

The influence of TiC-particle-size on the fracture toughness of Al₂O₃–30 wt.%TiC composites

Jianghong Gong*, Hezhuo Miao, Zhe Zhao

State Key Laboratory of New Ceramics and Fine Processing, Department of Materials Science and Engineering, Tsinghua University, Beijing 100084, PR China

Received 19 October 2000; received in revised form 2 December 2000; accepted 16 December 2000

Abstract

Seven grades of Al₂O₃–30 wt.% TiC composites were prepared by hot-pressing the Al₂O₃ powder mixed with TiC particles. The average sizes of the TiC particles used for preparing these composites were different with each other. The fracture toughness K_{IC} was measured in three-point bending for each composite. It was shown that the variation in the size of the TiC particles results in large changes in fracture toughness and there is a particular particle size for which the fracture toughness of the resultant composite reaches its maximum. A possible toughening mechanism was also proposed to explain these experimental findings. © 2001 Elsevier Science Ltd. All rights reserved.

Keywords: Al₂O₃; Mechanical properties; TiC; Toughness and toughening

1. Introduction

It has been well known that alloying of brittle materials to produce a two-phase structure may result in considerable improvement in their fracture toughness and several models have also been proposed to explain these experimental results theoretically.^{1,2} However, previously published papers have thus far emphasized the effect of the content of the second-phase particles on the mechanical properties of two-phase brittle ceramics. Little effort has been devoted to the observation and analysis of the effect of the size of the second-phase particles. As a preliminary study, the fracture toughnesses of composites in the system Al₂O₃–TiC were measured and compared in this paper. As will be shown later, the mechanical properties of the examined composites exhibit strong TiC-particle-size dependence.

Composites of Al₂O₃–TiC, which consist of finely dispersed titanium carbide grains in an alumina matrix, are being used in many applications, especially as excellent cutting tools.^{3,4} During the past years, numerous studies have been carried out to examine the effect of

TiC particle content on the microstructures, the properties and the cutting performance of the resultant Al₂O₃–TiC composites.^{4–9} It has been generally concluded that the highest bending strength can be achieved by adding about 30 wt.% TiC in Al₂O₃ matrix⁷ while the fracture toughness of Al₂O₃–TiC composite increases with increasing volume fraction of TiC.^{5,7} Based on these observations, the system Al₂O₃–30 wt.% TiC was chosen for the present study to observe the effect of TiC particle size on the mechanical properties of the composites.

2. Experimental

Seven grades of Al₂O₃–30 wt.% TiC composites were prepared. The basic raw materials used for preparing these composites were a commercial Al₂O₃ powder with an average grain size of 0.5 μm and seven batches of TiC particles with different particles sizes (see Table 1). The Al₂O₃ powder was mixed with one of the seven batches of TiC particles, respectively, in proper proportion by conventional ball milling. After being dried, the mixed powders were uniaxially pressed, cold isostatically pressed and then hot-pressed at 1700°C and 25 MPa for 30 min.

* Corresponding author. Fax: +86-10-62771160.

E-mail address: gong@tsinghua.edu.cn (J. Gong).

The polished surface of each hot-pressed sample was examined using electron scanning microscopy (SEM) and it was shown that, in each sample, the TiC particles were well dispersed in the Al_2O_3 matrix. This can also be seen from the SEM micrograph given later (Fig. 3).

The density of each hot-pressed sample was measured using the Archimedes principle. The results are summarized in Table 1. Note that, for each sample, the measured density is somewhat higher than its theoretical value, 4.268 g/cm^3 , which was calculated as the weighted average of the known theoretical densities of the two pure components, Al_2O_3 (3.987 g/cm^3) and TiC (4.911 g/cm^3). This can be explained by considering that, during hot-pressing of the composites, slow solid-state diffusional reactions may take place, forming solid reaction layers in the interfaces between Al_2O_3 grains and TiC particles.

The fracture toughness was measured with conventional single-edge-notched beam (SENB) method. Specimens with nominal dimensions of $4 \times 6 \times 30$ mm were cut directly from the hot-pressed products. The tensile surfaces of all the specimens were kept to be perpendicular to the hot-pressing direction. After being ground lengthwise using a 220 grit diamond wheel, all the specimens were notched to a depth of about 50% of their heights and then tested in three-point bending with a span of 24 mm and a crosshead speed of 0.05 mm/min.

