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# Fabrication, characterization and mechanical properties of SiC–whisker–reinforced Y–TZP composites

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The microstructure and mechanical properties of hot-pressed yttria-stablized tetragonal zirconia polycrystals (Y–TZP) reinforced with up to 30 vol % SiC whiskers were investigated. The homogeneously dispersed and fully dense SiC whisker/Y–TZP composites were fabricated by wet-mixing the constitutents and uniaxially hot-pressing the resulting powder. The grain size of the matrix depended on the whisker volume fraction and the hot-pressing temperature. The significant increase of fracture toughness of about MPa $m^{1/2}$ at 10 Vol % SiC and a small increase in strength were achieved by uniformly dispersing the whiskers in the Y–TZP matrix. Fracture surfaces revealed evidence of toughening by the mechanisms of crack deflection, pullout, and crack bridging by the whiskers and also a phase transformation of $ZrO_2$. The observed increase in the fracture toughness of Y–TZP due to the addition of SiC whiskers was correlated with existing models of toughening mechanisms. Good agreement was achieved between the theoretical predictions and the experimental toughness values, obtained from the Y–TZP/$SiC_w$ composites.

## 1. Introduction

Tetragonal zirconia polycrystalline ceramics have high fracture strength and fracture toughness [1–5]. Unfortunately, these excellent mechanical properties are superior to many other ceramic materials only at ambient temperatures. With increasing temperature, the properties of all the TZP ceramics degrade severely due to the boundary softening of the glass-wetted grains [6] and the fact that the stress-induced transformation become less effective. One possible method of improving the high-temperature strength is to add reinforcements into the materials. Fibre-reinforcing seems to be one of the most promising potential strategies [6] to strengthen $ZrO_2$-toughened ceramics at high temperatures. In order to study its applicability to TZP ceramics, SiC whiskers are used in the present work. Phase equilibrium calculations [7] indicated that the system $ZrO_2$–SiC was stable under the anticipated hot-pressing conditions. Although the toughening effect in fibre reinforced ceramic matrix composites (CMCs) are higher than those achieved by whisker reinforcement, whisker reinforcement offers the advantages of easier fabrication and a higher degree of isotropy in material properties due to the smaller aspect ratios involved. Previous studies on whisker reinforced CMCs suggested the possibility of five toughening mechanisms being operative. These mechanisms are crack deflection [8–10], crack bowing [9], microcracking [11–12], whisker pullout [13], and crack bridging [14–17]. Nearly fully dense, well-dispersed SiC-whisker-reinforced Y–TZP composites with fracture toughness values of up to twice the matrix value were fabricated [6]. The present work studies the mechanisms of toughening and strengthening in the SiC whisker $ZrO_2$ composites, and some theoretical calculations are made on the basis of an approach that combined individual contributions from effective toughening mechanisms.

## 2. Experimental procedure

SiC whiskers of 0.1–0.5 μm diameter, 4–10 μm in length were used for reinforcing Y–TZP. The whiskers were first dispersed by ultrasonic vibration in absolute alcohol. The whiskers were mixed with 3 mol % $Y_2O_3$ powder by the same tumbling procedure. The TZP powder consisted of tetragonal particles, typically $< 0.4$ μm in size. Powder mixture containing 10–30 vol % SiC whiskers were uniaxiallay hot-pressed to $> 99\%$ the theoretical density in BN-coated graphite dies at 1650 °C under a pressure of 25 MPa for 40 min in an argon atmosphere. Densities of the hot-pressed disks were measured by the Archimedes method. The degree of dispersion of the SiC whiskers and the grain-size distributions were evaluated by optical microscopy. The grain size of the

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matrix was estimated by a linear intercept method. The average distance between grain boundaries along randomly drawn lines intersecting at least 150 grain boundaries multiplied by a factor of 1.5 was taken as the grain size.

