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Effect of the electrical discharge machining on strength and reliability of TiN/Si_3N_4 composites

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Abstract

Conductive TiN/Si_3N_4 hot pressed composites were processed by electrical discharge machining (EDM) and their microstructure and conductivity investigated. The dependence of surface texture, surface roughness, and materials removal rate on electrical discharge machining conditions including working voltage and feed rate were analyzed. The flexural strength and strength reliability in terms of Weibull modulus of TiN/ Si_3N_4 processed by EDM and conventional cutting and polishing are compared. Higher working voltage and current, as well as higher content of TiN result in greater material removal rate. Comparison of the flexural strength and Weibull modulus of the composites processed by EDM with conventional polished samples revealed that the surface with low roughness lead to an increase in strength and reliability for electrical discharge machining.

Keywords: D. Silicon nitride; Titanium nitride; Electrical discharge machining

1. Introduction

Silicon nitride and its composites have been recognized as promising engineering materials for their high performance and excellent properties such as high strength at high temperature, oxidation resistance, thermal shock resistance, wear and creep resistance, etc. [1–5]. Unfortunately, the difficulties in machining and finishing these materials limit their applications. Therefore, recent developments in silicon nitride composites are focused not only on the improvements of strength and toughness, but also on different possibilities for massive production and cost reduction in manufacturing. A successful approach is to incorporate electrically conductive reinforcements such as TiN, TiC, TiB_2 and ZrB_2, etc. into the silicon nitride matrix. The composites are expected to have excellent properties for wear and cutting tool applications [4–8]. More importantly, the incorporation of a certain content of these conductive reinforcements makes the composite electrically conductive so that electrical discharge machining (EDM) can be used. Among all above mentioned materials, TiN-reinforced silicon nitride composite has relatively high strength, low density and good electric conductivity [9]. It has been attracting much research attention because of EDM applicability and the potential for making cost-effective and complex shapes of high-temperature heaters and igniters [5].

Recently, wire EDM and die sinking EDM of conductive particle reinforced Si_3N_4 composites were investigated in terms of composite preparation, microstructure and EDM feasibility, etc. [10–15]. In present research, the effects of EDM conditions on properties, reliability and microstructure of hot pressed TiN/Si_3N_4 composites are investigated. The reliability of the composites machined by EDM is determined by Weibull distribution analyses of flexural strength and is expressed in terms of Weibull modulus. The miniaturized structure and surface morphologies of TiN/Si_3N_4 by EDM were also analyzed. Microstructure evolution, material removal rate and surface finish under various machining conditions are discussed.

2. Experimental procedure

Si_3N_4 (UBE SN-E10) powder was mixed with 2 wt.% Al_2O_3(16SG, Alcoa, USA, 0.5 μm) and 6 wt.% Y_2O_3 (5603, Molycorp, USA, 1.8 μm) by ball milling for 24 h

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in a polyurethane container with high-purity silicon nitride balls and ethanol as medium and solvent. The ratio of ball, charge and vehicle was 6: 1: 5 in weight. Titanium nitride powder of 3.5 μm average particle size (H.C.Stark) was then added to the above slurry. The slurry was dried in a rotary evaporator, and the dried agglomerates were ground with a mortar and pestled to pass through a 100 mesh sieve to pulverize the aggregates. Samples with dimension of $\Phi 50\times 8$ mm were hot-pressed in a graphite die (Fuji Dempa High Multi 5000) at 1850 °C for 1 h and at 24.5 MPa. High purity nitrogen (1 atmosphere) was used as a protection gas all the way through the hot-pressing procedure. After hot pressing some samples were plasma etched for 2 min under flowing CF_4 and O_2 (the flow rate ratio was 93:7) in a radio frequency (RF) sputtering system (Plasma-Them Inc., Series 70). The samples were ultrasonically cleaned prior to be examined by SEM. Samples of $4\times 4\times 30$ mm were cut using wire-EDM from the sintered bodies, and then ground and polished for electrical resistivity measurements. Electrical resistivity (ρ) was measured on the polished surface using a four-point probe method (Napson corporation, RG-7, Japan). Bending test samples were also cut into bars with dimensions of 3.5 mm×4.5 mm×45 mm using wire-EDM under different conditions shown in Table 2. Some of them were directly used as bending test samples, but some others were polished and then went to bending test for comparison purpose. Bending test was conducted in the four-point bending mode on a universal testing machine (Instron 8511) with inner and outer spans 20 mm and 40 mm, respectively, and loading rate of 0.5 mm/min for all tests. The strength was then analyzed by a series of related techniques based on the Weibull distribution [16]. The mechanical and electrical properties of 30 vol.%TiN/Si_3N_4 and 40 vol.% TiN/Si_3N_4 composites conventionally prepared are summarized in Table 1. So-called conventional preparation means hot pressing, cutting into bars using diamond saw, grinding and polishing as for normal ceramic materials. The data in Table 1 is the reference for EDM study of TiN/Si_3N_4 composites.

