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Fabrication and Characterization of Electrical Discharge Machinable Si_3N_4-TiN Composites

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Electrical discharge machinable Si_3N_4 was fabricated with the addition of 20-60 vol% TiN by gas pressure sintering. Their sinterability, microstructure, mechanical and electrical properties were characterized as a function of the TiN content. The addition of TiN up to 20 vol% increased the flexural strength and fracture toughness as compared with those of the monolithic Si_3N_4. For the TiN content higher than 40 vol%, the electrical resistivity was lower than 10^2 Ω cm. The Si_3N_4 with the addition of 40 vol% of TiN appears to have the optimum considerable sinterability, mechanical and electrical properties, and machinability. A microstructural analysis showed that the enhanced toughening was due to the crack deflection.

Key words : Si_3N_4-TiN composites, Electrical discharge machinable ceramics

I. Introduction

Silicon nitride (Si_3N_4) is one of the most important engineering materials because of its excellent room and high temperature mechanical properties.[1] However, Si_3N_4 is very difficult to machine because of its high hardness.

In order to find out inexpensive machining routes, electical discharge machinable (EDM) Si_3N_4 ceramics have been fabricated by adding various conductive second phases such as carbides, nitrides or borides, or silicon carbide fibers.[2-5] Among these second phases, TiN is the most suitable material for EDM Si_3N_4 because of its high melting temperature, stiffness, hardness and electrical conductivity. Furthermore, addition of TiN improves mechanical properties of Si_3N_4 ceramics due to the reinforcing effect of TiN.[2,3]

In this study, EDM Si_3N_4 were prepared with the addition of 20, 40 and 60 vol% of TiN by gas pressure sintering. The effects of TiN content on the mechnical and electrical properties, and machinability were studied for an optimum EDM Si_3N_4 composition.

II. Experimental

A Si_3N_4 powder (E-10, Ube Industries, Tokyo, Japan) was mixed with 0-60 v/o TiN powder (Grade C, HCST, Berlin, Germany) and sintering additives in methanol using Si_3N_4 balls and a polyethylene jar. Sintering additives were 2 wt% Y_2O_3 (> 99.9%, Johnson Matthey Korea Ltd.) and 1 wt% Al_2O_3 (AKP30, Sumitomo Chemical Co., Tokyo, Japan). The mixed slurry was dried in an evaporator (WB2000, Heidolph, Germany) and sieved through a 60 mesh screen. The powder was uniaxially pressed into a bar shape and cold isostatically pressed under 140 MPa. The green compacts were sintered at 1800°C under 2 MPa of nitrogen for 2 hrs followed by a second step 1900°C under 8 MPa of nitrogen for 2 hrs.

Densities were measured using the Archimedes method. Theoretical densities were calculated based on the rule of mixtures. Sintered specimens were cut and surface-grounded using an 800 grit diamond wheel into $4\times3\times25$ mm bars. Four-point flexural strengths were measured at room temperature with the outer span of 20 mm and inner span of 8 mm. Fracture toughnesses were measured by the indentation method[6] with a load of 196 N. The samples were etched in molten NaOH for the observation of microstructures by a scanning electron microscopy (SEM). Foils were prepared by ion milling and the grain boundary phases were characterized by a transmission electron microscopy (TEM). The electrical conductivity was measured using a 4-point probe method. EDM was done using a wire electrode (Cu-30% Zn wire, 0.25 mm-diameter).

III. Results and Discussion

All compositions were sintered close to theoretical densities without difficulty (Table I).

Fig. 1 shows the microstructures of various Si_3N_4-TiN composites prepared in this study. The elongate grains are Si_3N_4, and the equiaxial grains are TiN. It shows clearly the role of TiN as grain growth inhibitor. Monolithic Si_3N_4 reveals large exaggerated grains among small columnar grains, which is typical of Si_3N_4 sintered with a

small amount of additives at relatively high temperatures.[7,8] With the addition of 20 vol% of TiN, abnormal grain growth is largely suppressed and a fairly uniform microstructure of columnar grains of Si_3N_4 is produced. With further addition of TiN, the TiN phase becomes the matrix and the growth of Si_3N_4 grain is completely ceased.