Rectangular bend bars ($3 \times 4 \times 40$ mm) were cut from the hot-pressed disks and the tensile surface were polished. Flexural strength and toughness were measured at room temperature using these bars. The fracture toughness of six to ten samples were measured for each whisker addition and processing condition. The measurements of 3-point bend strength were performed using a 30 mm span and a crosshead speed of 0.5 mm $min^{-1}$. The room temperature fracture toughness was also measured in a 3-point bending test configuration using a 30 mm span and a crosshead speed of 0.05 mm $min^{-1}$. The chevron notches were prepared with a 0.2 mm thick diamond saw blade. The fracture surfaces were analysed by scanning electron microscopy (SEM) X-Ray diffraction using CuK$\alpha$ radiation and the microstructure at the whisker/matrix interfaces was examined by transition-electron microscopy (TEM). The fraction of monoclinic zirconia phase was calculated from the ratio of the diffraction intensities of the monoclinic (111) and (11$\bar{1}$) peaks to the tetragonal (111) peak [10].

## 3. Results

### 3.1. Microstructural characterization

#### *3.1.1. SEM microstructure features of the composites*

Composites with 10–30 vol % whiskers were densified to 99.5% of theoretical density (TD). Metallography observations indicated that the distribution of whiskers was homogeneous with a preferred long-axes orientation perpendicular to the hot-pressing direction. The composites were free from large pores, although voids associated with whisker agglomerates were occasionally observed. The SiC whiskers in Y–TZP retained their rod-like morphology after hot-pressing.

Fig. 1(a–c) show SEM micrographs of the fracture surfaces of the hot-pressed composites with 10, 20, and 30 vol % SiC whiskers, respectively. Fracture of the whiskers due to the hot-pressing was rarely observed. Protruding whiskers and holes where whiskers were lodged prior to fracture were observed; these are evidence of whisker pullout and crack bridging. Most whiskers were located between Y–TZP grains as is shown in Fig. 1c. The fracture mode was a mixture of transgranular and intergranular types with the degree of transgranular fracture decreasing with increasing whisker content.

Fig. 2 is an SEM micrograph that shows the path of a crack produced by Vickers indentation at a surface of a 10 vol % SiC whisker composite. The fracture propagated in a very ragged way in the composite. In contrast, the fracture surfaces of the pure TZP samples were relatively smooth. The crack is deflected along the whisker/matrix interface. The interfacial crack probably grows at a debonded whisker in the wake of the crack-tip [18]. Crack deflection along a grain

