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Properties of Electrical Discharge Machinable SiC-TiB_2 Composites

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Electrical discharge machinable SiC-TiB_2 composites were fabricated by hot-pressing Their mechanical and electrical properties were determined as a function of TiB_2 content. The addition of TiB_2 to SiC matrix increased the strength and toughness and decreased electrical resistivity The flexural strength and fracture toughness of SiC-40 vol% TiB_2 composites were approximately 50% higher than those of monolithic SiC ceramics. Microstructural analysis showed that the toughening was mainly due to the crack deflection, with some possible contribution from crack branching or microcracking.

Key words : SiC, TiB_2, Composites, Electrical discharge machinable ceramics

I. Introduction

Silicon carbide ceramics are one of the most attractive engineering materials under investigation because of their excellent wear and creep resistance. However, low fracture toughness (2.5-4 $MPa \cdot m^{1/2}$) and expensive cost for machining limit their wide applications under stresses or impact.[1]

Recently, toughening of SiC ceramics has been achieved by using liquid phase sintering and developing duplex microstructure.[2-4] Several researchers have reported fracture toughness of above 8 $MPa\ m^{1/2}$ in liquid phase sintered SiC.[5,6] However, high cost of machining was still remained as a problem in the liquid phase sintered SiC.

Reinforcement of particles with higher hardness, higher elastic modulus, and higher thermal expansion coefficient than SiC is one of the promising methods to increase the toughness. The higher thermal expansion of the reinforcing particles causes radial tensile stress and tangential compressive stress in the SiC matrix.[7] The stresses make the crack deflection around the reinforcing particles easy, resulting in the increased fracture toughness. Also, addition of material with lower electrical resistivity than SiC makes electrical discharge machining (EDM) of the material possible. EDM is especially effective for machining of complicate shapes, which is impossible or costly by other conventional method. Dispersing particles such as TiB_2[8,9] and TiC[10,11] satisfy the above requirements. However, TiB_2 is more promising reinforcing material for electrical discharge machinable SiC ceramics because of the lower electrical resistivity of TiB_2 ($\approx 10^{-5}\ \Omega$ cm) than TiC ($\approx 0.5\ \Omega \cdot$cm).[12,13] Furthermore, TiB_2 has higher hardness and elastic modulus than TiC.

In this study, electrical discharge machinable SiC-TiB_2 composites were prepared by hot-pressing. Their mechanical and electrical properties were determined as a function of TiB_2 content. The feasibility of EDM for SiC-TiB_2 composites was also investigated.

II. Experimental Procedure

Silicon carbide powder (Betarundum, Ultrafine, Ibiden Co., Ltd, Nagoya, Japan) was mixed in alcohol with 4 wt% phenolic resin, which was equivalent to 1.6 wt% addition of carbon. The slurry was dried and pyrolysed at 650°C for 1 h under argon. The temperature was raised at a rate of 2°C/min. The pyrolysed powders were mixed with 0-60 vol% TiB_2 (grade F, H. C. Starck, Laufenburg, Germany) and 1 wt% aluminum (< 44 μm, Wako Pure Chemical Industries, Ltd., Tokyo, Japan) using polyethylene jar and WC balls. The mixed slurry was then dried, sieved through a 60 mesh screen, and hot-pressed at 1950°C for 40 min with 25 MPa of applied pressure under argon atmosphere.

Densities were measured using the Archimedes method. Theoretical densities were calculated based on the rule of mixtures. The microstructures of hot-pressed samples were observed by optical microscopy and scanning electron microscopy (SEM). The hot-pressed materials were cut into 3×4×25 mm bars and their surfaces and edges were polished with an 800 grit diamond wheel for flexural testing. Four point flexural strength (σ_f) was measured at room temperature with outer span of 20 mm and inner span of 8 mm. The fracture toughness (K_{IC}) was measured using a Vickers indentor with a load of 49 N.

Electrical resistivity was measured using a 4-point

probe method. EDM was done using wire electrode (Cu wire with the diameter of 0.3 mm) and cutting rates were ranged from 300 to 600 mm/h.

