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Preparation, microstructures and properties of Al_2O_3–TiC composites

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Abstract

Al_2O_3–TiC composites with various amounts of TiC, from 16 to 30 vol.%, were pressureless-sintered using 1 wt.% Al as an additive. The electrical conductivity, Vickers hardness, fracture toughness and wear resistance of the composites were studied. The hardness and toughness increased gradually with the increase of TiC volume fraction. The electrical conductivity increased with the increase of TiC volume fraction and increased about 10 orders of magnitude as the TiC volume fraction changed from 0.20 to 0.23. A sample whose TiC volume fraction is 0.23, which is close to its percolation threshold, had the best wear resistance. © 2002 Elsevier Science Ltd and Techna S.r.l. All rights reserved.

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1. Introduction

Al_2O_3–TiC composites have been widely used as cutting tools recently, because of their high strength, hardness, chemical stability and excellent wear resistance. However, chemical reactions between Al_2O_3 and TiC [1] (e.g. $Al_2O_3 + TiC = Al_2O\downarrow + TiO + CO\downarrow$) can occur at high temperatures, introducing pores into the composites which deteriorate the mechanical properties. Thus, due to these gas-generating reactions, fully dense Al_2O_3–TiC composites are difficult to obtain by conventional sintering. In order to prevent these reactions and to obtain a high density material, hot pressing (HP) [2], hot isostatic pressing (HIP) [3] and pressureless sintering (PS) at 1950 °C with a fast heating rate (400 °C/min) [1] have been used.

Comparing with HP and HIP, PS gives somewhat worse mechanical property materials, however, its production costs are also lower. Therefore, preparation of Al_2O_3–TiC composites by PS has still attracted attention [1,4–6]. Chae et al. [7] studied the effect of Y_2O_3 addition on the densification of Al_2O_3–TiC. Their results show that Y_2O_3 can effectively inhibit the gas-generating reactions. In this work, PS and Al as an additive were selected to prepare Al_2O_3–TiC composites. The purpose of the Al additive was to lower the sintering temperature and, possibly, to suppress the gas-generating reaction. The mechanical properties (hardness, toughness and wear resistance) as functions of the volume fraction of TiC in the composites were studied, which has not been previously done in a systematic way.

2. Experimental procedures

Al_2O_3 (AKP-50, 0.3 μm), TiC (average grain size 1.5 μm) and Al (~5 μm) were used as starting powders. The Al_2O_3 powder, various amounts of TiC powders and 1 wt.% Al powder were homogenized by planetary ball milling for 5 h in hexane. The milled powders were then dried under vacuum and uniaxially pressed into pellets (20 mm in diameter and 5 mm thick). The pellets were embedded in the powders of the same composition and sintered at 1750–1800 °C for 30 min under argon in an induction furnace. The heating rate was about 30 °C/min.

After grinding and polishing, the density of the samples was measured using the method given in Ref. [8].

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The theoretical density of the samples was determined by the rule of mixtures, using 3.986 and 4.92 g/cm^3 for the theoretical densities of Al_2O_3 and TiC, respectively. After painting silver paste electrical contacts on both surfaces, the d.c. electrical conductivity was measured using a Keithley 617 programmable electrometer and an LR400 self-balancing bridge, respectively for high and low resistance samples. Vickers hardness was evaluated at a load of 10 kg. Fracture toughness was determined from radial cracks produced by Vickers indentation at a load of 1 kg [9]. Each data point was obtained from the average of 3–5 indentations. The wear resistance was tested using a commercial dimple grinder [10,11]. The samples were ground under a load of 20 g for 1 min. The wear volume was calculated from the diameter of the crater, which was measured using optical microscopy. Each data point was obtained from the average of 3–5 dimples. The microstructure of polished, fractured and worn surfaces of the samples was observed by scanning electron microscopy (SEM).

