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Effect of Loading Speed on Fracture Behaviour of $ZrO_2(Al_2O_3)$ Matrix Composites Reinforced with SiC Whiskers

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Abstract: The fracture toughnesses, fracture behaviour and toughening mechanisms of 20 vol% SiCw–ZrO_2 (2 mol% Y_2O_3), 20 vol% SiCw–ZrO_2 (6 mol% Y_2O_3), 20% SiCw–Al_2O_3 and 20 vol% SiCw–20 vol% ZrO_2 (2 mol% Y_2O_3)–Al_2O_3 composites (SiCw means SiC whisker) were investigated by three-point bending tests, XRD and scanning electron microscopy techniques. The results show that the fracture toughnesses of the composite materials increase with increasing loading speed because of the increase in the amount of dynamic t→m ZrO_2 transformation and whisker pullout effect. Whisker/matrix characteristis have a significant influence on the effect of loading speed on the fracture behaviour. © 1998 Elsevier Science Limited and Techna S.r.l.

1 INTRODUCTION

The incorporation of β-SiC whiskers into $ZrO_2(Y_2O_3)$, Al_2O_3, $ZrO_2(Y_2O_3) + Al_2O_3$ and other ceramic matrices has been shown to produce significant increases in mechanical strength and fracture toughness.[1–4] The increase in strength can be understood in terms of the reinforcing effect of the whiskers and is dependent on the *in-situ* strength of each phase. The increase in toughness is attributed to several energy-absorbing processes, particulary crack deflection and crack bridging by the whiskers, whisker pullout, and the stress-induced martensitic transformation of tetragonal zirconia grains.[1–6] The operation of various toughening mechanisms depends, to a large extent, on the nature of the whisker/matrix interface.[1,3,4,7,8]

Ceramic matrix composites are increasingly being utilized in structural applications where severe environments are encountered. Their low specific gravity, high hardness, thermal stability, and resistance to corrosion make them important materials in technological application. However, ceramic materials are sensitive to dynamic loading.[9–11] So far, studies in this field are very limited.

The research reported in this article deals specifically with the effect of loading speed on the fracture behaviour of 20 vol% SiCw–ZrO_2 (2 mol% Y_2O_3), 20 vol% SiCw–ZrO_2 (6 mol% Y_2O_3), 20 vol% SiCw–Al_2O_3 and 20 vol% SiCw–20 vol% ZrO_2 (2 mol% Y_2O_3)–Al_2O_3 composites.

2 EXPERIMENTAL

The materials used in this study were ZrO_2 (2 mol% Y_2O_3), ZrO_2 (6 mol% Y_2O_3), α-Al_2O_3 and 20 vol% ZrO_2 (2 mol% Y_2O_3)–Al_2O_3 composites reinforced with 20 vol% SiC whiskers. The β-SiC whiskers were supplied by Tokai Carbon,

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Japan, with a diameter of 0.5 ~ 1 mm and a length of 30 ~ 60 mm. Ultrafine zirconia powders, stablized by 2 or 6 mol% Y_2O_3, had an average particle size of 0.65 mm. Ultrafine Al_2O_3 powders, with a grain size of about 0.1 mm, were shown, by the XRD technique, to be α-Al_2O_3. The 20 vol% SiCw–$ZrO_2(Y_2O_3)$ composites were hot-pressed under a pressure of 25 MPa for 1 h at 1600°C. The SiCw–Al_2O_3 and SiCw–ZrO_2–Al_2O_3 composites were hot-pressed under a pressure of 25 MPa for 1 h at 1650°C. As-received and acid-leached whiskers were incorporated into α-Al_2O_3 matrix, respectively, to elucidate the effect of whisker surface treatment on the properties of the composites. The whiskers were leached with 5 vol% HF acid for 10 h and washed five times with distilled water and absolute alcohol to remove the oxide-rich layer present on the surface of the as-received whiskers.

Fracture toughness measurements of the composites were performed on single-edge notched bar (SENB) specimens (2 mm×4 mm×25 mm) with a span of 16 mm, and a half-thickness notch was made using a diamond wafering blade. The test was conducted at cross-head speeds of between 0.002 mm min^{-1} and 2 mm min^{-1}.