III. Results and Discussion

Hot-pressing of SiC and SiC-TiB_2 compositions (doped with 1 wt% Al and 1.6 wt% C) containing up to 60 vol% TiB_2 to densities > 99% of theoretical was achieved without difficulty. Table I lists the hot-pressing results. Aluminum additive in the sintering of SiC was known to form grain boundary phases in the form of oxides and also to segregate to grain boundaries to aid densification of SiC.[14] Polycrystalline TiB_2 (doped with 2 wt% carbon) can easily be fabricated to over 99% of theoretical density by hot-pressing at 1700°C and 35 MPa of applied pressure.[15] Such hot-pressing behavior of TiB_2 and the beneficial function of aluminum and carbon in the densification of SiC resulted in the successful densification of the SiC-TiB_2 composites in the present work.

Table 1. Results of Hot-pressing and Electrical Resistivity of Monolithic SiC and SiC-TiB_2 Composites Doped with 1 wt% Al and 1.6 wt% C

Composition	Density		Electrical resistivity
(vol%)	g/cm^3	% of theoretical	(20°C, Ω · cm)
SiC	3.181	99.1	5.76×10^{3}
SiC+20% TiB_2	3.469	99.9	3.52×10^{1}
SiC+40% TiB_2	3.734	100	4.43×10^{-3}
SiC+60% TiB_2	3.996	100	2.51×10^{-3}

Analysis by X-ray diffraction of the hot-pressed samples revealed β-SiC and TiB_2 as major phases and α-SiC as a minor phase. Fig. 1 shows optical micrographs of polished surfaces of samples with TiB_2 content of 0-60 vol%. The bright phase is TiB_2, and the gray matrix is SiC. As shown, the SiC-TiB_2 composites are a two phase particulate composites consisted of randomly distributed TiB_2 particles ranging from 1 to 10 μm diameter in the SiC matrix. Most of TiB_2 particles are single grains, but some are clusters of several grains. Fig. 2 shows the fracture surfaces of samples. As shown, grain size of the SiC-TiB_2 composites increases with the content of TiB_2. It may be due to the increased tendency of TiB_2 clustering with the content of TiB_2, as evidenced by Fig. 1. The fracture mode was mostly intergranular.

The changes in frexural strength and fracture toughness with the volume fraction of TiB_2 are shown in Fig. 3, where both flexural strength and fracture toughness are seen to increase with the content of TiB_2 up to 40 vol%. It means that TiB_2 addition has a beneficial effect on the mechanical properties of SiC. For the flexural strength, SiC-TiB_2 composites have approximately 20-40% higher values than monolithic SiC. The relative increases in flexural strength and fracture toughness with TiB_2 content are comparable, as one expects from the strength-fracture