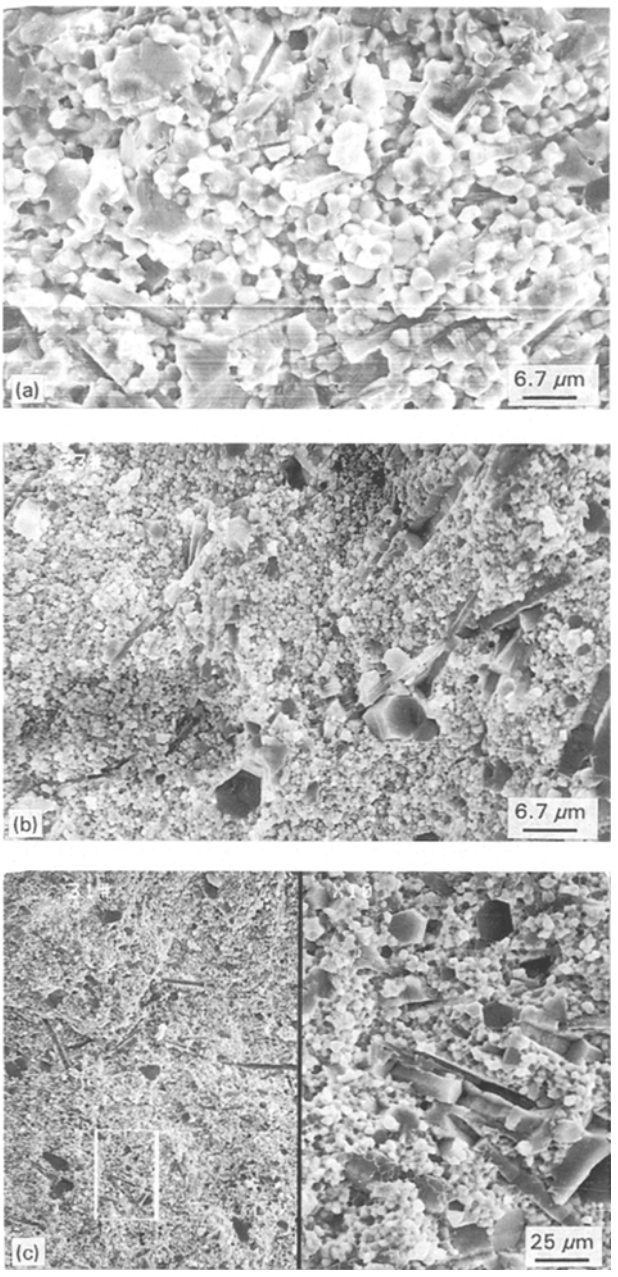


*Figure 1* Scanning Electron Micrographs of the fracture surface of (a) 10, (b) 20, and (c) 30 vol % SiC whisker/Y–TZP composites

boundary was also observed. Whiskers were pulled out of the matrix from one crack face in the wake of the crack tip, leaving protruding whiskers and associated holes (Fig. 2). Crack deflection around SiC and whisker pull-out appear to be the dominant toughening mechanisms, as will be discussed later.

Crack bridging by the whiskers is also a likely mechanism of toughening because the whiskers are under residual compressive stresses due to the $SiC_w$/Y–TZP thermal expansion mismatch ($\alpha_{sic} = 4.7 \times 10^{-6}\,K^{-1}$ and $\alpha_{tzp} = 10 \times 10^{-6}\,K^{-1}$), and they are effectively strengthened. The Y–TZP matrix is in tension along the direction of the long axis of the whiskers. Qualitative analysis of micrographs, similar to those shown in Figs 1 and 2, showed that the maximum deflection distance (the distance that a crack travels along the whisker/matrix interface before whisker fracture) and therefore the maximum

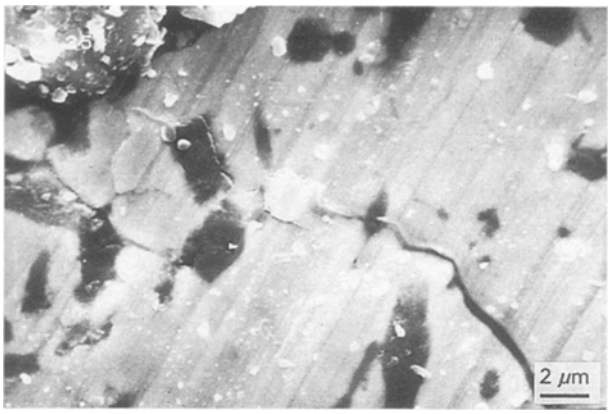


*Figure 2* SEM micrograph showing the interaction of a crack with whisker in a 10 vol % whisker content

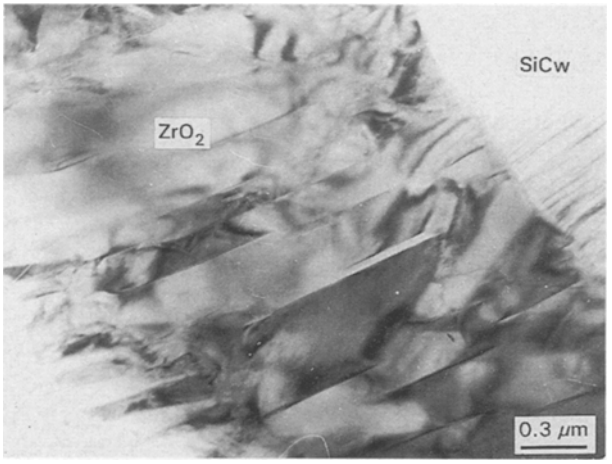


*Figure 3* TEM micrograph of the Y–TZP grain close to the whiskers showing *t-m* transformation and microcracks

pullout length were limited to about two whisker diameters for all processing conditions.