Fig. 2 shows TEM photomicrographs of the Si_3N_4-40 vol% TiN composites. Glassy phases are found at triple junctions and Si_3N_4-TiN interfaces eventhough small amount of additives were used in this study. This feature is almost identical to numerous observations found in Si_3N_4 ceramics.[9,10] It is expected, therefore, that the prepared samples would behave similarly to monolithic Si_3N_4 ceramics showing degradation of strength and creep at high temperatures which are caused by the liquid phase at grain boundaries.[10-12]

Table 1. Sintered Densities and Electrical Resistivities of the Si_3N_4-TiN Composites.

Composition (vol%)	g/cm^3	Density % of theoretical density	Electrical resistivity (20°C, Ω·cm)
0	3.21	100	$> 10^{10}$
20	3.64	99.9	1.0×10^{8}
40	3.99	99.8	1.7×10^{-3}
60	4.43	99.8	4.2×10^{-4}

Fig. 3 shows the flexual strength and fracture toughness of Si_3N_4-TiN composites in terms of the TiN content. Both properties show their maxima at 20 vol% addition of TiN. These results can be explained by referring the microstructures shown in Fig. 1. For the monolithic Si_3N_4, the large exaggerated grains of Si_3N_4 act as crack-initiating flaw (Fig. 4) resulting a relatively low strength.[13] With the addition of 20 vol% TiN, large exaggerated grains are disappeared and the processing-related flaws such as pores act as crack-initiating flaw (Fig. 4). Therefore, higher strength could be expected since the size of crack-initiating flaws are decreased from the large exaggerated grains to the small pores. Furthermore, uniform columnar grains of Si_3N_4 interlock each other making the material stronger and tougher. With further ad-