It should be pointed out that, for a valid K_{IC} measurement on ceramics using notched specimen, it is essential to keep the notch-root radius ($\approx 1/2$ notch width) smaller than a critical value which is a material parameter.¹⁰ It has been suggested¹⁰ that, for fine-grained Al_2O_3 , a notch width of about 0.1 mm is necessary for valid K_{IC} measurements. In the present study, the notch widths were kept in the range of 0.12–0.15 mm. Undoubtedly, such a somewhat larger notch-width may result in an over-estimation in K_{IC} . However, it seems to be reasonable to make a direct comparison between the composites based on K_{IC} data measured with the specimens with notches of the nearly same widths.

3. Results and discussion

Variation in K_{IC} with the average size of TiC particles, d , is represented in Fig. 1. Each of the data points represents an average of measurements from at least five tests. As can be seen, the measured K_{IC} strongly depends on the size of the second-phase particles.

Fig. 2 shows typical micrograph of the fracture surface of sample AC02. As can be seen, the matrix Al_2O_3 grains and the TiC particles are nearly identical in their sizes and the morphology of crack propagation during strength test is largely intergranular within the whole fracture surface. Note that, as shown in Fig. 1, the fracture toughness is nearly independent of TiC particle

Table 1
Typical properties of the test samples

Sample denotation	Average size of TiC particles (μm)	Density (g/cm^3)
AC01	0.5	4.282
AC02	1.7	4.282
AC03	3.8	4.291
AC04	5.6	4.300
AC05	8.2	4.305
AC06	10.3	4.271
AC07	12.7	4.270

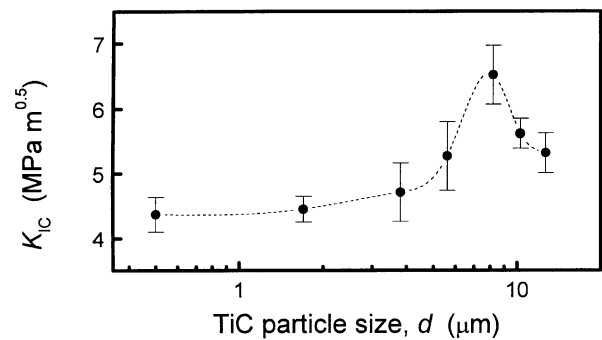


Fig. 1. Variation in fracture toughness of the composites with the average size of TiC particles.

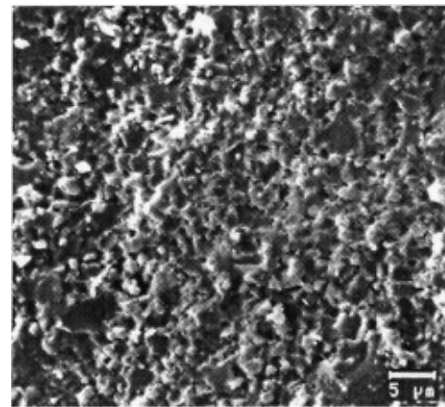


Fig. 2. Fracture surface of a strength specimen of sample AC02.

size up to $d \approx 2 \mu\text{m}$. Besides, the fracture toughness data measured with samples AC01 and AC02 are comparable with that reported for monolithic fine-grained Al_2O_3 ceramics, about 4 $\text{MPa m}^{0.5}$.¹¹ Combining these observations, one can conclude that adding smaller TiC particles in Al_2O_3 matrix has little effect on the toughness of the resultant composite.

A small but distinct increase in K_{IC} was observed when the average size of TiC particles increases from 2 μm and then a maximum in K_{IC} was obtained when d increases to 8 μm . When the TiC particle size increases further, however, the measured K_{IC} exhibits a decreasing tendency. Experimental results similar to those shown in

Fig. 1 have been frequently observed in studies concerning the influence of grain size on the toughness of monolithic ceramics. For example, when studying the toughness variation of polycrystalline Al_2O_3 , Rice et al.¹² found that variation in grain size can produce large changes in toughness and there is a particular grain size for which toughness is a maximum. However, similar phenomenon has rarely been reported for two-phase brittle ceramics. As mentioned above, previously published papers have thus far emphasized the effect of content of the second-phase particles on mechanical properties of two-phase brittle ceramics and little effort has been devoted to the observation and analysis of the effect of size of the second-phase particles.

There have been many studies^{1,2} concerning the toughness improvement in hard second-phase particle reinforced ceramics and two mechanisms are thought to be the particularly important in determining toughness: (1) crack deflection along weak grain boundaries and (2) crack trapping combined with crack face bridging by second-phase particles. However, our observations reveal that both mechanisms seem not to play significant roles in the Al_2O_3 -TiC composites examined in the present study.