The percentage of t-ZrO_2 in the total ZrO_2 was estimated by XRD on both as-polished and the fractured surfaces. Fracture surfaces of the composites were examined using a Hitachi S-570 scanning electron microscope.

3 RESULTS

The hot-pressing conditions used in this study result in almost theoretically dense composites with a very homogeneous distribution of whiskers. Each of the different materials will be discussed separately.

3.1 Effect of loading speed on fracture behaviour of 20 vol% SiCw–$ZrO_2(Y_2O_3)$ composites

The influence of loading speed on the fracture toughness from 0.002 mm min^{-1} to 2 mm min^{-1} for SiCw–ZrO_2 composites is shown in Fig. 1, indicating that the fracture toughness increased with increasing loading speed. For ZrO_2 (2 mol% Y_2O_3) composite, the fracture toughness increases from 14.67 $MPam^{1/2}$ at 0.002 mm min^{-1} to 17.00 $MPam^{1/2}$ at 0.2 mm min^{-1}. However, in the higher loading speed range of 0.2–2 mm min^{-1}, the fracture toughness of the composite is not sensitive to the loading speed. In contrast, the fracture toughness of ZrO_2 (6 mol% Y_2O_3) composite always increases with increasing loading speed from 0.002 to 2 mm min^{-1}.

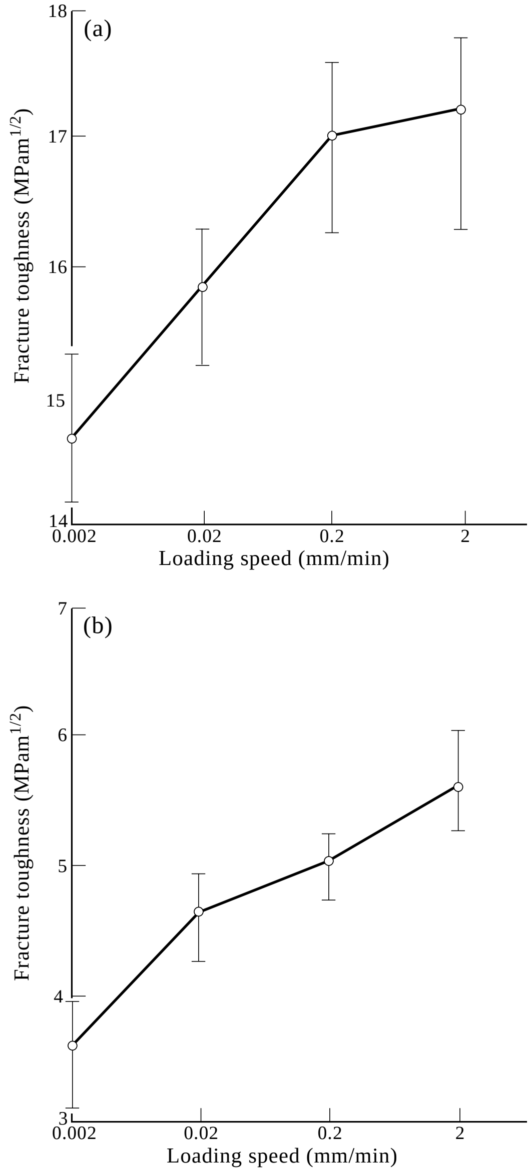

Fig. 1. Effect of loading speed on fracture toughness of $ZrO_2(Y_2O_3)$ + 20vol% SiCw composites: (a) ZrO_2(2Y) + SiCw composites; (b) ZrO_2(6Y) + SiCw composites.

The results of XRD analysis of the fracture surfaces of ZrO_2 (2 mol% Y_2O_3) composite are shown in Table 1. It can be seen that the dynamic t→m ZrO_2 transformation exists at both the low and high loading speed, and the amount of t→m transformation increases with loading speed. The increase in dynamic t→m transformation undoubtedly makes a greater contribution to the fracture toughness of the composite. This is consistent with the measured results of fracture toughness. For ZrO_2(6Y) composite, there is no dynamic t→m ZrO_2 transformation within the whole loading speed range in our study, as shown in Table 2, indicating no effect of transformation toughening.