Electric discharging machining tests (Wire-EDM) were conducted on Fanuce Robocut-α-OA EDM equipment. Three different cutting conditions of the wire EDM, namely coarse, medium, and fine cutting of the TiN/Si_3N_4 composites are described in Table 2.

3. Results and discussion

3.1. Microstructure

Fig. 1 shows SEM micrographs of plasma etched surfaces of Si_3N_4 containing 0, 30 and 40 vol.% TiN hot-pressed at 1850 °C for 1 h. Si_3N_4 containing 0% TiN (Fig. 1a) exhibited a microstructure with rod-like β-Si_3N_4 distributing within fine matrix grains (Fig. 1a), a typical microstructure of in-situ reinforced silicon nitride [17–19]. The microstructures shown in Fig. 1b and c belong to Si_3N_4 composites with 30 vol.% and 40 vol.% TiN dispersions, respectively. TiN/Si_3N_4 composite features a duplex microstructure in which TiN particles distribute in the Si_3N_4 matrix. As compared with Fig. 1a, the average grain size of silicon nitride in the matrix (Fig. 1b and c) was considerably reduced by adding TiN. It is suggested that TiN addition inhibits the rod-like grain growth of β-Si_3N_4. Fig. 1b and c also show that that a large number of TiN grains in the Si_3N_4 matrix could be connected to each other and formed a thorough electrical conductive network leading to the increase in electrical conductivity [20].

3.2. Material removal rates

In EDM processing, the workpiece and electrode do not contact each other. The material removal rate is an important parameter, which is usually evaluated together with the surface roughness and machining ability. Fig. 2 shows the average material removal rate as func-

Table 1
Mechanical properties of TiN/Si_3N_4 composites

Composition (vol.%)	Flexural strength (MPa)	Fracture toughness (MPa m1/2)	Electrical resistivity (Ω cm)
30% TiN	530±30	7.35±0.5	6.8×10^{-2}
40% TiN	510±35	7±0.4	1.25×10^{-3}

Table 2
Wire electrical discharge machining conditions with brass wire a 0.25 mm diameter

Machining condition	Average working voltage (V)	Off time (μs)	Average working current (A)	Surface roughness Ra (μm)
Coarse	85	8	1.7	2.52
Medium	65	8	0.7	2.3
Fine	50	8	0.2	1.2