Fig. 3 shows the surface crack pattern produced by Vickers indentation on sample AC05. The light phase in this micrograph is TiC particles. As can be seen, the fracture morphology within the Al_2O_3 matrix in sample AC05 is similar to that shown in Fig. 2, characterizing an intergranular crack propagation mode. When intersecting a large TiC particle, however, the crack propagated directly through the particle instead of bypassing it. This observation is in agreement with the result of Wahi and Ilschner⁵ who observed TiC particles with clear fine cleavage steps running through the grains in Al_2O_3 ceramics reinforced with different volume fractions of TiC particles. Examination of also revealed the same features. These observations indicate that crack deflection and crack bridging are not the dominating toughening mechanisms in Al_2O_3 -TiC composites.

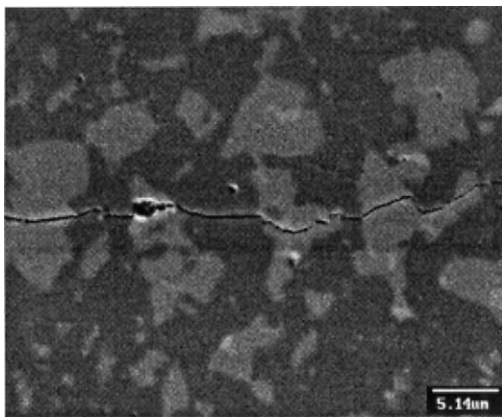


Fig. 3. Indentation-induced crack path observed in the surface of sample AC05.

A possible explanation for the improvement in toughness due to the addition of TiC particles may be proposed by noting that there is a slight difference between the linear coefficient of thermal expansion, α , of TiC ($\alpha_P = 2.4 \times 10^{-6}/^\circ\text{C}$)⁵ and that of Al_2O_3 ($\alpha_M = 8.0 \times 10^{-6}/^\circ\text{C}$)⁵. As it has been well known, the difference between the linear coefficients of thermal expansion would result in residual stresses developing within the composite upon cool-down from the hot-pressing temperature. Since $\alpha_P < \alpha_M$, the Al_2O_3 matrix would be placed in “hoop-tension” and, when intersecting the TiC particle, the crack would be “attached” to and pinned, rather than deflected, by the particle. Due to the intrinsic brittleness of TiC, a further propagation of the crack inside the TiC particle would directly result in the fracture of the particle. This may be the reason why no bridging was observed in the vicinity of the TiC particles. The improvement in toughness, on the other hand, may be attributed to the fact that the TiC particles embedded in the Al_2O_3 matrix are subjected to a radial compression field which creates extra resistance to the propagating crack front.

Since the magnitude of the residual stresses resulting from the mismatch between the thermal properties of the Al_2O_3 matrix and the TiC particles depends on the size of the TiC particle as well as the difference between α_P and α_M , the effect of such a residual stress on toughness may be too small to be detected when the particle size is smaller than a critical value. Thus, little improvement in toughness can be observed in composites having small TiC particles, e.g. samples AC01 and AC02.

To support the above discussion, the sample AC05 was examined using high-resolution transmission electron microscopy (HREM). A typical HREM image of an area in a TiC particle adjacent to the Al_2O_3 /TiC interface is shown in Fig. 4. The lattice constant of TiC near the Al_2O_3 /TiC interface was determined directly from the HREM image to be 0.4261 nm. On the other

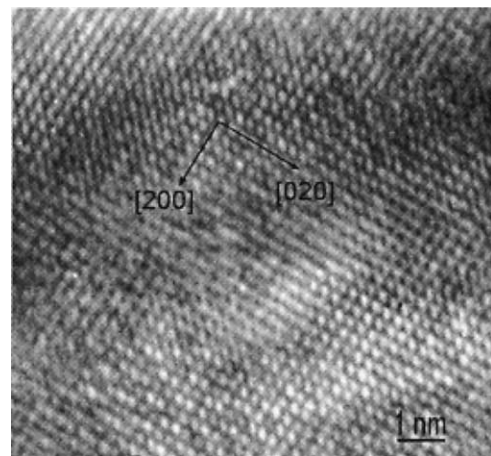


Fig. 4. HREM image of an area in a TiC particle adjacent to TiC/ Al_2O_3 interface in sample AC05.

hand, similar observations at the center of the same TiC particle yield a lattice constant of 0.4313 nm. Considering that the residual thermal stresses acting on the $\text{Al}_2\text{O}_3/\text{TiC}$ interface may relax to a certain degree during the thinning of the sample, one can expect that the difference between the lattice constant near the interface and that in the center of the particle may be larger than the result determined from the HERM observations. Thus, it can be concluded from the these observations that the TiC particles embedded in the Al_2O_3 matrix in the sample AC05 are indeed subjected to a radial compressive stress field resulting from the mismatch between the thermal expansion coefficients of Al_2O_3 and TiC, being consistent with the above analysis.