3. Results and discussion

The relative densities of 16, 20, 23, 27 and 30 vol.% TiC samples are 98.0, 97.5, 98.2, 97.7 and 98.4%, respectively, which indicates that using the embedding powder and the Al additive can efficiently reduce porosity creating chemical reactions between Al_2O_3 and TiC at high temperatures.

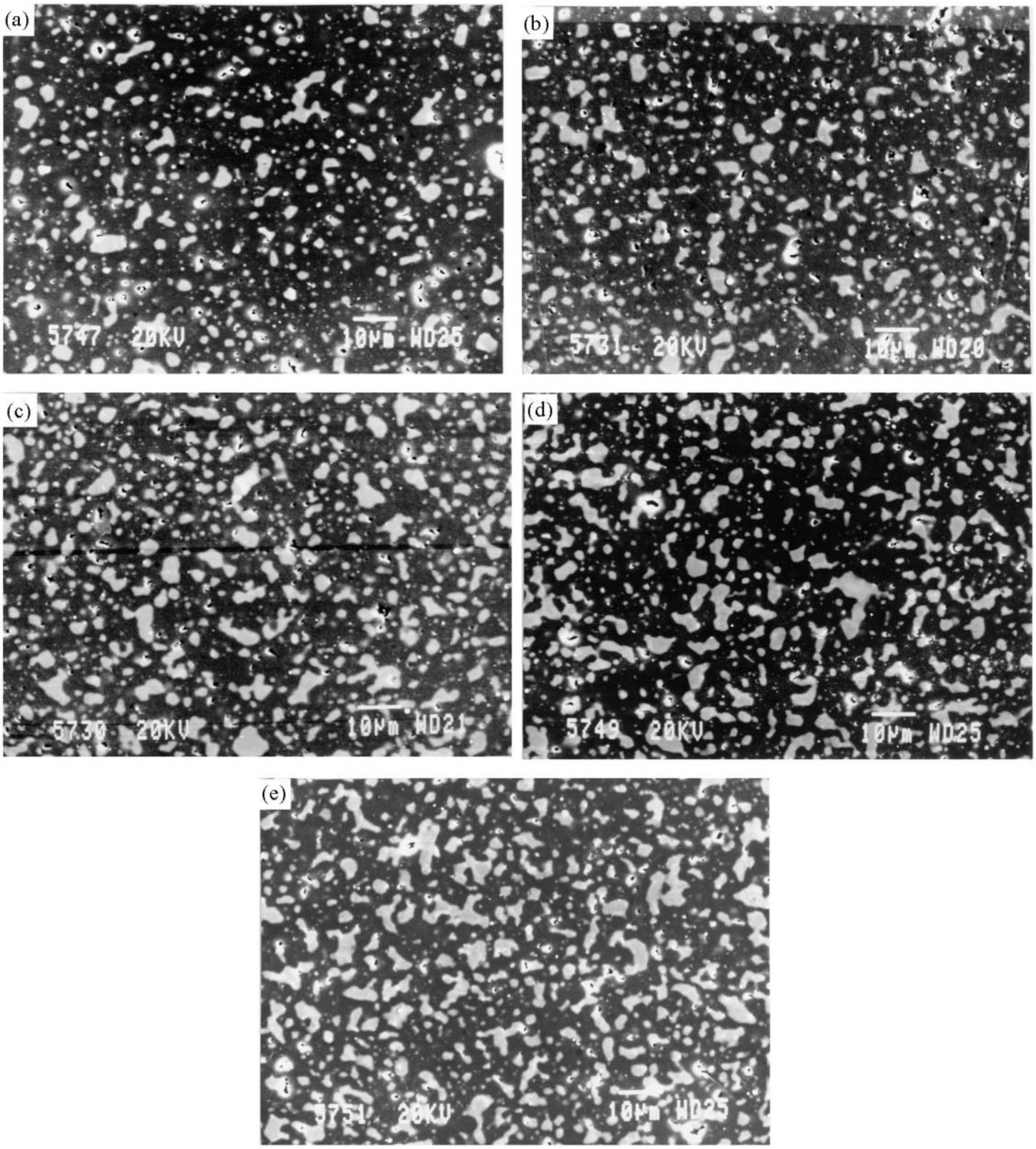

Fig. 1. Typical SEM micrographs of the polished samples: (a) 16 vol.%; (b) 20 vol.%; (c) 23 vol.%; (d) 27 vol.%; (e) 30 vol.% TiC.