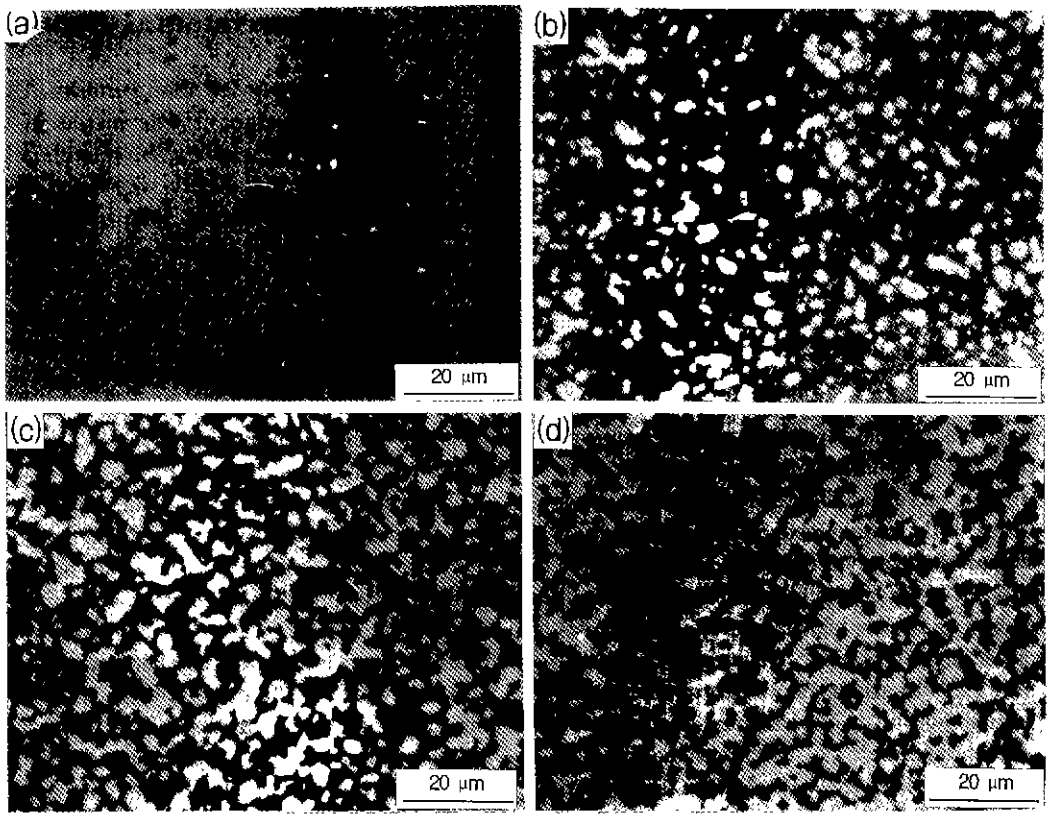

Fig. 1. Optical micrographs of polished cross section of SiC and SiC-TiB_2 composites: (a) monolithic SiC, (b) SiC-20 vol% TiB_2, (c) SiC-40 vol% TiB_2, and (d) SiC-60 vol% TiB_2.