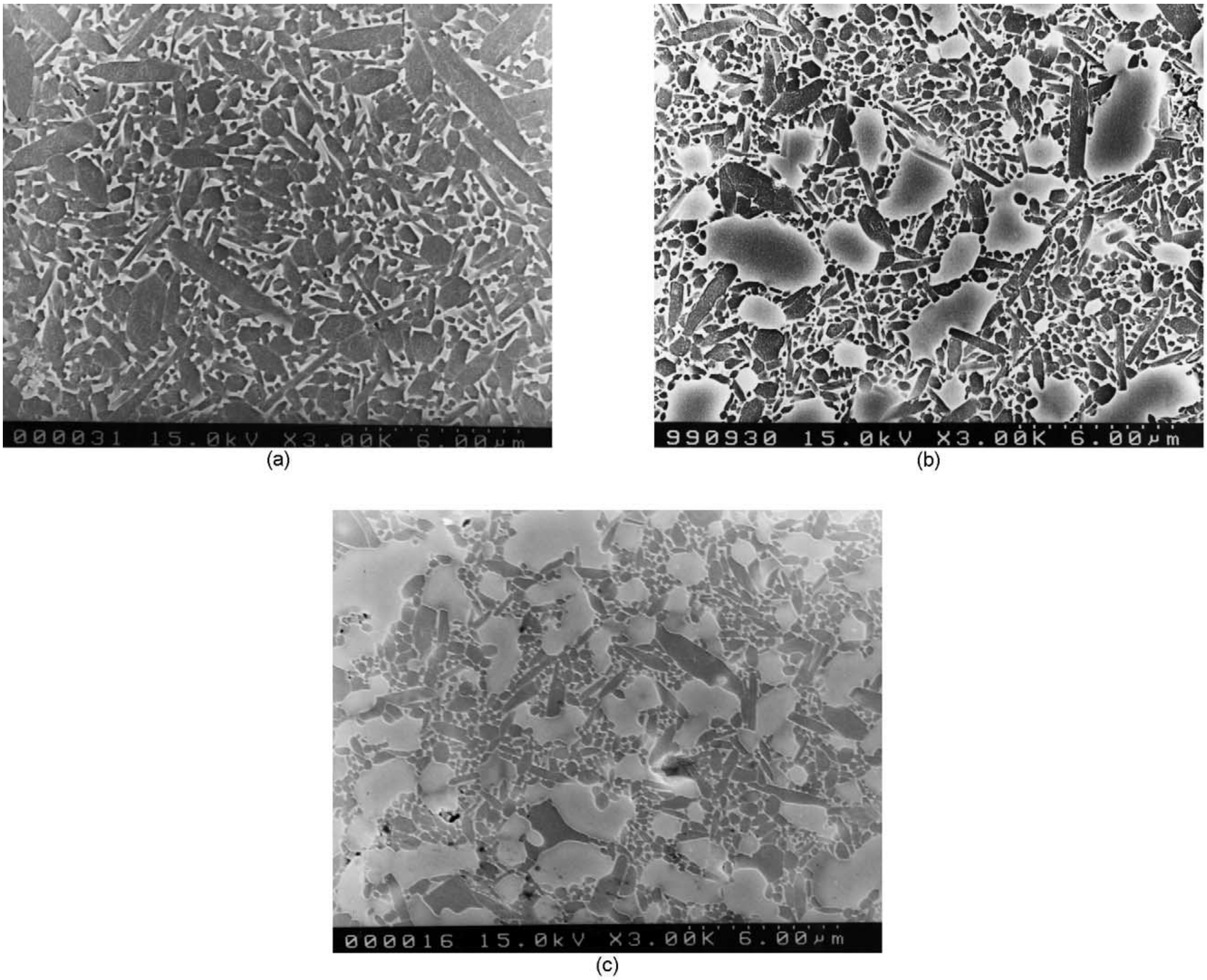

Fig. 1. Scanning electron micrograph of polished and plasma etched surfaces of silicon nitride composites sintered at 1850 °C under mechanical pressure of 25 MPa (a) 0 vol.% TiN, (b) 30 vol.% TiN and (c) 40 vol.% TiN.

tion of working voltage obtained in our wire-EDM experiment, in which a tensioned brass wire with the diameter of 0.25 mm was used as the electrode. It can be seen in Fig. 2 that the average material removal rate increases with the increase in the working voltage and higher TiN content leads to higher material removal rate. The dependence of the material removal rate of 40 vol.% TiN/Si_3N_4 on the working voltage is much stronger than that of 30 vol.% TiN/Si_3N_4, particularly when the working voltage is over 77.5 V. The EDM behavior of higher TiN content composite (e.g. 40 vol.%TiN/Si_3N_4) has been found to be similar to the EDM behavior of metallic materials based on the curve displayed in Fig. 2. For 30 vol.% TiN/Si_3N_4 the material removal rate is just slightly increased, or can be approximately regarded as a constant, with the working voltage varying from 65 to 85V. Under the working voltage of 85 V the material removal rate of 40 vol.% TiN/Si_3N_4 composite is seven times as high as that of 30 vol.% TiN/Si_3N_4. This may suggest that higher TiN loading forms more perfect conductive network and promotes the decrease in electrical resistivity.