Fig. 1a–e shows the typical microstructure of the polished surface of the samples. It can be seen from Fig. 1 that the samples are highly densified. The grey–white phase in Fig. 1 is TiC and the dark phase is Al_2O_3. The pores are mainly closed and correspond to the black spots inside the TiC grains. It can be observed that the higher the TiC content, the larger the TiC grains or clusters. This is mainly because as the TiC content increases more TiC particles are in contact, forming larger clusters. Previously, no Al_2O_3–TiC samples were pressureless-sintered to closed porosity below 1840 °C [12]. In this work, the sintering temperature is lower than 1840 °C, due mainly to the Al additive.

As the Al content was only 1 wt.% Al and no Al grains wherever observed after sintering, its effect on the properties of the samples is not discussed below.

3.1. Electrical conductivity

As the relative density of all the samples is around 98%, the effect of porosity on the electrical conductivity of the samples can be neglected. Because the TiC is a metallic conductor and Al_2O_3 is an insulator, the electrical conductivity of the sample increases with TiC content and increases rapidly as the TiC volume fraction goes from 0.20 to 0.23 as shown in Fig. 2. This implies that the TiC phase becomes continuous in this range and that the percolation threshold (φ_p) for this series of Al_2O_3–TiC composites is in the range of 0.20 to 0.23. Using the equation given by Wu and McLachlan [13] to fit the experimental data, gives a percolation threshold of 0.22 and the percolation exponents $s = 0.9280$ and $t = 2.857$, which are close to those observed in [13]. Generally, for two components composite material with equal sized grains, φ_p is about 0.16 [14,15]. In this work it is a little higher, probably because the grain size of the Al_2O_3 is somewhat smaller than that of the TiC at the percolation threshold (see Fig. 7).

3.2. Mechanical properties

3.2.1. Vickers hardness and fracture toughness

The Vickers hardness and fracture toughness of the samples are plotted in Fig. 3 vs. the TiC volume fraction. Both properties increase with increasing TiC volume fraction up to 30 vol.% and can, almost certainly, be extrapolated to still higher values. Wahi and Ilshner [2] also found that the fracture toughness of HPed Al_2O_3–TiC composites increased with TiC content up to 35.1 vol.%. Both the hardness and toughness are somewhat lower for the present materials than for HPed or HIPed composites with 98.7–99.5% porosity [2] and fully dense [3], probably on account of porosity. The hardness of the composites increases gradually as the TiC content increases, because TiC is relatively harder than Al_2O_3.

The fracture toughness of the samples also increases with the TiC content. This is almost certainly due to effects of crack deflection and grain bridging by TiC grains as shown in Fig. 4. Crack deflection is due to the intrinsic stresses produced by the thermal mismatch of the two phases during cooling. As the TiC content increases, the TiC grains or clusters become coarser as can be seen in Fig. 1. The coarser the TiC grains, the more the cracks deflect and bridge. Therefore, as the TiC content increases, the fracture toughness increases.

3.2.2. Wear resistance

The wear volume of the samples as a function of the volume fraction of TiC is plotted in Fig. 5. It is seen