### 3.1.2. TEM microstructure features of the composites

Fig. 3 shows that most of the grains close to the whiskers had transformed to monoclinic symmetry with microcracking along the grain boundaries. The microcracks, which were caused by the thermal mismatch stresses and/or a phase transformation from tetragonal to monoclinic symmetry during cooling, may contribute to the toughening to a certain extent.

The TEM micrograph in Fig. 4 shows a thin interaction layer (0.1–1.0 μm in thickness) at the whisker/matrix interface. The thickness of the layer varies at the interfaces between the TZP grains and SiC whiskers. Whiskers are believed to be strongly bonded with the matrix, as the interface layers were found to be amorphous-like (Fig. 4) and contain elements like Y, Al and Fe in addition to Si and Zr, and even Zr and Si were detected respectively in SiC whiskers and Y–TZP grains with energy dispersive analysis with X-rays (EDX). In addition, dislocations and some precipitates in the TZP grains, and the interaction between them were observed (Fig. 5).

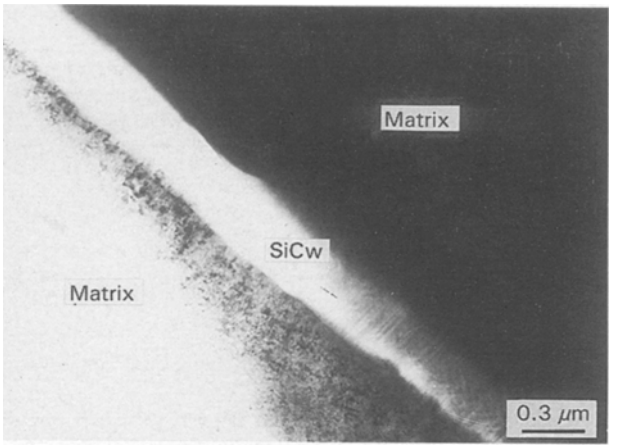


*Figure 4* TEM micrograph whisker/matrix interface showing a thin interaction layer

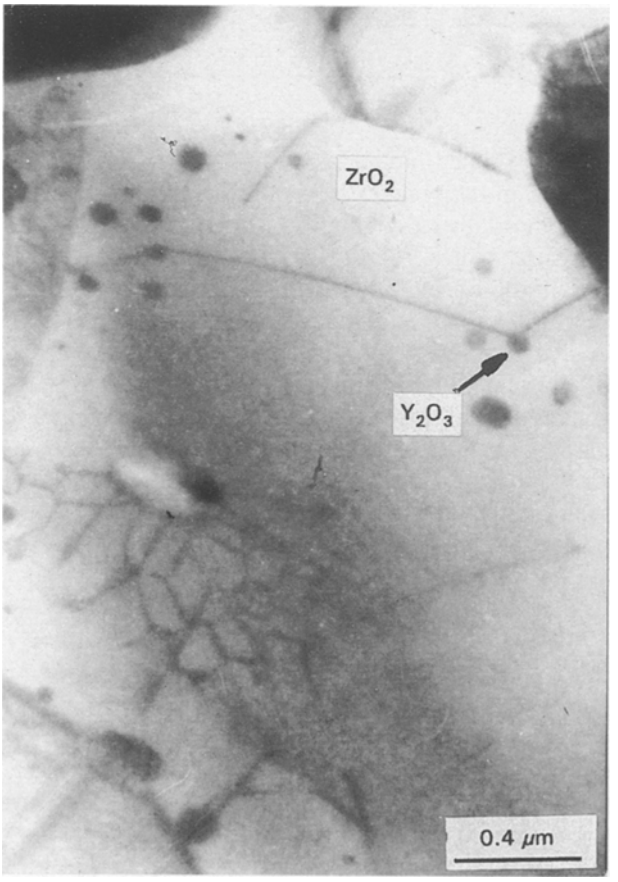


*Figure 5* TEM micrograph 30 vol % $SiC_w$/Y-TZP showing dislocations and some precipitates in the Y–TZP matrix and the interaction between them

### 3.1.3. Effects of SiC whisker content on phase transformation

From those observations as shown in Figures 1–5, it appears that whisker incorporation results in crack deflection, whisker pullout, and crack bridging mechanisms, which operate in conjunction with one another. The phase content of the composites was measured by X-ray diffraction from fracture surfaces (Table I). When compared with the original samples, the results indicate that the metastable tetragonal phase content decreases with increasing whisker content. The residual