3.3. Surface analysis

For surface analyses, samples of 40 vol.% TiN/Si_3N_4 composite were processed using electrical discharge machining at different feed rates (mm/min) using a brass wire of 0.25 mm in diameter as the electrode. Fig. 3 displays the variation of the surface roughness with the feed rate of the EDM. It is evident that higher feed rate gives higher average surface roughness (R_a). The increase in surface roughness with the feed rate increased from 0.25 to 1.0 mm/min is almost linear and the increase rate is then lowered as the feed rate is over 1.0 mm/min. The surface roughness of 40vol%TiN/Si_3N_4 composite by

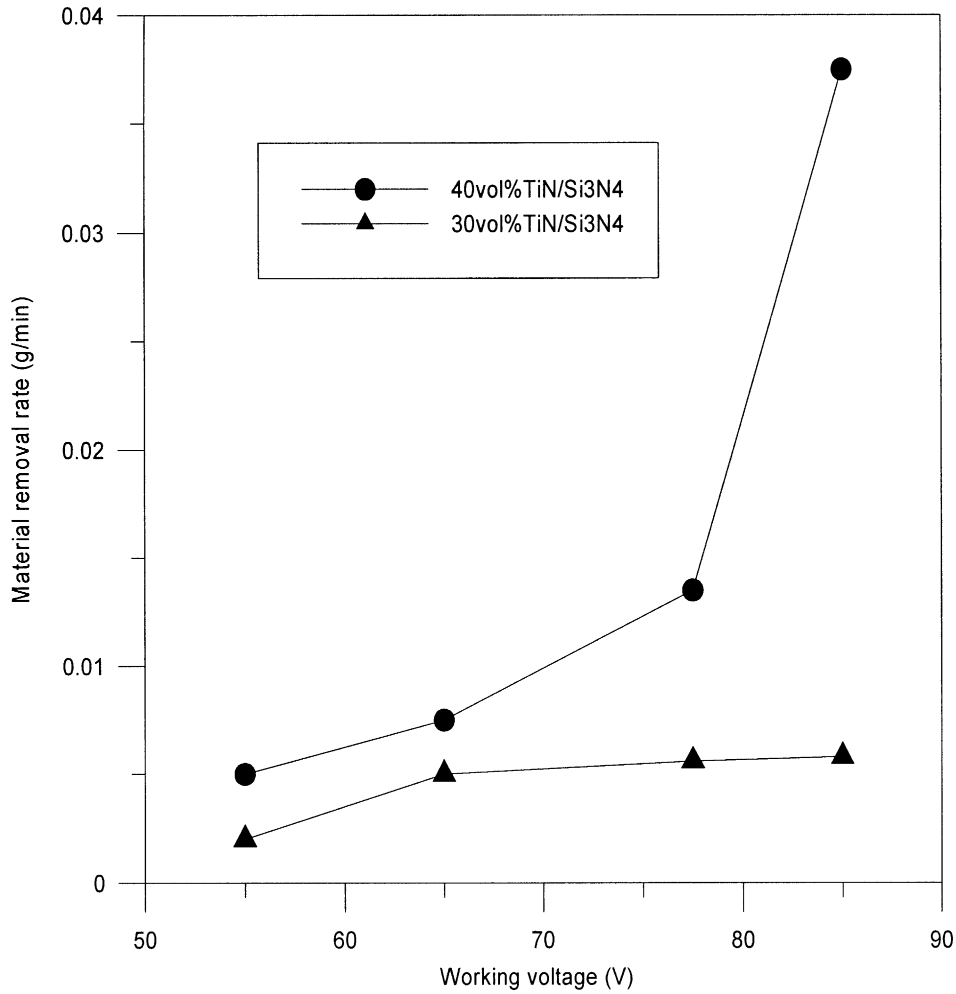

Fig. 2. Material removal rate at different working voltage during EDM of TiN/Si_3N_4 composite materials.