SEM fractographs of the ZrO_2(2Y) composite are shown in Fig. 2, indicating that the number and length of whisker pullout increase with increasing loading speed, in particular at the lower loading

Table 1. Fracture toughnesses (K_{IC}, $MPam^{1/2}$) of $ZrO_2(Y_2O_3)$+20 vol% SiCw composites at different loading speeds during SENB tests

Materials	Loading speed (v) ($mm\,min^{-1}$)			
	0.002	0.02	0.2	2
ZrO_2(2Y)+20 vol% SiCw	14.67±0.50	15.80±0.55	17.00±0.70	17.20±0.85
ZrO_2(6Y)+20 vol% SiCw	3.60±0.35	4.66±0.30	5.01±0.25	5.60±0.30

speeds [Fig. 2(a) and (b)]. At a higher loading speed, this effect is not apparent [Fig. 2(c) and (d)]. For ZrO_2(6Y) composite (Fig. 3), at a loading speed of 0.002 $mm\,min^{-1}$, the number of whisker pullouts is very small, and the fracture surface is rather smooth [Fig. 3(a)]. With increasing loading speed, the effect of whisker pullout increases at both low and high loading speeds in our studies, which are also consistent with the results of fracture toughness.

The large difference in fracture toughness of ZrO_2 (Y_2O_3)–SiCw composites due to the Y_2O_3 content have been explained very clearly in the previous article.[1]

3.2 Effect of loading speed on the fracture behaviour of 20 vol% SiCw–Al_2O_3 composites

The fracture toughnesses of α-Al_2O_3 matrix composites reinforced with as-received or acid-leached 20 vol% SiCw under different loading speed are summarized in Table 3. It can be seen that the fracture toughnesses of both composites increase with increasing loading speed from 0.002 $mm\,min^{-1}$ to 2 $mm\,min^{-1}$, and the dependence of the composite using as-received SiC whiskers on loading speed is stronger. With increasing loading speed, the difference in fracture toughness between the two types of Al_2O_3 composites disappears, as shown in Fig. 4.

The fracture surfaces of two composites are shown in Figs 5 and 6. For the composite with as-received SiC whiskers, at a lower loading speed (such as 0.002 $mm\,min^{-1}$), the fracture surface was rather smooth, and few pulled-out whiskers were observed [Fig. 5(a)]. However, the effect of whisker pullout obviously increased with increasing loading speed [Fig. 5(b)–(d)], resulting in a large increase in fracture toughness when the loading speed was increased from 0.002 $mm\,min^{-1}$ to 2 $mm\,min^{-1}$. In