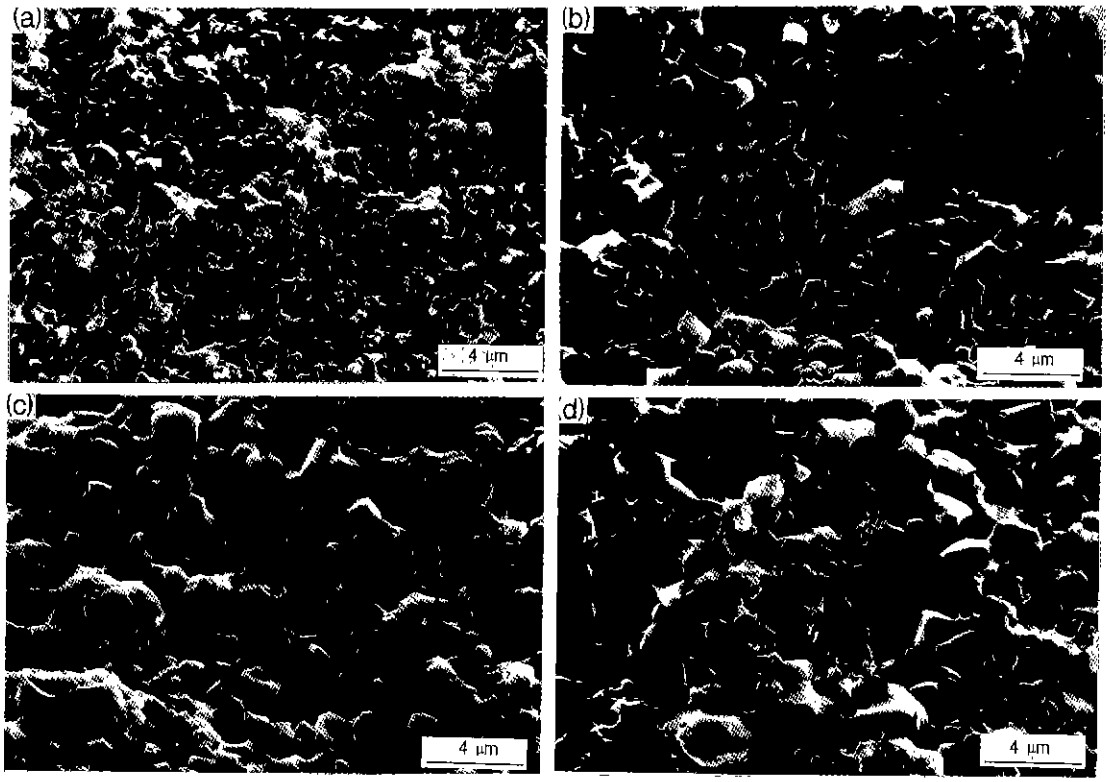

Fig. 2. Scanning electron micrographs of fracture surfaces of SiC and SiC-TiB_2 composites: (a) monolithic SiC, (b) SiC-20 vol% TiB_2, (c) SiC-40 vol% TiB_2, and (d) SiC-60 vol% TiB_2.

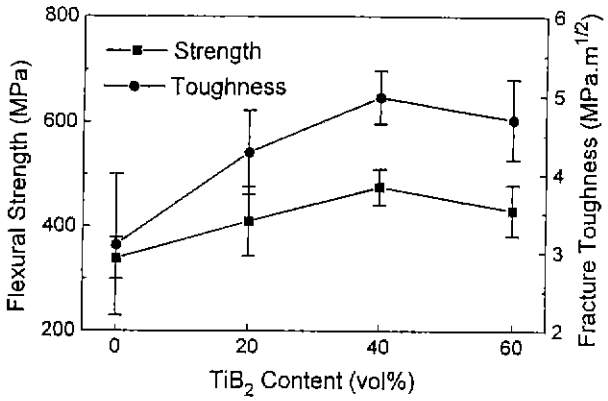

Fig. 3. Flexural strength and fracture toughness of SiC and SiC-TiB_2 composites as a function of TiB_2 content.

toughness relationship for Griffith material:

$$\sigma_f = Y K_{IC} c^{-1/2} \quad (1)$$

where Y is a dimensionless constant which depends on the geometry of the loading and crack configration and c is the critical flaw size. The observed relative increase in flexural strength with the content of TiB_2 is comparable to the relative increase in fracture toughness, indicating that the toughness is the dominant factor in the improved strength. This behavior is analogous to that of the SiC-TiC composites.[10,16] Lower strength of SiC-60 vol% TiB_2 composites than SiC-40 vol% TiB_2 composites is considered due to the excessive clustering of TiB_2 particles in SiC-60 vol% TiB_2 composites (Fig. 1). Clustering of TiB_2 particles may increase the effective critical flaw size and result in lower strength of the composites because the large TiB_2 grains can act as a critical flaw.[17]

The fracture toughness of SiC-TiB_2 composites increased with the content of TiB_2 and the maximum was obtained at 40 vol%. The fracture toughness of SiC-40 vol% TiB_2 composites is about 60% higher than that of monolithic SiC. Fig. 4 shows SEM micrographs of cracks induced by a Vickers indenter. The crack front is deflected at TiB_2 particles. It means that the composites have a weak bond between SiC and TiB_2. The TiB_2 particles have a higher thermal expansion coefficient (8.65×10^{-6} C^{-1}) than the SiC matrix (4.16×10^{-6} C^{-1}). The thermal expansion mismatch would result in TiB_2 shrinkage away from the SiC matrix during cooling from the hot-pressing temperature generating a weak SiC-TiB_2 interface. The mismatches between SiC matrix and TiB_2 particle in the linear thermal expansion coefficient (α) and elastic modulus (E) also result in the generation of the residual stresses in the particles and surrounding matrix during cooling after hot-pressing. The developed residual stress around the particle, radial matrix stress (σ_{mr}), can be calculated from the following equation:[16]

$$\sigma_{mr} = \frac{(\alpha_p - \alpha_m)\Delta T}{[(1+\nu_m)/2E_m] + [(1-2\nu_p)/E_p]} \quad (2)$$

where ν is the Poisson's ratio, ΔT is the temperature difference over which stresses are not relieved by a diffusive