EDM under fine, medium and coarse conditions (Table 2) are also studied. Typical surface morphologies of 40 vol% TiN/Si_3N_4 sample after EDM machining under the three conditions are shown in Fig. 4(a)–(c), respectively. It is evident that the fine machining produced a surface with low roughness, but the surfaces created by medium and coarse machining have high roughness. Many craters that are large as compared with the texture can be seen on the surfaces by medium and coarse machining, not on the surface by fine machining.

EDM machined surface of 40 vol.% TiN/Si_3N_4 sample was further observed under SEM with higher magnifications. Fig. 5a is a typical micrograph obtained from this observation. The micrograph shows that the surface has a porous layer and a few of re-solidified droplets being planted on the layer (Fig. 5a). This can be simply attributed to the melting and evaporation of the material on the surface during the EDM machining [11]. For the same surface Ti and Si elemental distribution maps was performed using EDS and the results are shown in Fig. 5b and c, respectively. The re-solidified droplets are found Ti rich and Si poor, indicating that the resolidifed droplets were mainly TiN during the EDM process. This observation was confirmed by an EDS analysis on the re-solidified droplets, in which Ti was found dominant (Fig. 6).

3.4. Flexural strength of machined TiN/Si_3N_4

The flexural strength of the composites prepared in this way are listed in Table 1 as the reference for EDM study. The Weibull distributions for two groups of bars cut from 40 vol.%TiN/Si_3N_4 specimens by coarse and fine EDM without polishing are displayed in Fig. 7 and summarized in Table 3, in which the data from polished samples are also shown for comparison. The slope of a straight line that fits the data points perfectly is the Weibull modulus for each distribution. The average flexural strength and Weibull modulus of each group of specimens are shown related to the machined surface morphology. The average flexural strength of coarse and fine EDM specimens is 332 MPa and 379 MPa, and