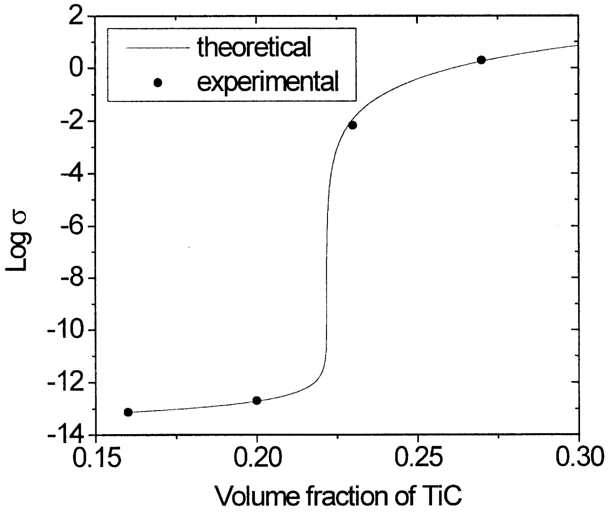

Fig. 2. Electrical conductivity of the composites as a function of the volume fraction of TiC. The theoretical line through the points is discussed in the text.

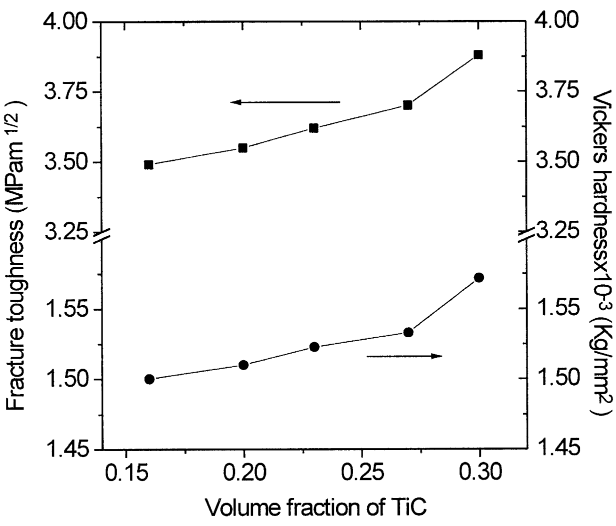

Fig. 3. Fracture toughness and Vickers hardness of the composites as functions of the volume fraction of TiC.

from Fig. 5 that the wear volume of the samples does not have a linear relation with the volume fraction of TiC. The lowest value of the wear volume corresponds to a TiC volume fraction of 0.23, i.e. the higher limit of the estimated φ_p.

Fig. 6a and b are typical micrographs taken in a dimple of the 16 vol.% sample after abrasion test, for a low wear loss area and a high wear loss area, respectively. Comparing Fig. 6a with b, it can be seen that there are fewer pits in Fig. 6a than in Fig. 6b and that the content of the white phase (TiC) is similar in the two different wear loss positions. This implies that the TiC is more difficult to wear off than Al_2O_3. In other words, adding TiC particle can increase the wear resistance of the composites. However, as the TiC volume fraction increases, the TiC forms ever larger clusters, isolated below φ_p and continuous above φ_p (Fig. 1 and Fig. 7). Note that we only considered the effect of grain size of the TiC because we do not see any major difference in the grain size of the Al_2O_3. It is also known that the wear resistance decreases with increasing of the grain size [16]. Therefore, the two competing processes lead to the smallest value of wear volume when the volume fraction of TiC is around φ_p.

The above results emphasize the fact that the distribution of the grains and the grain size of the TiC phase are very important for both the electrical conductivity and wear resistance of the composites. The value of φ_p depends strongly on the relative grain size and shape of the Al_2O_3 and TiC components, therefore using different starting powders and processing will lead to different values of φ_p. This means that the composition of the composite with the optimum wear resistance will change with the starting materials and processing, as will the composition with the best overall mechanical properties. Recall, from 3.2.1 that the hardness and toughness increase monotonically with TiC content. It is reported [17] that the composite in which the TiC content is about 30 wt.% (25.8 vol.%) has the best mechanical properties. However, our work shows that the best wear resistance is about 23 vol.%, while both the hardness and toughness increase with TiC content in the range of 16 to 30 vol.%.