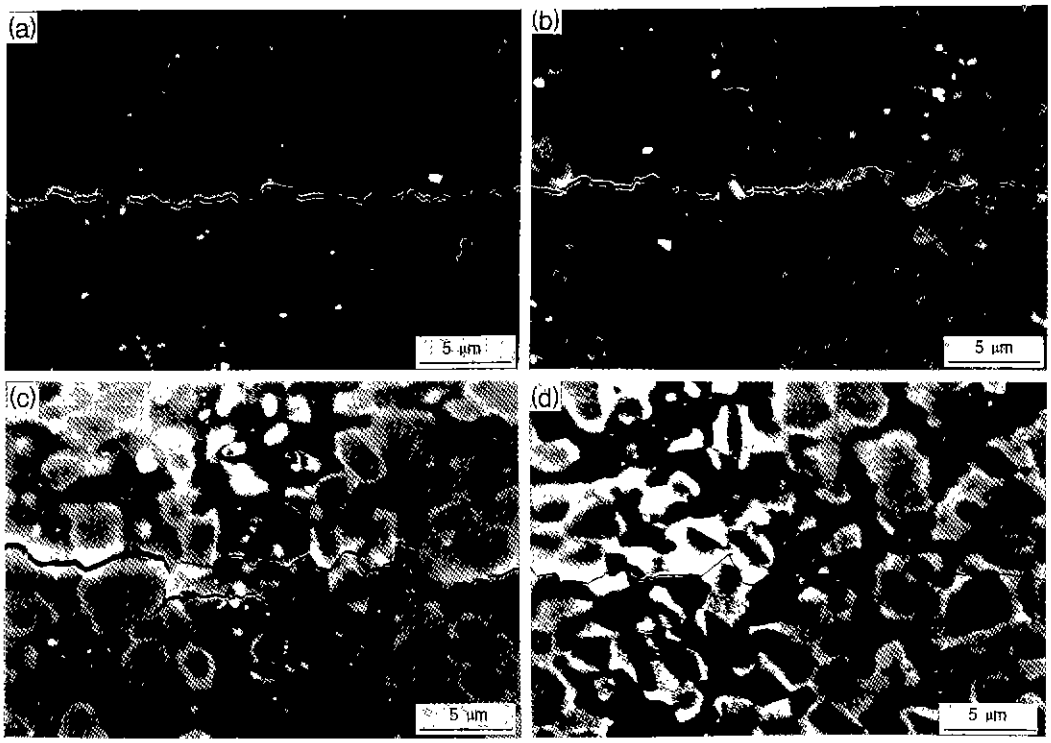

Fig. 4. Scanning electron micrographs of a crack path induced by a Vickers indentor in SiC and SiC-TiB_2 composites: (a) monolithic SiC, (b) SiC-20 vol% TiB_2, (c) SiC-40 vol% TiB_2, and (d) SiC-60 vol% TiB_2.

Fig. 5. Electrical discharge machined SiC-40 vol% TiB_2 composites.

process,[18] and the subscripts p and m refer to the particle and matrix, respectively.

Taking 8.65×10^{-6} C^{-1} and 4.16×10^{-6} C^{-1} for α_p and α_m,[7] 531 GPa and 410 GPa for E_p and E_m,[12] 0.25 and 0.17 for ν_p and ν_m,[19] and 1000°C for ΔT, the developed radial matrix tensile stress is $\approx$1900 MPa. The high tensile stress could be relieved by generating crack branching or microcracking in the vicinity of TiB_2. The tangential matrix compressive stress ($\sigma_{mt}=-\sigma_{mr}/2$) is $\approx$950 MPa. These stress levels will decrease with decreasing the particle radius and increasing the distance from the center of the particles.