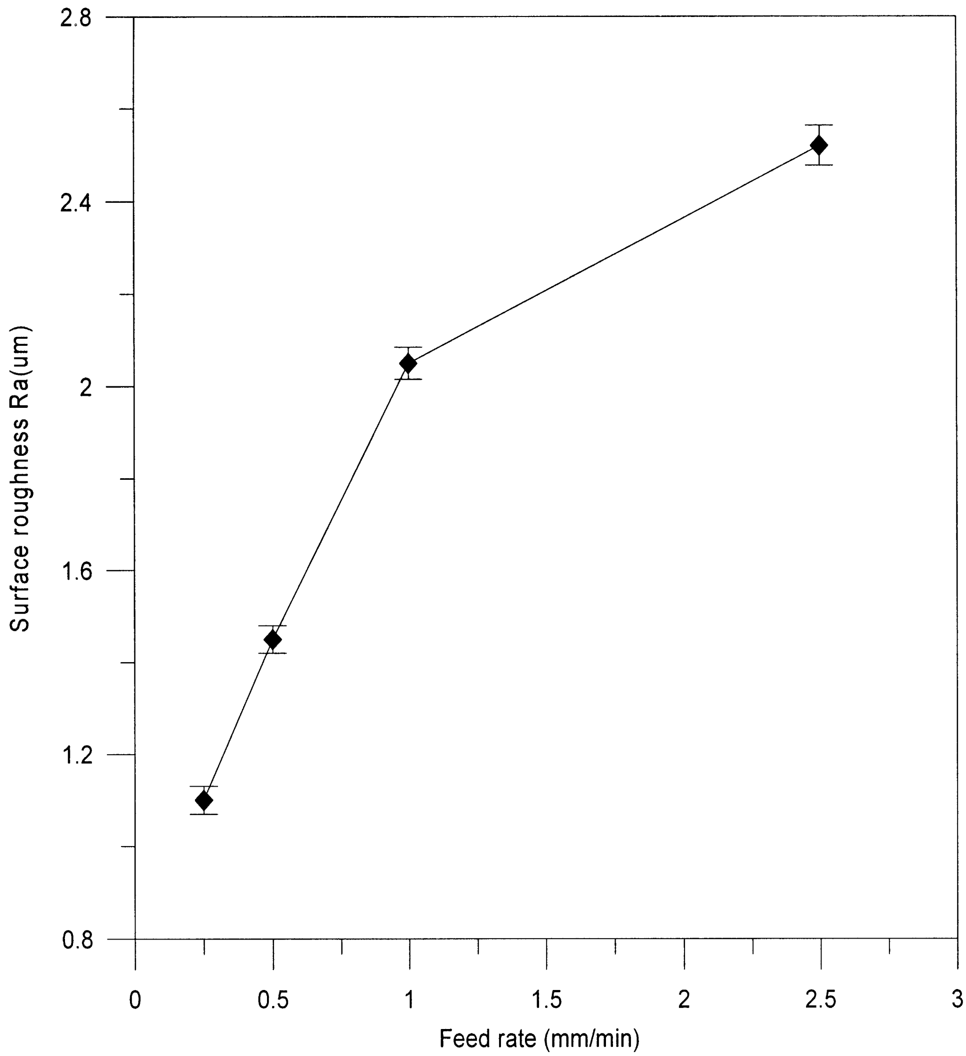

Fig. 3. Surface roughness of 40 vol.% TiN/Si_3N_4 composite at different feed rate in wire-EDM.

the corresponding Weibull modulus is 7.3, and 10.5, respectively. For polished samples the average flexural strength is 510 MPa and the Weibull modulus is 16.2. Fine EDM machining and polishing can give materials higher strengths and high reliability. This is in agreement with some of previous studies. For example, Mordecai et al. [21] reported that high Weibull modulus values were achieved using small spark energy and the indicated strength distribution was improved with surface texture refining.

Table 3
Weibull parameters for the strength distribution of EDMed 40 vol.% TiN/Si_3N_4 composite

Condition	Weibull modulus	Average flexural strength (MPa)
Coarse EDMed	7.3	332
Fine EDMed	10.5	379
Polishing	16.2	510

3.5. Micro-machining by EDM

Two examples of making use of EDM are shown Fig. 8. Fig. 8a is a set of parallel walls with 220 μm in width and 217 μm of spacing formed by EDM on the hot pressed 40 vol.% TiN/Si_3N_4 composite plate with thickness of 8 mm. A brass wire with diameter of 0.1 μm was used as the electrode. The EDM parameters including working voltage, working current and feed rate are 50 V, 0.2 A and 0.5 mm/min, respectively. The second example is a hole of 72 μm in diameter with good roundness drilled by EDM on the same composite of 0.8 mm in thickness shown in Fig. 8b. The drilling was done on a micro-EDM system with a rode electrode made of tungsten carbide. The drilling process was described in Ref. [9]. The successful demonstrations suggest that the EDM is an attractive machining technique, suitable for the formation of extremely hard and brittle, but conductive ceramic composites and for forming miniaturized structures on these materials.