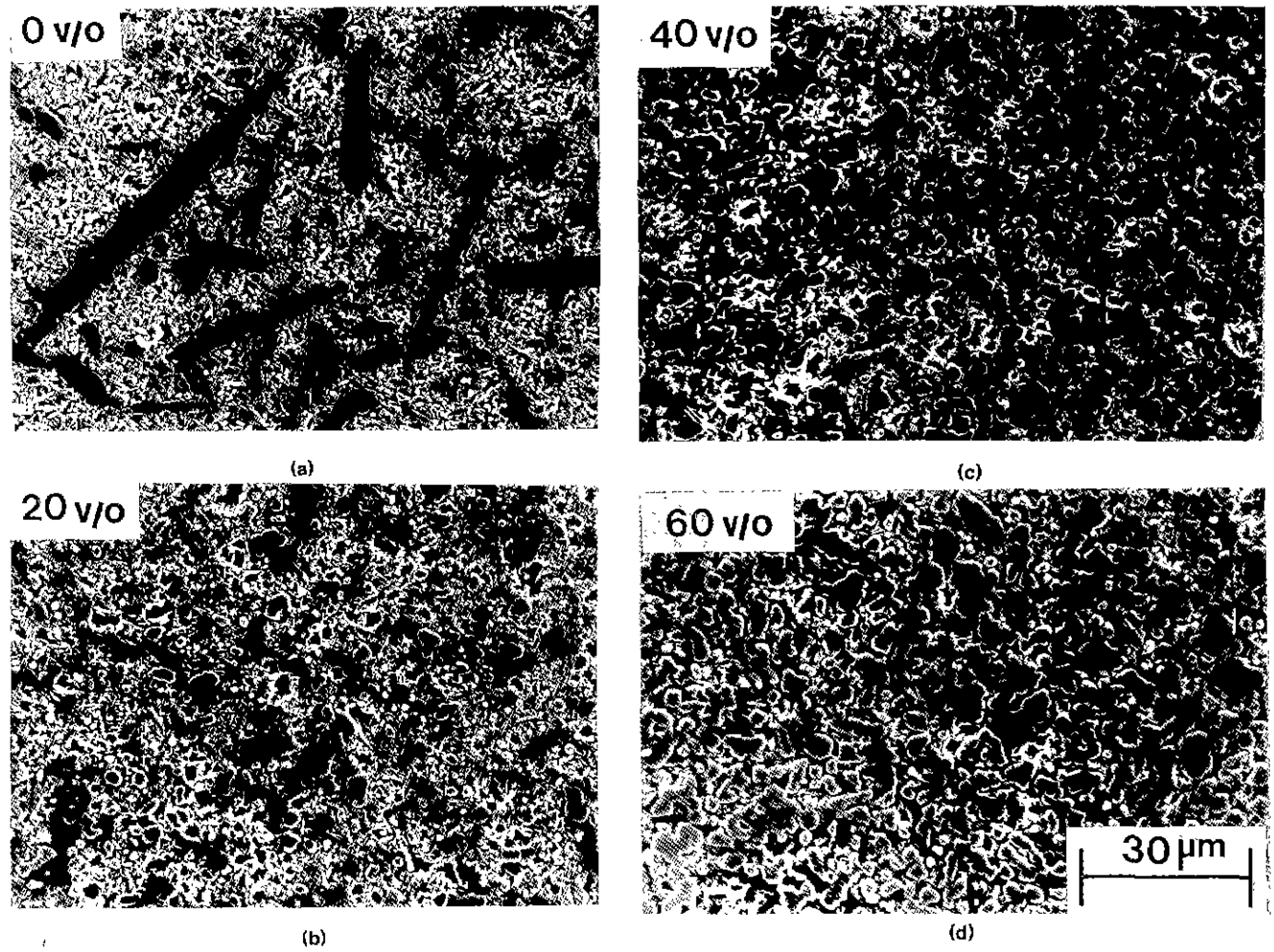

Fig. 1. Microstructures of the polished and etched surfaces of Si_3N_4-TiN composites; (a) 0 v/o TiN, (b) 20 v/o TiN, (c) 40 v/o TiN and (d) 60 v/o.

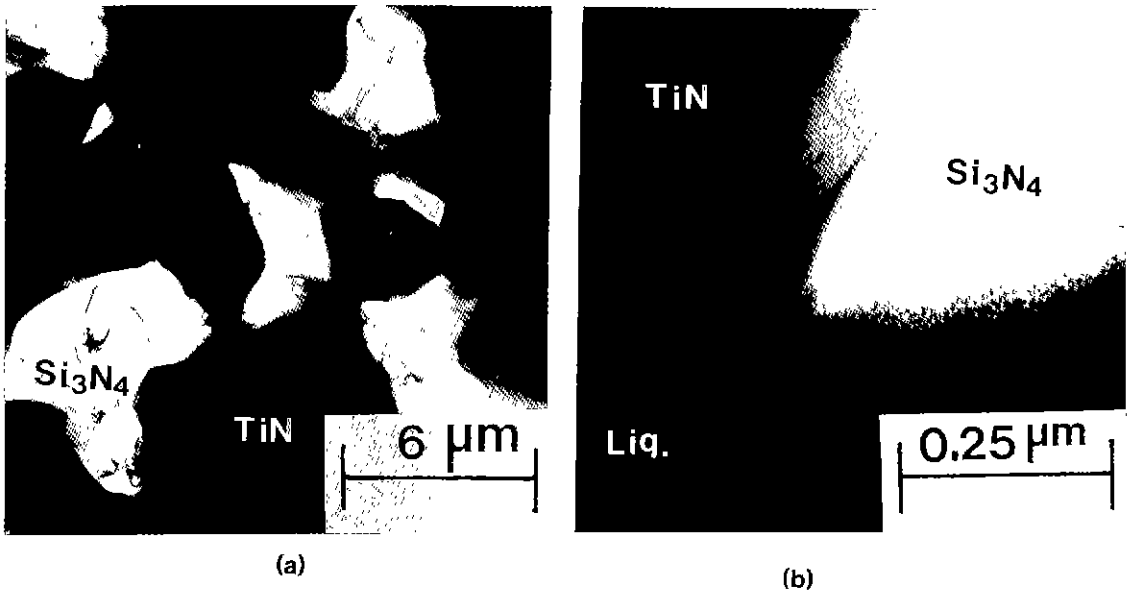

Fig. 2. TEM photomicrographs of Si_3N_4-40 vol% TiN composites showing liquid phase at triple junutions and Si_3N_4-TiN interfaces; (a) low magnification and (b) high magnification.