A crack will be expected to propagate in a direction parallel to the axis of the local compressive stress and perpendicular to the axis of the local tensile stress in the matrix surrounding the particle. Generally, a crack approaching a particle above or below the crack plane would be deflected toward the radial matrix tensile stress. Observation on the polished surfaces revealed that the cracks were deflected by the TiB_2 particles (Fig. 4). In contrast, monolithic SiC had a fairly planar crack path. Deviations in the crack paths were a result of intergranular fracture in this sample. The amount of crack deflection out of plane (degree of deflection) appeared to increase with increasing TiB_2 content up to 40 vol%. When a crack front interacts with second-phase particles, the crack is deflected. This alters the crack-tip stress intensity and results in improved toughness.[20] Hence, the higher toughness of SiC-TiB_2 composites compared with monolithic SiC is mainly due to the increased crack deflection around the TiB_2 particles. Crack branching or microcracking caused by high radial tensile stress around TiB_2 particles may also have some contribution to the increased fracture toughness (Fig. 4(c)). The fracture toughness of SiC-60 vol% TiB_2 composites is slightly lower than that of SiC-40 vol% TiB_2 composites. It may be due to the transgranular fracture of large TiB_2 grains (Fig. 4(d)) and the partial loss of composite effect in SiC-60 vol% TiB_2 composites, where the major phase is TiB_2. Transgranular fracture of large TiB_2 grains reduces the contribution of deflection in toughening and results in

lower fracture toughness. Also, SiC in the TiB_2 matrix cannot act as a reinforcing particles because of lower elastic modulus and thermal expansion coefficient than TiB_2.

Figure 4 also reveals the existence of a core/rim structure in the TiB_2 grains. Microanalysis using electron probe (EPMA) on the rim area revealed 91.6 at% Ti and 8.4 at% W. In contrast, W-free Ti core was detected. B and C could not be analyzed because of the limited sensitivity of the equipment. W in the rim area is believed to be contaminated during ball milling by WC balls. The above results suggest that the observed core/rim structure might have been developed by the dissolution of nonequilibrium phases in the liquid and a subsequent reprecipitation of equilibrium phases onto undissolved nonequilibrium particles (TiB_2 grains), which constitute cores. It indicates that the grain growth of TiB_2 grains was achieved by solution and reprecipitation mechanism suggested by Rüdiger and Exner.[21] The effect of W on mechanical properties may not be appreciable because TiB_2 grain consisted of core/rim structure behaves as a single grain, as evidenced by Fig. 4 (b) and (c).

The measured electrical resistivities were also summarized in Table 1. SiC is inherently a semiconductor and TiB_2 is inherently a good conductor. The SiC-TiB_2 composites had the electrical resistivity in-between SiC and TiB_2, as expected. The electrical resistivity of SiC-60 vol% TiB_2 composites is slightly lower than that of SiC-40 vol% TiB_2 composites. It may be due to the formation of continuous skeleton of conducting phase (TiB_2) in SiC-60 vol% TiB_2 composites. Core/rim structure in Fig. 4 may increase the electrical resistivity of the composites because the electrical resistivity of WC is higher than that of TiB_2. Hence, use of WC balls are not desirable for the fabrication of EDM SiC-TiB_2 composites. A material which has a resistivity of < 1 cm could be electrical discharge machined.[12] Our result shows that SiC-TiB_2 composites with $>$ 40 vol% TiB_2 could be electrical discharge machined. Fig. 5 shows an example of EDM for SiC-40 vol% TiB_2 composites.

IV. Conclusions

Electrical discharge machinable SiC-TiB_2 composites have been fabricated by hot-pressing. Addition of TiB_2 improved the flexural strength and fracture toughness and decreased the electrical resistivity of SiC. Flexural strength and fracture toughness of the SiC-40 vol% TiB_2 composites were 40% and 60%, respectively, higher than those of monolithic SiC. SiC-TiB_2 composites with>40 vol% TiB_2 had a resistivity lower than $10^{-2}\ \Omega \cdot$ cm and could be electrical discharge machined.

Fracture mode of the SiC-TiB_2 composites was mostly intergranular and cracks were deflected by the TiB_2 particles. The toughening was mainly due to the crack deflection with some possible contribution from crack branching or microcracking.

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