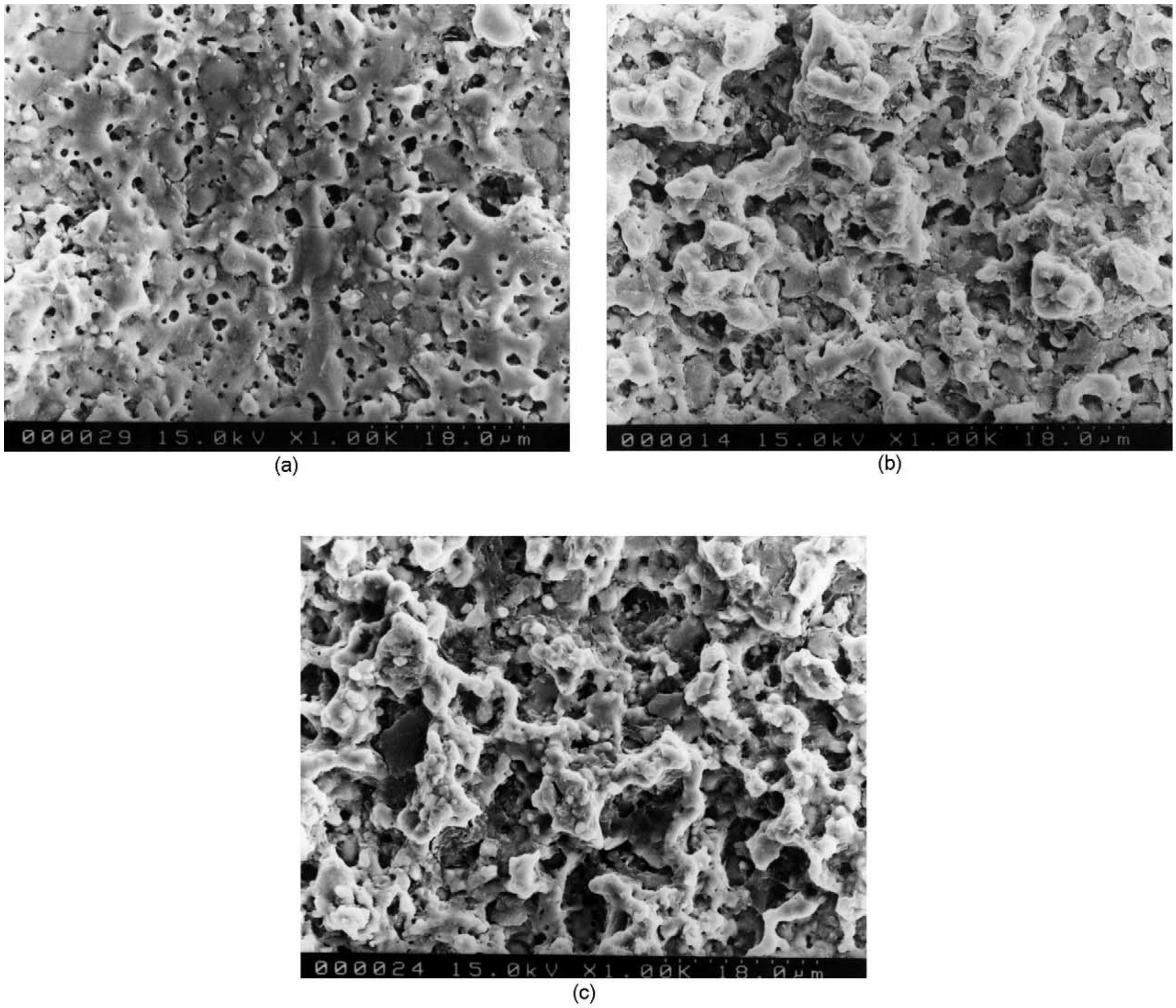

Fig. 4. Typical wire-EDM cutting surfaces of 40 vol.% TiN/Si_3N_4 composite under different cutting conditions, (a) fine, (b) medium, and (c) coarse.

4. Summary

Electrical discharge machining of TiN/Si_3N_4 composites was performed and the results suggest that EDM is an attractive machining technique, suitable for the formation of extremely hard and brittle, but conductive ceramic composites and for forming miniaturized structures on these materials. For TiN content higher than 30 vol.%, the electrical resistivity of the composite was approximately 10^{-2} Ω cm, so that EDM can be used for processing of this material. On the basis of this study, the following conclusions are drawn.

1. TiN/Si_3N_4 composite with higher content of TiN has higher electrical conductivity, leading to a higher material removal rate when processed using EDM. The material removal rate also increases with working voltage of EDM or higher content of TiN. At working voltage of 85 V, the material removal rate of 40 vol.% TiN/Si_3N_4 composite processed by EDM is 7 times higher than for 30 vol.% TiN/Si_3N_4 processed at the same condition. However, at working voltage of 55 V, it becomes twice as much as that for 30 vol.% TiN/Si_3N_4.
2. Microstructure analysis shows that the specimens of 40 vol.% TiN/Si_3N_4 composite processed with fine cutting had improved surface texture.
3. In comparison to the polished samples, the flexural strength of the samples processed by coarse and fine EDM decreased approximately 35% and 26%, respectively. The strength reliability of Weibull modulus is also lowered in turn as processed by fine and course EDM as compared with polished samples.
4. Micro-EDM is capable of forming small dimension and complex structure of TiN/Si_3N_4 composite.