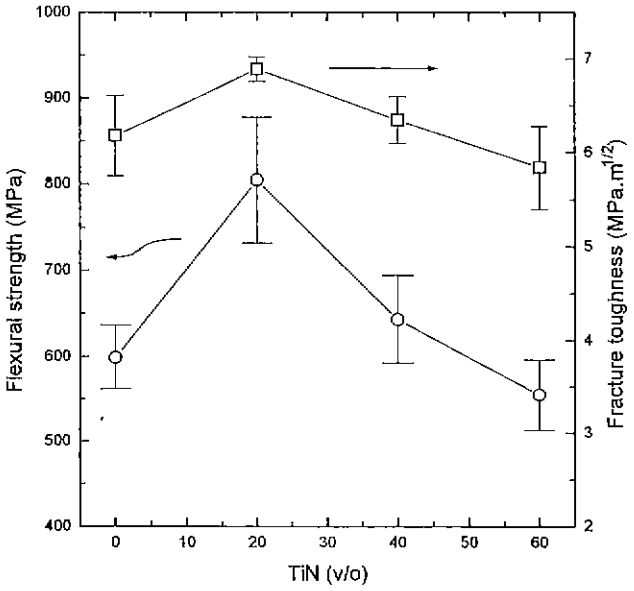

Fig. 3. Flexural strengths and fracture toughnesses of the Si_3N_4-TiN composites as a function of the TiN content.

dition of TiN, the toughening and strengthening effect of columnar Si_3N_4 grains do not exist since equiaxial TiN grains become the dominant phase.[14]

Fig. 5 shows the change of crack propagating behaviors with the addition of TiN. In monolithic Si_3N_4, it can be seen that the large exaggerated grains of Si_3N_4 do not contribute to toughening by letting the crack go through (transgranular fracture mode). In Si_3N_4 with 20 vol% TiN, however, typical crack deflection by columnar Si_3N_4 and TiN grains takes place. Therefore, it is believed that the slightly higher fracture toughness shown at 20 vol% of

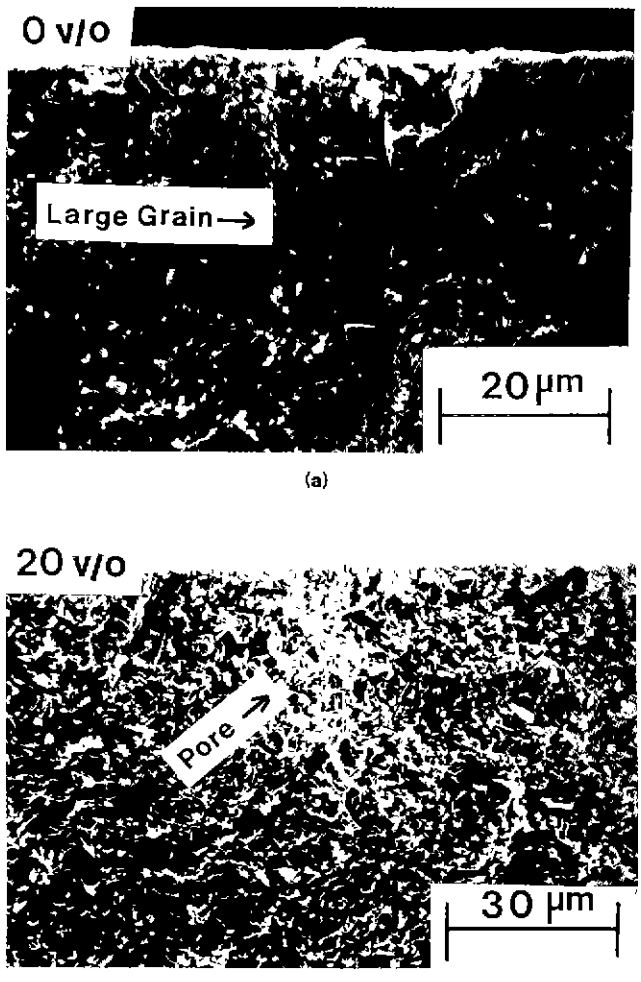

Fig. 4. Typical fracture origins of Si_3N_4-TiN composites; (a) 0 v/o TiN and (b) 20 v/o TiN

TiN is mainly due to the crack deflection by Si_3N_4 and TiN grains without transgranular fracture of large exaggerated grains. On the other hand, the lower fracture toughness of monolithic Si_3N_4 is due to the transgranular fracture of large exaggerated grains.