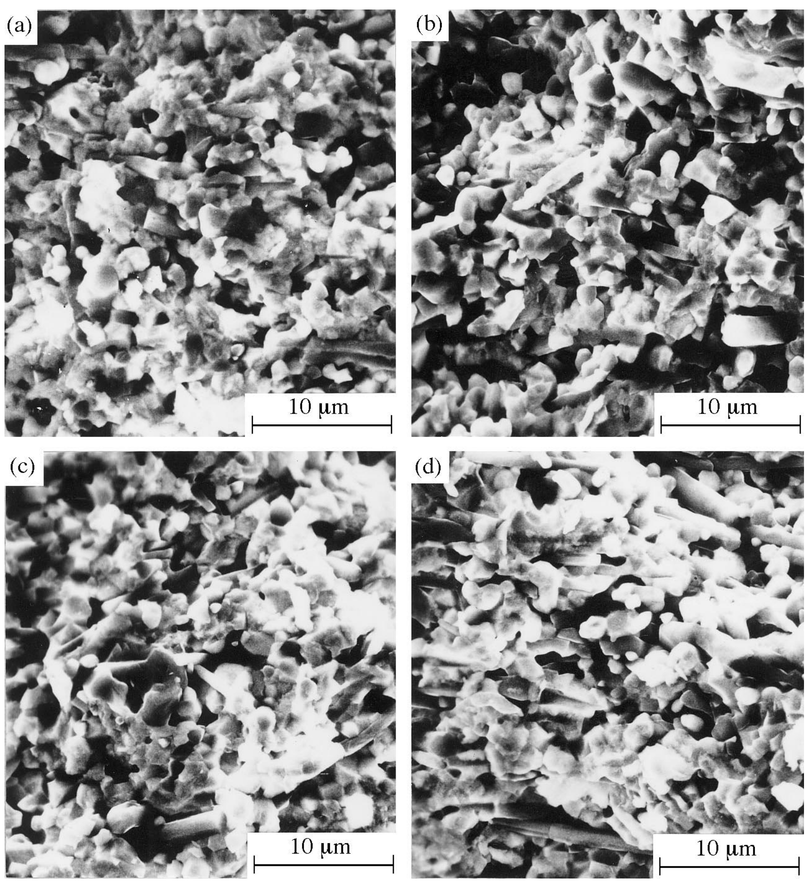

Fig. 2. SEM fractographs of bending fractured surfaces of ZrO_2(2Y) + 20vol% SiCw composite under different loading speeds: (a) 0.002 $mm\,min^{-1}$; (b) 0.02 $mm\,min^{-1}$; (c) 0.2 $mm\,min^{-1}$; (d) 2 $mm\,min^{-1}$.

Table 2. Amount of t→m transformation of ZrO_2 in $ZrO_2(Y_2O_3)$+20 vol% SiCw composites during fracture of SENB tests under different loading speeds

Materials	ZrO_2(2Y)+20 vol% SiCw				ZrO_2(6Y)+20 vol% SiCw			
	Loading speed (v) (mm min^{-1})				Loading speed (v) (mm min^{-1})			
	0.002	0.02	0.2	2	0.002	0.02	0.2	2
Amount of m-ZrO_2 at the polished surface	26.4	26.4	26.4	26.4	0	0	0	0
Amount of m-ZrO_2 at the fracture surface	60.5	61.9	63.5	69	0	0	0	0
Amount of ZrO_2t→m transformation	34.1	35.5	37.1	42.6	0	0	0	0

contrast, the composite produced with acid-leached whiskers exhibited substantial crack/microstructure interactions at a loading speed of 0.002 mm min^{-1}, with some evidence of whisker pullout [Fig. 6(a)]. Although the effect of whisker pullout also increased with loading speed, this increase was less than that of the composite reinforced with as-received whiskers [Fig. 6(b)–(d)]. This may explain the results of mechanical properties as shown in Fig. 4.

3.3 Effect of loading speed on the fracture behaviour of 20 vol% SiCw–20 vol% ZrO_2(2 mol% Y_2O_3)–Al_2O_3 composite

The fracture toughnesses of SiCw–ZrO_2–Al_2O_3 composites under different loading speed are shown in Table 4 and Fig. 7. The effect of loading speed on the fracture toughness of the composite is very sensitive at the lower loading speeds (0.002 and 0.02 mm min^{-1}), resulting in an increase in fracture toughness from 7.39 $MPam^{1/2}$ (0.002 mm min^{-1}) to 9.05 $MPam^{1/2}$ (0.02 mm min^{-1}). Conversely, at the higher loading speed region (0.02 ∼ 2 mm min^{-1}), the magnitude of toughness enhancement observed is smaller than that at the lower loading speed region, only a 0.45 $MPam^{1/2}$ increase in fracture toughness when the loading speed increased from 0.02 mm min^{-1} to 2 mm min^{-1}.