TABLE I $m$-phase content in $SiC_w$/Y–TZP composites on polished surface and fracture surface

| Whisker content (vol %) | Polished surface $m$-phase (%) | Fracture surface $m$-phase (%) |
|---|---|---|
| 0 | 2 | 27 |
| 10 | 10.7 | 32 |
| 20 | 21.3 | 31 |
| 30 | 25.9 | 30 |

TABLE II Variation of grain size in composites with whisker content and hot-pressing temperatures

| Whisker content (vol %) | Grain size at 1550 °C (μm) | Grain size at 1600 °C (μm) | Grain size at 1650 °C (μm) |
|---|---|---|---|
| 0 | 0.60 | 0.85 | 1.20 |
| 10 | 0.80 | 1.50 | 2.00 |
| 20 | 0.71 | 1.38 | 1.62 |
| 30 | 0.55 | 1.36 | 1.60 |

tensile stresses induced by the differential thermal expansion between $ZrO_2$ and SiC have led to transformation during cooling from the hot-pressing temperature, which results in a sluggish $t \rightarrow m$ transformation during fracture. However, for less than 20 vol % SiC content, the $t \rightarrow m$ transformation is still an effective toughening mechanism. Above 20 vol % $SiC_w$ content, the whiskers in the Y–TZP matrix prevent the $t$-phase from transforming during the fracture process. In contrast, Claussen [6] observed no $m$-$ZrO_2$ phase on the fracture surfaces, and concluded that the increase in fracture toughness of the composites originates solely from the whiskers.

### *3.1.4. Effect of whisker content and hot-pressing temperature on grain size*

The grain size of the TZP matrix is very important for the phase transformation related toughening effect. It was found that the grain size is considerably influenced by the whisker content and the hot pressing temperature, as is shown in Table II. For composites containing whiskers, the average grain size was found to increase with increasing whisker content up to 10 vol %, and then to decrease considerably for higher SiC whisker contents. The largest increase in grain size of Y–TZP was found at 10 vol % SiC whisker addition. This increase is probably resulted from a liquid phase at the hot pressing temperature formed by the reaction between the SiC whiskers and the matrix. Above a 20 vol % SiC whisker content, the pinning effect of the whiskers on the grain growth of TZP become more significant and therefore causes the observed reduction to the TZP grain sizes.

The average grain size was found to increase remarkably with an increase in hotpressing temperature for all the whisker composites. The average grain size of the 10 vol % $SiC_w$/Y–TZP composite, sintered at 1550 °C for 40 min, is 0.8 μm, compared with 2.00 μm

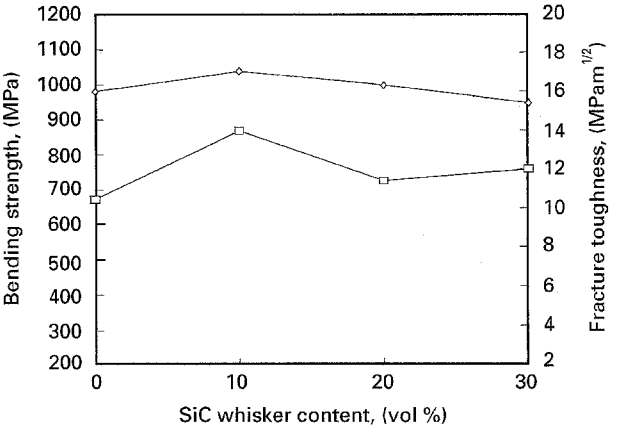


*Figure 6* Room-temperature strength (◇) and fracture toughness (□) of $SiC_w$/Y–TZP composites as a function of whisker content a 1650 °C hot-pressing temperature

for the sample sintered at 1650 °C. For a 10 vol % $SiC_w$/Y–TZP composite, the fracture strength and toughness increase with increasing grain size. For 20 and 30 vol % $SiC_w$/Y–TZP composites, both the toughness and strength remain almost constant although the grain size changes with the hot-pressing temperature.