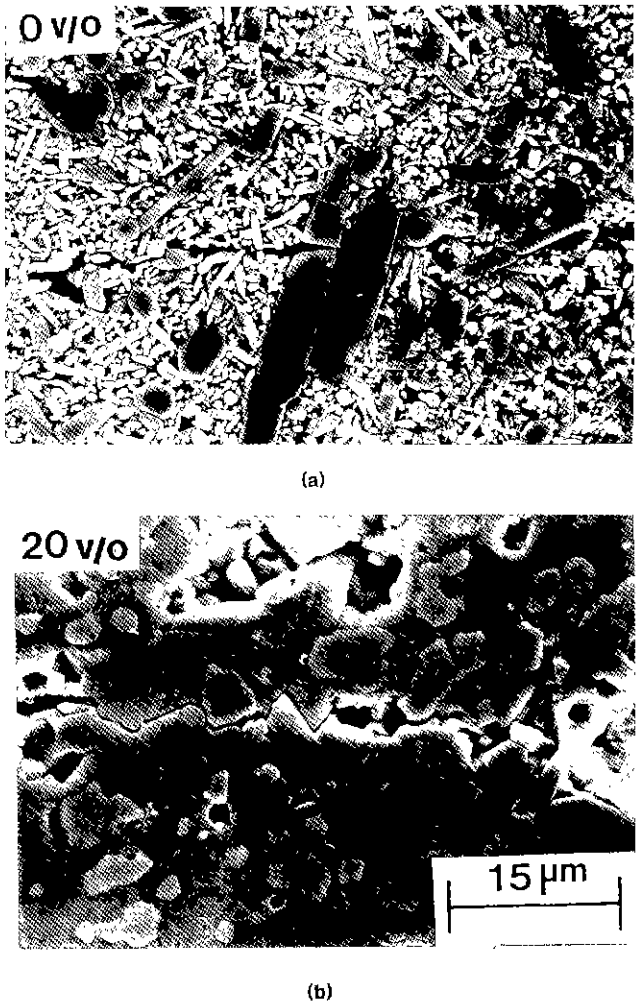

Fig. 5. Typical crack propagations of the Si_3N_4-TiN composites induced by a Vickers indenter; (a) 0 v/o TiN and (b) 20 v/o TiN.

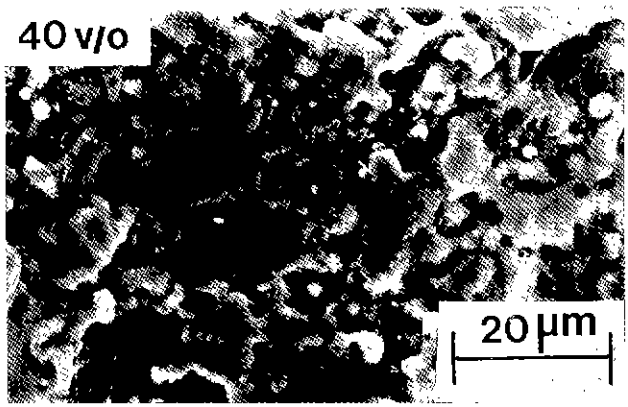

Fig. 6. Morphology of the electrical discharge machined surface of Si_3N_4-40 vol% TiN composites showing melted and oxidized products of Si_3N_4 and TiN.

The electrical resistivity of Si_3N_4-TiN composites as a function of the TiN content is shown in Table I. As expected, the electrical resistivity is decreased with the amount of TiN. A material with a resistivity lower than 1 Ω·cm could be electrically discharge machined,[15] but a resistivity lower than 10^{-2} Ω·cm is preferred for Si_3N_4 for good machinability.[3] Tests showed that Si_3N_4-40 vol% TiN composites (10 mm thick) could be cut at the rate of 10.2 mm/min and it's average surface roughness (R_a) was 4.476 μm. The machined surface was covered by melted and oxidized products of Si_3N_4 and TiN (Fig. 6).

IV. Conclusion

Electrical discharge machinable Si_3N_4 has been fabricated by adding TiN. When Si_3N_4 is sintered with a small amount of additives (3 wt%) at a relatively high temperature, the addition of TiN can improve the flexural strength and fracture toughness by suppressing exaggerated grain growth of Si_3N_4. Si_3N_4 containing 40 vol% of TiN showed a flexural strength and fracture toughness comparable to those of monolithic Si_3N_4, and a low electrical resistivity enough to give good electrical discharge machinability.

Acknowledgement

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