The XRD results of polished and fractured surfaces of SiCw–ZrO_2–Al_2O_3 composites are shown in Table 5, indicating the existence of dynamic t→m ZrO_2(2Y) transformation, which increased

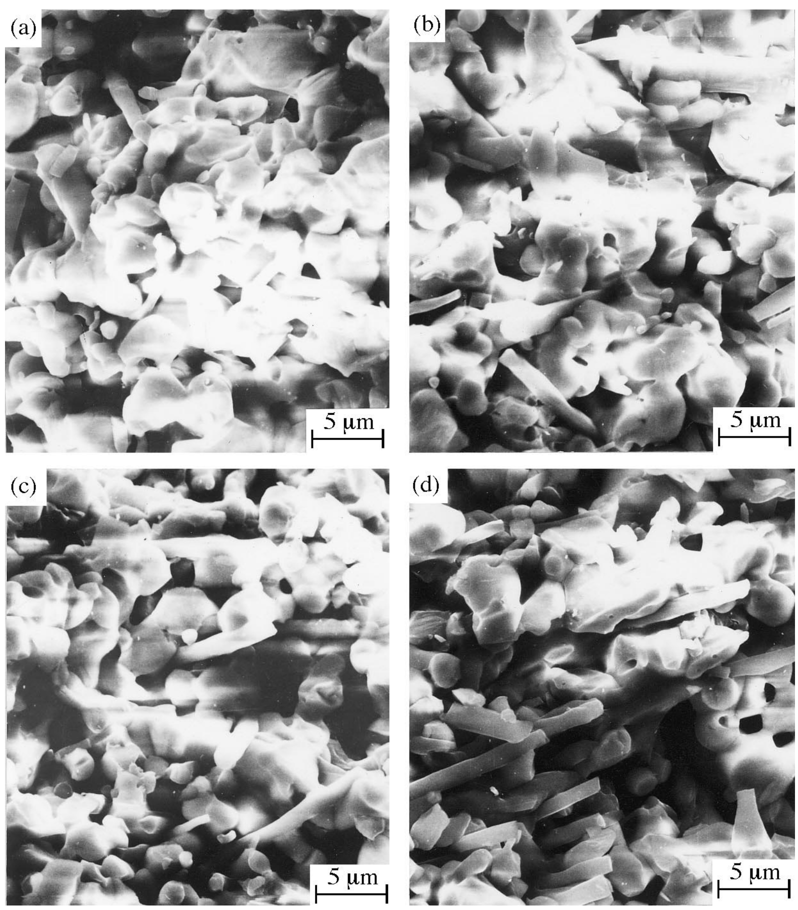

Fig. 3. SEM fractographs of bending fractured surfaces of ZrO_2(6Y) + 20vol% SiCw composite under different loading speeds: (a) 0.002 mm min^{-1}; (b) 0.02 mm min^{-1}; (c) 0.2 mm min^{-1}; (d) 2 mm min^{-1}.

Fig. 4. Effect of loading speed on fracture toughness of $Al_2O_3 + 20$vol% SiCw composites.