## 3.2. The mechanical properties of the composites

The room-temperature toughness and strength of sample with 0, 10, 20, and 30 vol % SiC whiskers are given in Fig. 6. The experimental results indicate that the addition of whiskers to Y–TZP at around 10 vol % leads to an increase in both the fracture strength and toughness of the Y–TZP. The highest bend strength and $K_{1c}$ values, observed for a 10% $SiC_w$/Y–TZP sample which was hot pressured at 1650 °C, are thought to be mainly related to its large grain size of about 2.00 μm, and also to the homogeneous distribution of the $SiC_w$ in the Y–TZP matrix (Fig. 1). The larger grain size favours a higher phase transformation volume (Table I), while the homogeneous distribution of the whiskers, which is of primary importance to the successful toughening of ceramics by whisker reinforcement, limits the whisker agglomeration effect.

Above 20 vol % whisker content, the fracture toughness and strength decrease. Claussen [6] also observed a decrease in the strength of Y–TZP with the addition of whiskers, with the fracture strength being reduced to almost half of the strength of the TZP matrix by the incorporation of the whiskers. One of the main reasons for this reduction is the high tensile stresses in the matrix due to the thermal mismatch. At room temperature, the average thermal mismatch stress in the matrix is $\sigma_m = 550$ MPa for the 30 vol % whisker content in our materials, and local stresses may be much higher than this value. Whisker clustering may be another reason for the reduction of the fracture strength. Whisker agglomerates are usually large in size and will probably act as the origin of fracture [21], thus limiting the fracture strength of the

composite especially at higher whisker contents. Low density regions were indeed observed in the hot-pressed material containing 30 vol % whiskers [21].

## 4. Discussion

### 4.1. Whisker pull-out

Debonding is a prerequisite for whisker pull out and thereby the toughening of the material, but the properties of the composites are dominated by the sliding resistance of a debonded interface during the pull out, which dictates the major contribution to toughness caused by the pull-out [23]. Therefore the effect of pull-out on toughening, as calculated in the present study, involves the contributions of both debonding and other processes involved in the pull out of a whisker. The features observed at fracture surfaces were used to calculate the contributions of various toughening mechanisms for composites after a 1600 °C hot-pressing temperature. Whisker pullout is frequency observed in the composites. The fracture surfaces of the composites shown in Fig. 1 contain producing whiskers, which indicate the operation of toughening via a pullout mechanism. Because the whiskers were oriented randomly in the plane perpendicular to the hot-pressing direction, not all of the whiskers could be involved in the pullout process. The importance of the pull-out contribution to toughening was estimated by counting the number of pullouts from SEM micrographs of the fracture surfaces. For composites with 10, 20, and 30 vol % whiskers, the number of pulled-out whiskers per unit projected area of fracture surface, $N_A$, are 0.018, 0.025, and 0.028 pullouts $\mu m^{-2}$ respectively.

Becher *et al.* [19] evaluated whisker pull-out toughening by considering the work done by sliding the whiskers. The increase in toughening was determined by the relation [19]:

$$G^{po} = (4v_f\tau_i^2 l_{po}^3)/(3E_w r^2) \quad (1)$$

where $y_f$ is the whisker content $l_{po}$ is the pullout length, $E_w$ is the elastic modulus of the whiskers, $r$ is the whisker radius and $\tau_i$ is the whisker/matrix interfacial shear strength. $\tau_i$ can be expressed by the equation [6]:

$$\tau_i = \mu\sigma_m = \mu(\alpha_m - \alpha_w)E_w V_f \Delta T_g/\{1 + V_f(E_w/E_m)\} \quad (2)$$

where $\mu$ is the coefficient of friction, $E_m$ is Young's modulus of the matrix, $\Delta T_g (= 1200\,°C)$ is the temperature below which no interfacial relaxation take place, $\alpha_m$ $(10 \times 10^{-6}\,K^{-1})$ and $\alpha_w$ $(= 4.7 \times 10^{-6}\,K^{-1})$ are coefficients of thermal mismatch for the matrix and whisker, respectively.