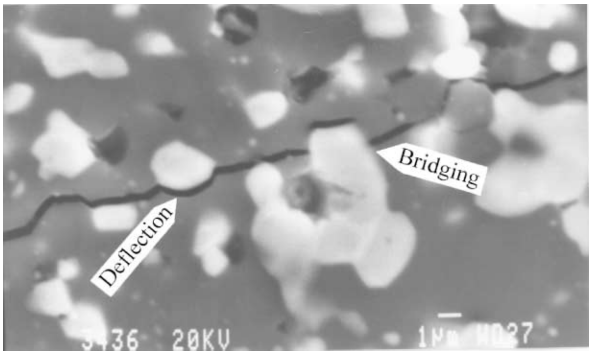

Fig. 4. Typical SEM micrograph of crack path of 16 vol.% TiC sample induced by a Vickers indention.

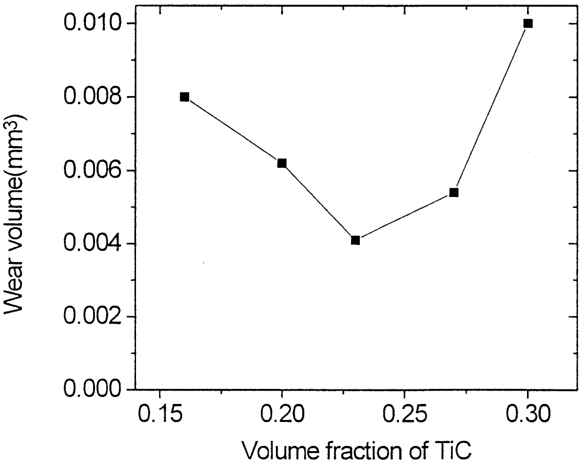

Fig. 5. Wear volume of the composites as a function of the volume fraction of TiC.

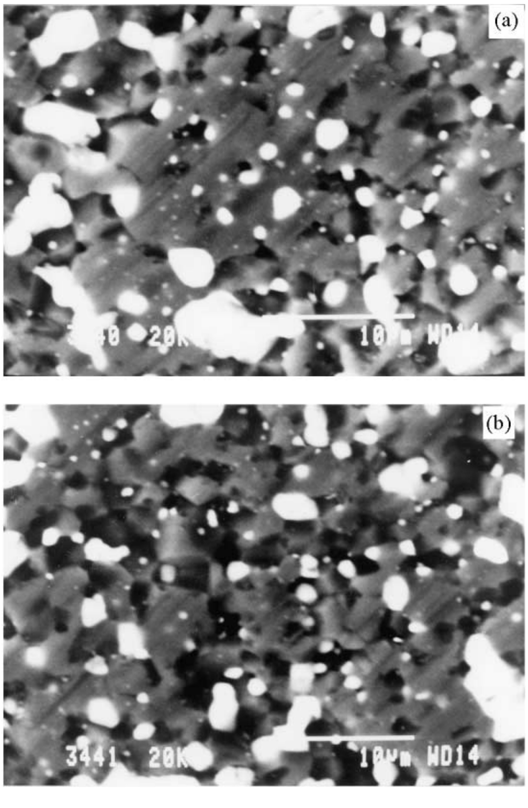

Fig. 6. Micrographs of the 16 vol.% sample after abrasion test: (a) low wear loss; (b) high wear loss.