Now let us turn to account for the sharp downturn in toughness in composites having extremely larger TiC particles, $d > 8 \mu\text{m}$. Considering two special cases shown in Fig. 5. Fig. 5 (a) illustrates the crack propagation path in a monolithic Al_2O_3 . Based on the above-mentioned observations, we can reasonably assume that the crack propagation is intergranular in this case. Fig. 5 (b) shows the crack propagation path in a TiC particle reinforced Al_2O_3 composite. The crack front intersects with a spherical TiC particle and then propagates directly through it. Clearly, the difference between the toughnesses measured in both cases may be related directly to the difference between the energies dissipated for creating the so-called shielding zone developed behind the crack tip during crack propagation.

The energy dissipated in the vicinity shown in Fig. 5 (a), W_1 , can be expressed approximately as:

$$W_1 \approx 2b\Delta c(\gamma_s)_M \quad (1)$$

where $(\gamma_s)_M$ is the fracture surface energy of Al_2O_3 , Δc is the dimension of the shielding zone and b is the width of the test specimen.

On the other hand, the energy dissipated in the vicinity shown in Fig. 5 (b), W_2 , can be divided into three components, the first being related to the fracture surface energy dissipated within the Al_2O_3 matrix, the second related to the fracture surface energy dissipated

within the TiC particle and the third related to the effect of residual thermal stress on the crack propagation within the particle. Following the analysis of Zhang et al.,¹³ W_2 can be expressed as:

$$W_2 \approx 2(b\Delta c - \pi r^2)(\gamma_s)_M + 2(\pi r^2)(\gamma_s)_P + \frac{1}{2}\pi r^2 pu \quad (2)$$

where r is the radius of the TiC particle, $(\gamma_s)_P$ is the fracture energy of TiC particle, u is the crack open displacement within the particle (see Fig. 6) and p is the residual stress resulting from the mismatch between thermal properties of the particle and the matrix¹⁴,

$$p = \frac{2(\alpha_P - \alpha_M)\Delta T E_M E_P}{E_P(1 + \nu_M) + 2E_M(1 - 2\nu_P)} \quad (3)$$

where E and ν are Young's modulus and Poisson's ratio, respectively, ΔT is the temperature difference, and the subscripts P and M denote the particle and the matrix, respectively.

In general, fracture would occur once the elastic deformation of the brittle particle reaches its critical value, about 0.1%.¹⁴ Thus, one can reasonably assume that $u \approx 2r \times 0.1\% \propto r$ at the critical state. Note that, as predicted in Eq. (3), p is independent of r . Thus, we obtained

$$\Delta W = W_2 - W_1 = 2\pi r^2[(\gamma_s)_P - (\gamma_s)_M] + \beta r^3 \quad (4)$$

where β is positive and can be treated approximately as a constant for a given system.

It should be pointed out that both $(\gamma_s)_P$ and $(\gamma_s)_M$ in Eq. (4) were taken as their apparent values, respectively, and depend strongly on crack propagation paths. Note that intergranular crack propagation was observed in the Al_2O_3 matrix while transgranular crack propagation was observed within the TiC particle. As a result, $(\gamma_s)_P$ would be very near to the free surface energy of TiC and $(\gamma_s)_M$ would be somewhat larger than the free surface energy of Al_2O_3 . Thus there is reason to believe that $(\gamma_s)_P < (\gamma_s)_M$ in the Al_2O_3 -TiC composites examined

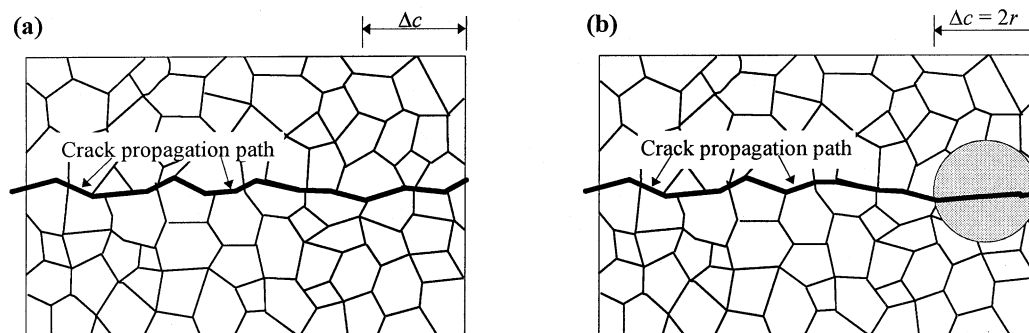


Fig. 5. Illustrations of the crack propagation paths in (a) monolithic Al_2O_3 and (b) Al_2O_3 -TiC composite.