For a unit width of a crack front and process zone length $d$, the energy dissipated for incremental crack extension is $\delta w = N_A F\, d\, \delta y$, where $\delta y$ is the mean whisker-matrix displacement and $F$ is the mean shear force on the whiskers ($F = 2\pi r l_{po}\tau_i$). The first approximation of $\delta y$ gives $\delta y = (u^*/d)\delta c$, where $u^*$ is the crack opening displacement at the end of the process zone given by $u^* = K_{Ic}^2/E\sigma_s$. Thus, the energy release rate for pull-out is given by [21]:

$$G^{po} = \delta u/\delta c = N_A F u^* \quad (3)$$

As noted earlier, $r = 0.3\,\mu m$ and $l_{po}$ is 3.2, 1.07, and 1.5 μm respectively for 10, 20 and 30 vol % whisker contents. An upper-bound approximation of $u^*$ is the pull-out length. Inserting the appropriate values into Equation 3 gives $K^{po} = (E_c G^{po})^{1/2} = 3.58$, 5.28, and 5.41 MPa $m^{1/2}$ respectively for 10, 20, and 30 vol % composites, as shown in Table IV. The calculated contribution due to pull-out is an upper-bound estimate because the pull-out length is assumed to be the maximum for all whiskers involved in the process, and the crack opening displacement is also taken as the maximum observed pull-out length. The amount of whisker pull-out will depend on the composition and processing parameters. We have observed substantial whisker pull-out in our composites. Pull-out toughening can be promoted by increasing the pull-out lengths. This can be done by preventing the formation of interfacial layers between the whiskers and the matrix, and using whiskers with a smooth surface to prevent mechanical locking.

### 4.2. Crack bridging

Crack bridging phenomena have been observed in various composites [14–17]. The present experimental SEM results also confirm the existence of crack bridging in whisker toughened composites. The bridging of crack faces by whiskers in the wake of the crack tip has been shown to be an important toughening mechanism. The increase in critical strain energy release rate due to the elastic stretching of partially debonded, fractionally constrained reinforcements can be given by [20–21]:

$$G^{wb} = V_{wb} r (S_w)^3/(6E_w\tau_i) \quad (4)$$

where $V_{wb}$ is the volume fraction of the bridging whiskers, $r$ is the whisker radius, $S_w$ is the whisker strength, $E_w$ is the elastic modulus of the whiskers, and $\tau_i$ is the interfacial shear strength. We have calculated whisker toughening from Equation 4 using $S_w = 1$ GPa and other parameters shown in Table III.

This calculation represents an upper-bound estimate of toughening by whisker bridging because not all the whiskers are oriented so as to participate in the bridging process. Calculations of the toughening effect by whisker bridging and pull-out, based on the measurement of pull-out/bridging sites on fracture surfaces in the present paper, indicate that the mechanism of pull-out contributes more significantly than

TABLE III Experimental parameters for the calculation of toughening mechanism

| Whisker content (vol %) | $E_c$ (GPa) | $r$ (μm) | $S_w$ (GPa) | $l_{po}$ (μm) | $N_A$ (pullouts $\mu m^{-2}$) |
|---|---|---|---|---|---|
| 10 | 210 | 0.3 | 1.0 | 3.2 | 0.018 |
| 20 | 220 | 0.3 | 1.0 | 1.07 | 0.025 |
| 30 | 230 | 0.3 | 1.0 | 1.56 | 0.028 |

TABLE IV Toughening results for different mechanisms at a 1650 °C hot-pressing temperature

| Whisker content (vol %) | $dK^{po}$ (MPa.m$^{1/2}$) | $dK^{wb}$ (MPa.m$^{1/2}$) | $dK^{d}$ (MPa.m$^{1/2}$) | $dK^{trans}$ (MPa.m$^{1/2}$) |
|---|---|---|---|---|
| 0 | 0 | 0 | 0 | 4.55 |
| 10 | 3.58 | 0.13 | 1.87 | 3.19 |
| 20 | 5.28 | 0.20 | 2.18 | 1.60 |
| 30 | 5.41 | 0.32 | 2.35 | 0.83 |

that of bridging, as supported by the experimental observations.