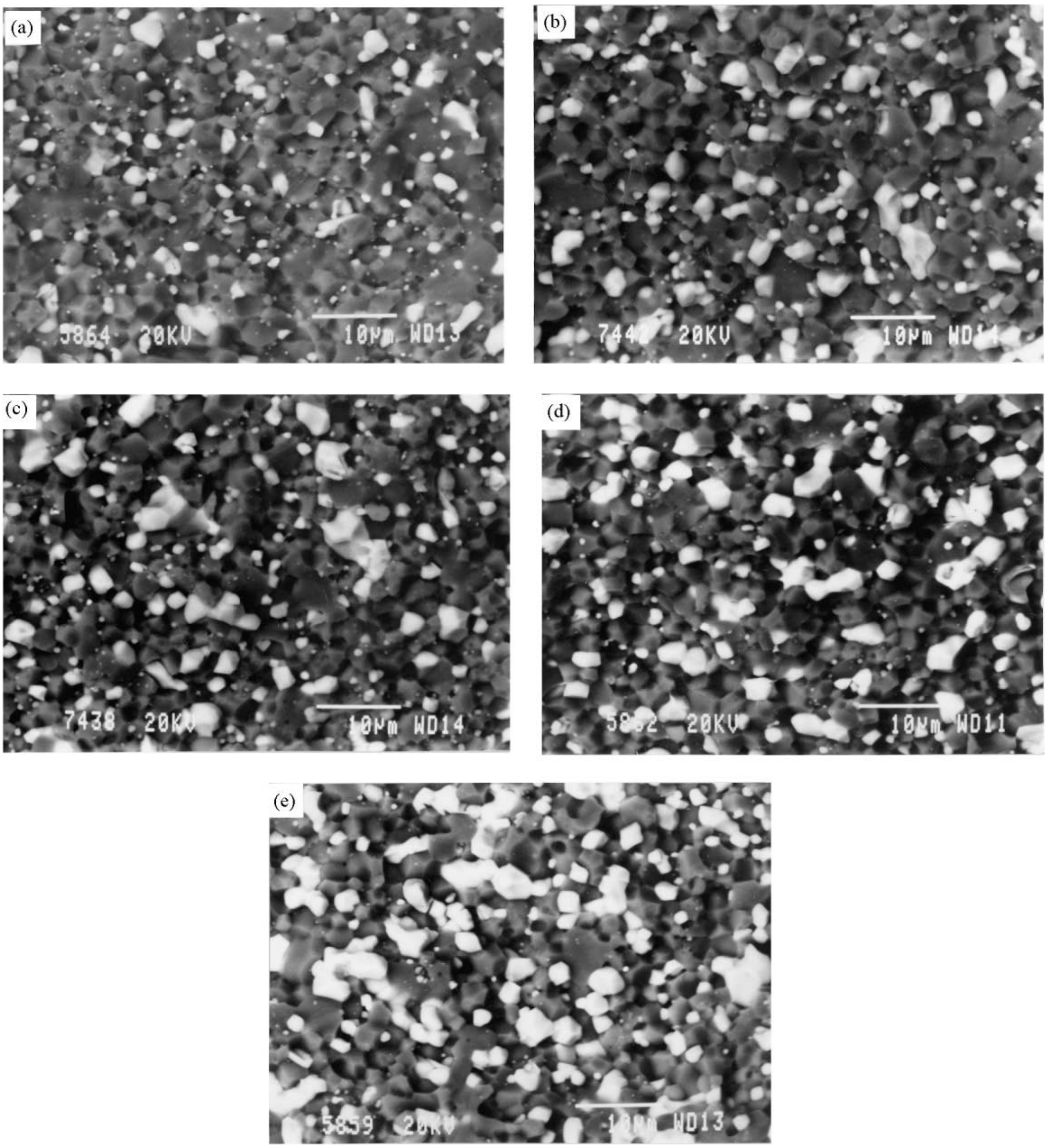

Fig. 7. Back-scattered electron micrographs of fracture surface of the samples: (a) 16 vol.%; (b) 20 vol.%; (c) 23 vol.%; (d) 27 vol.%; (e) 30 vol.%.

4. Conclusions

Using embedding powder and 1 wt Al% as an additive, a high density Al_2O_3–TiC composite can be prepared using pressureless sintering. The electrical conductivity, hardness, toughness and wear resistance of the composites are studied. Both the hardness and toughness increased with TiC content up to the maximum of 30 vol.% studied in this paper. Crack deflection and bridging mechanisms are revealed as the mechanisms responsible for the toughening behavior. When the TiC content is close to the percolation threshold, the electrical conductivity changes rapidly and the wear resistance reaches its highest value.

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