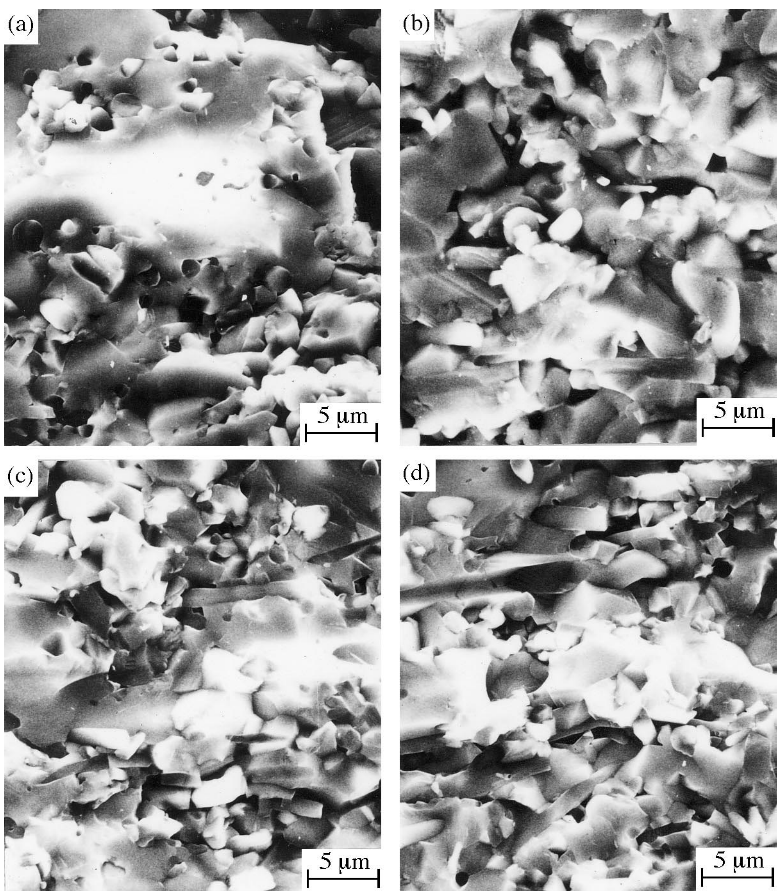

Fig. 5. SEM fractographs of bending fractured $Al_2O_3 + 20$vol% SiCw (as-received) composite under different loading speeds: (a) $0.002\,mm\,min^{-1}$; (b) $0.02\,mm\,min^{-1}$; (c) $0.2\,mm\,min^{-1}$; (d) $2\,mm\,min^{-1}$.

zirconia polycrystals (TZP) and zirconia-toughened ceramics (ZTC) materials.[12] Becher[13] concluded that the transformation-toughening contribution to the materials fracture resistance is:

$$\Delta K_{IC}^{T} = A\, B^{1/2} V_f\, E_c\, e^{T}(K_{IC}^{M}/\sigma_C^{T}). \quad (1)$$

The terms, A and B are constants related to the stress state, V_f is the fraction of tetragonal phase that transforms, E_C is Young's modulus of the composite, e^T is the transformation strain, K_{IC}^M is the fracture toughness of the matrix and σ_C^T is the

Table 3. Fracture toughnesses (K_{IC}, $MPam^{1/2}$) of Al_2O_3+20 vol% SiCW composites at different loading speeds

Materials	Loading speed (v) ($mm\,min^{-1}$)			
	0.002	0.02	0.2	2
Al_2O_3+received SiCw	6.02±0.30	6.78±0.28	8.02±0.45	9.00±0.40
Al_2O_3+acid-leached SiCw	8.04±0.55	8.68±0.57	8.95±0.30	9.10±0.45

Table 4. Fracture toughness (K_{IC}, $MPam^{1/2}$) of Al_2O_3+20 vol% ZrO_2(2Y)+20 vol% SiCw composites at different loading speeds

Materials	Loading speed (v) ($mm\,min^{-1}$)			
	0.002	0.02	0.2	2
Al_2O_3+20 vol% ZrO_2(2Y)+20 vol% SiCw	7.39±0.34	9.05±0.70	9.25±0.55	9.50±0.40

Table 5. Amount of t→m transformation of ZrO_2 in Al_2O_3+20 vol% ZrO_2(2Y)+20 vol% SiCw composites during fracture of SENB tests under different loading speeds

Loading speed (v) ($mm\,min^{-1}$)	0.002	0.02	0.2	2
Amount of m-ZrO_2 at the polished surface	68.5	68.5	68.5	68.5
Amount of m-ZrO_2 at the fracture surface	71.2	78.0	79.5	83.4
Amount of ZrO_2t→m transformation	2.7	9.5	11	14.9