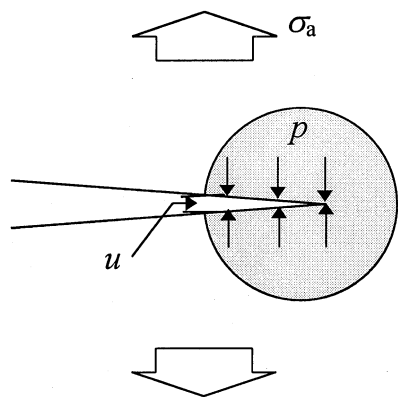


Fig. 6. Illustration of the formation of a shielding zone behind the crack tip within TiC particle.

here. If this is true, the first and the second terms in the right-hand side of Eq. (4) would decrease and increase with r , respectively. Consequently, a well-defined maximum in ΔW would be observed in some certain situations.

4. Summary and conclusions

The fracture toughness of Al_2O_3 -30 wt.%TiC composite was measured as a function of the average size of TiC particles. It was found that the average size of TiC particles plays an important role in determining the mechanical properties of the Al_2O_3 -TiC composite. Adding small TiC particles in Al_2O_3 has little effect on the toughness of the resultant composite while significant improvement in toughness may be obtained when the TiC particle size is kept within an appropriate range. Especially, there is a particular TiC particle size for which toughness reaches a maximum.

Based on the observations on the fracture surfaces and the crack propagation paths, the improvement in

toughness was attributed to the effect of residual stresses resulting from the mismatch between the thermal properties of TiC particles and Al_2O_3 matrix.

References

1. Evans, A. G., Perspective on the development of high-toughness ceramics. *J. Am. Ceram. Soc.*, 1990, **73**, 187–206.
2. Becher, P. F., Microstructural design of toughened ceramics. *J. Am. Ceram. Soc.*, 1991, **74**, 255–269.
3. Furukawa, M., Studies on ceramic cutting tool materials and development of manufacturing technology. *J. Jpn. Soc. Powder & Powder Metall.*, 1991, **38**, 824–830.
4. Lo Casto, S., Lo Valvo, E., Ruisi, V. F., Lucchini, E. and Maschio, S., Wear mechanism of ceramic tools. *Wear*, 1993, **160**, 227–235.
5. Wahi, R. P. and Ilschner, B., Fracture behavior of composites based on Al_2O_3 -TiC. *J. Mater. Sci.*, 1980, **15**, 875–885.
6. Furukawa, M., Nakano, O. and Takashima, Y., Fracture toughness of Al_2O_3 -TiC ceramics. *Inter. J. Refrac. Hard Metals*, 1988, **7**, 37–40.
7. Katsumura, Y., Takahashi, T. and Suzuki, H., Flaking phenomenon of Al_2O_3 -TiC ceramics tools. *J. Jpn. Soc. Powder & Powder Metall.*, 1991, **38**, 591–596.
8. Krell, A. and Klaffke, D., Effects of grain size and humidity on fretting wear in fine-grained alumina, Al_2O_3 /TiC and zirconia. *J. Am. Ceram. Soc.*, 1996, **79**, 1139–1146.
9. Sakaguchi, S., R-curve behavior of Al_2O_3 -TiC ceramic composites. *Silica. Indus.*, 1997, **62**, 15–19.
10. Simpson, L. A., Use of the notched-beam test for evaluation of fracture energies of ceramics. *J. Am. Ceram. Soc.*, 1974, **57**, 151–156.
11. Steinbrech, R. W., R-curve behavior of ceramics. In *Fracture Mechanics of Ceramics*, Vol. 9, ed. R. C. Bradt, D. P. M. Hasselman, D. Munz, M. Sakai and V. Ya. Shevchenko. Plenum, New York, 1992, pp. 187–208.
12. Rice, R. W., Freiman, S. W. and Becher, P. F., Grain-size dependence of fracture energy in ceramics: I, experiments. *J. Am. Ceram. Soc.*, 1981, **64**, 345–350.
13. Zhang, G. J., Yue, X. M. and Jin, Z. Z., Microprocesses of crack propagation in particulate toughened ceramics. *J. Chinese Ceram. Soc.*, 1995, **23**, 365–372.
14. Lawn, B. R., *Fracture of Brittle Solids*. Cambridge University Press, New York, 1993.