### 4.3. Crack deflection

Crack deflection is commonly observed in whisker reinforced ceramics [8–10] regardless of the matrix type. Faber and Evans [9] have formulated a model for the evaluation of toughening due to crack deflection by randomly arranged rod-shaped particles. Evidence of crack deflection by strong whiskers has been demonstrated by an increase in crack-front deflection angles in whisker composites as compared with deflection angles of the unforced materials [10]. The analytical model of Faber and Evans [9] suggests that the main crack is evenly deflected at all angles up to a maximum of 90°, if undeflected cracks are ignored. The toughness contribution due to crack deflection for all composites, evaluated from the analysis of Bengisu *et al.* [22] is shown in Table IV. The degree of intergranular fracture increased with increasing SiC whisker addition; thus, the toughening due to crack deflection by Y–TZP grains was increased. In contrast, Smith *et al.* [21] discovered that the toughening due to crack deflection by alumina grains was reduced with whisker content. It was therefore expected that the net deflection contribution due to SiC whisker addition would be lower than that predicted by the analysis.

The present results indicate that whisker toughening results mainly from the combination of three mechanisms, namely whisker pull-out, crack deflection, and crack bridging. Consequently, an attempt was made to define whisker toughening as the additive contributions of these mechanisms. It was assumed that the three mechanisms do not interact either positively or negatively since occurrence of either phenomenon could not affect any parameters of the toughening models to a large extent. It was also assumed that the whiskers are uniformly distributed in the matrix and that their mechanical and physical properties do not deviate significantly from the average values used in the calculations. Our SEM observations of crack propagation revealed that during crack growth and opening, all of these processes can occur simultaneously. Based on these assumptions, the total toughening on whisker reinforcement may be calculated by simply adding the individual contributions together since fracture toughness is an additive term for a crack subjected to mode I loading [21], that is

$$dK_{total} = dK_{pullout} + dK_{deflection} + dK_{bridging} \quad (5)$$

### 4.4. Transformation toughening

Considering the presence of the transformation toughening in the matrix at whisker additions up to 10 vol % $SiC_w$, contributions from the transformation toughening ($dK_{trans}$) should be included in Equation 5. According to Ref. [23] and the results of Table I, the toughening values from transformation were 4.55, 3.19, 1.60 and 0.86 MPa.m$^{1/2}$ for 0, 10, 20 and 30 vol % $SiC_w$/Y–TZP, respectively. Assuming that all the toughening contributions are additive, the predicted upper-bound increase in fracture toughness due to addition of SiC whiskers and transformation toughening should be expressed as follows:

$$dK_{total'} = dK_{pullout} + dK_{deflection} + dK_{bridging} + dK_{trans} \quad (6)$$

For the addition of 10 vol % SiC whiskers, the predicted upper-bound increase in fracture toughness was 8.77 MPa.m$^{1/2}$. The experimentally observed increase in toughness of Y–TZP due to the addition of whiskers and transformation toughening was 9.13 MPa.m$^{1/2}$. Considering the numerous assumption made in the above calculations, and other toughening mechanisms, such as microcracking, there is good agreement between the calculated and measured toughness increases.

## 5. Conclusions

1) SiC whisker reinforced Y–TZP composites containing up to 30 vol % SiC whiskers were hot-pressed to 99.5% of theoretical density at 1650 °C for 40 min.

2) The addition of SiC whiskers increased both the room temperature fracture strength and toughness of Y–TZP for less than 20 vol % SiC whisker content.

3) The grain size depended on the whisker content and the hot-pressing temperature. The largest grain sizes were obtained at a 10 vol % whisker content and thereafter it decreased with increasing whisker content.

4) SiC Whiskers at less than a 20 vol % content in the Y–TZP matrix do not significantly prevent *t*-phase transformation during the fracture process. The SiC whiskers reacted with the Y–TZP grains and formed amorphous like layers.

5) Comparison of the experimental data with theoretical predictions showed that whisker pullout, crack deflection and transformation toughening are the main toughening mechanisms. The predicted magnitudes are in reasonable agreement with the measured values.

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