase of ZrO_2 (2 mol % Y_2O_3) on fractured surfaces increases with SiC_w content in a more distinct manner so that the amount of t–m phase transformation during fracture in the toughness tests obviously increases with the SiC_w content. It has been proved in the Al_2O_3 + ZrO_2 (2 mol % Y_2O_3) ceramics [10] that a mixture of the t + m phases can give a better toughening effect because the t-phase can give transformation toughening by the dynamic t–m transformation during fracture and the prior-existing m-phase can give a microcrack toughening effect by inducing microcracks around the particles which transformed from t- to m-phase during cooling from the sintering or hot-pressing temperature. Therefore, such microcracks are also beneficial in the toughening of the AZS materials but may be harmful to the strengthening, as shown in Fig. 3 for the flexure strength of AZS3.

4. Conclusions

1. The flexure strength and fracture toughness of the Al_2O_3 + SiC_w composites are increased by increasing the SiC_w content. The flexure strength of

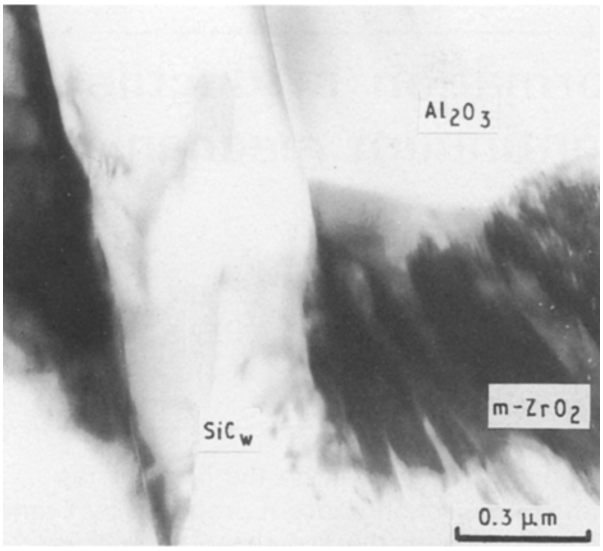

Figure 6 Transmission electron micrographs of Al_2O_3 + 20 vol % ZrO_2 (2Y) + 20 vol % SiC_W composite showing an interface of SiC_W/matrix and m-phase structure in ZrO_2 particles near the SiC whiskers.

Al_2O_3 + 20 vol % ZrO_2 (2 mol % Y_2O_3) + SiC_w increases with SiC_w content only to 20 vol % SiC_w, after which the strength decreases due to excessive microcracking but the fracture toughness increases monotonically with the SiC_w content.

2. The flexural strength and fracture toughness of the Al_2O_3 + 20 vol % ZrO_2 (2 mol % Y_2O_3) + SiC_w composite are always higher than those of the Al_2O_3 + SiC_w composite. The toughening effect of both SiC_w and the ZrO_2 component has an obvious additive effect, at least for the Al_2O_3 matrix. The addition of ZrO_2 decreases the strengthening effect of SiC_w to some extent.

3. The residual stresses caused by the thermal incompatibility between the SiC_w and the matrix enhance the t–m ZrO_2 transformation. The amount of t–m transformation increases with increasing SiC_w content.

4. The main mechanisms of strengthening and toughening by SiC whiskers are whisker pull-out and bridging (and in some cases the whiskers can be ruptured) and crack deflection. The grain refinement and the dynamic t–m phase transformation of ZrO_2 (2 mol % Y_2O_3) are very important in the toughening of the composites.

References

1. T. N. TIEGS and P. F. BECHER, *Mater. Sci. Res.* **20** (1986) 639.
2. P. F. BECHER, T. N. TIEGS, J. C. OGLE and W. H. WARWICLE, in "Fracture Mechanics of Ceramics", Vol. 7, edited by R. C. Bradt (Plenum Press, New York, 1986) pp. 61–73.
3. T. N. TIEGS and P. F. BECHER, *Amer. Ceram. Soc. Bull.* **66** (1987) 2.339.
4. N. CLAUSSEN and G. PETZOW, *Mater. Sci. Res.* **20** (1986) 649.
5. N. CLAUSSEN and M. V. SWAIN, *Mater. Forum* **11** (1988) 194.
6. M. BOHMER and E. A. ALMOND, *Mater. Sci. Engng* **A105/106** (1988) 105.
7. A. C. SOLOMAHA and W. REICKER, *J. Amer. Ceram. Soc.* **73** (1990) 740.
8. B. CALES, P. MATHIEW and J. P. TOVRE, *Sci. Ceram.* **14** (1987) 171.
9. M. YANG and R. STEVENS, *J. Mater. Sci.* **26** (1991) 726.
10. T. C. LEI and Y. ZHOU, *Mater. Chem. Phys.* **25** (1990) 269.

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