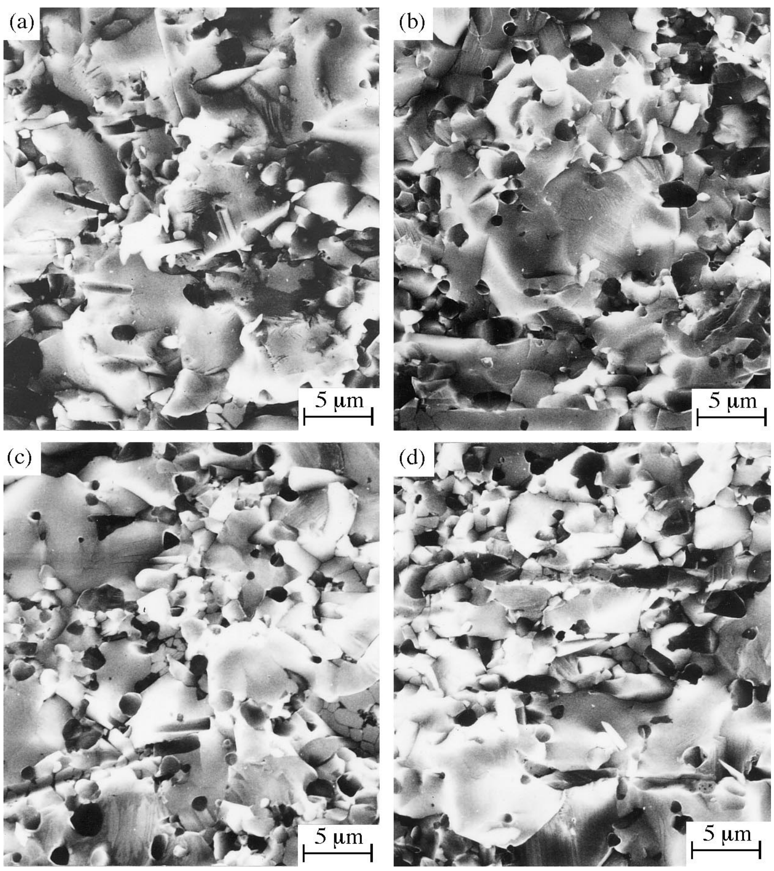

Fig. 6. SEM fractographs of bending fractured Al_2O_3 + 20vol% SiCw (acid-leached) composite under different loading speeds: (a) 0.002 mm min^{-1}; (b) 0.02 mm min^{-1}; (c) 0.2 mm min^{-1}; (d) 2 mm min^{-1}.

Fig. 7. Effect of loading speed on fracture toughness of $Al_2O_3 + 20vol\%$ $ZrO_2(2Y) + 20vol\%$ SiCw composite.

increasing leading speed, the increasing effect of whisker pullout is not obvious, especially at higher loading speeds [Fig. 2(c) and (d)]. In contrast, for ZrO_2(6Y) composites, the strong interface bonding due to the interface amorphous layer restrains the whisker pullout.[1] This was confirmed by the limited whisker pullout at low loading speed [Fig. 3(a)]. With increasing loading speed, the number of whisker pullouts increased greatly, indicating that whisker pullout is sensitive to loading speed.

For SiCw–Al_2O_3 composites, the acid-leaching treatment on SiC whiskers decreased the amount of SiO_2 and other impurities on the whiskers surface,[14,15] resulting in a weak interface bonding in the composite. Therefore, for the Al_2O_3 composite with acid-leached whiskers, even at low loading speeds, the number of whisker pullouts is large [Fig. 5(a)] and is not sensitive to the loading speed [Fig. 5(b)–(d)] which is similar to ZrO_2(2Y)–SiCw composites. For the composite with as-received whiskers, the strong interface bonding inhibited the whisker pullout.[14] The effect of whisker pullout is sensitive to loading speeds in the range of loading speeds in our study (Fig. 6).

As stated above, the dynamic t→m ZrO_2 transformation and effect of whisker pullout increased with increasing loading speed, resulting in the increase in fracture toughness.

5 CONCLUSIONS

1. In the range of loading speed in our study (from 0.002 mm min^{-1} to 2 mm min^{-1}), the fracture toughnesses of ZrO_2(2Y), ZrO_2(6Y), Al_2O_3 and Al_2O_3 + 20 vol% ZrO_2(2Y) matrix composites reinforced with 20 vol% SiCw increase with increasing loading speed.
2. A high loading speed promotes the dynamic t→m ZrO_2(2Y) transformation during fracturing and hence increases the transformation toughening contribution to the fracture toughnesses of the composites.
3. The increase in loading speed promotes the effect of whisker pullout. The characteristic of whisker/matrix interfaces has a strong influence on the effect of whisker pullout on loading speed. The composite with strong interface bonding is more sensitive to loading speed than that with weak interface bonding.

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