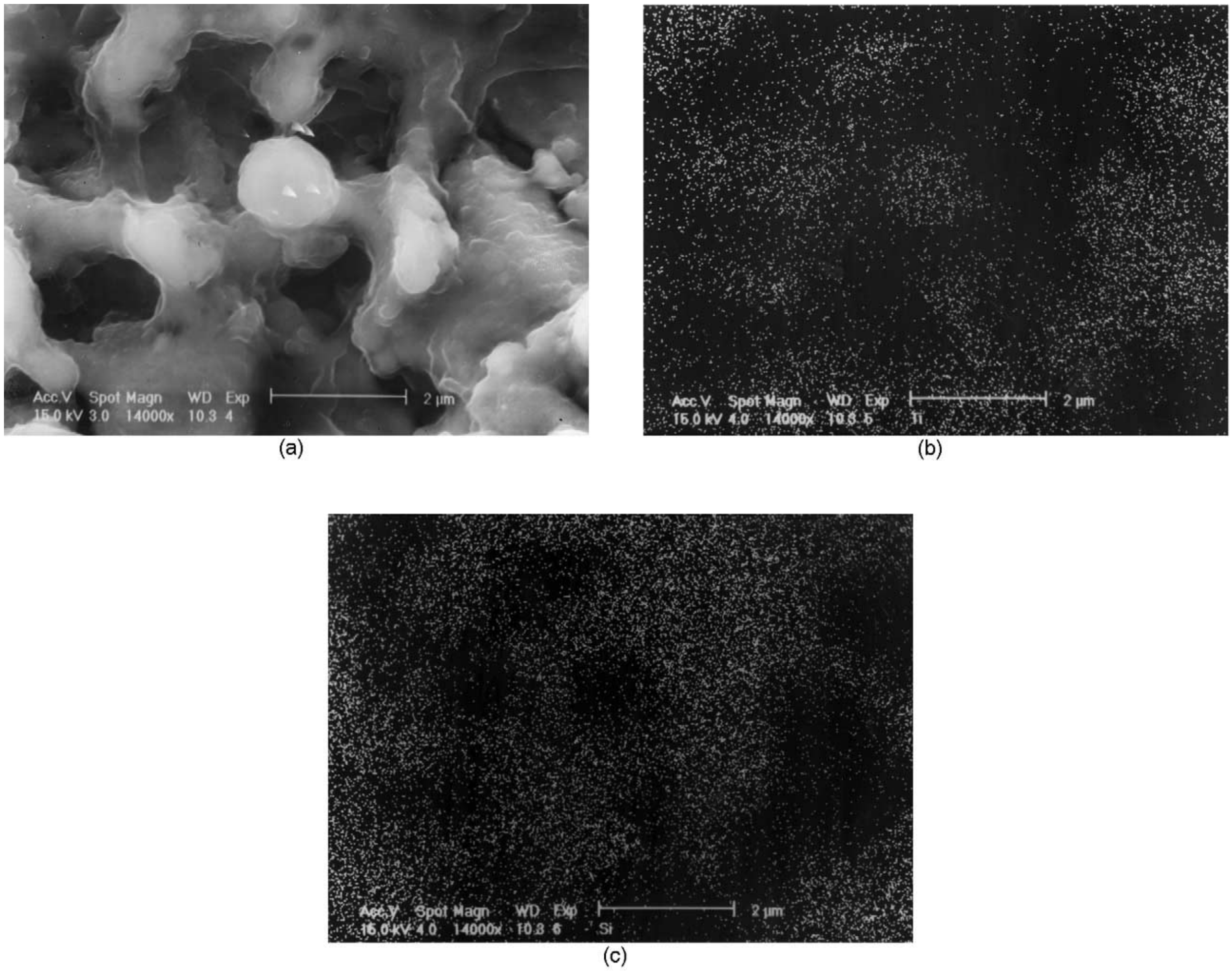

Fig. 5. Morphology of the EDM surface of 40 vol.% TiN composite showing, (a) melt-formation droplets, (b) Ti mapping (c) Si mapping.

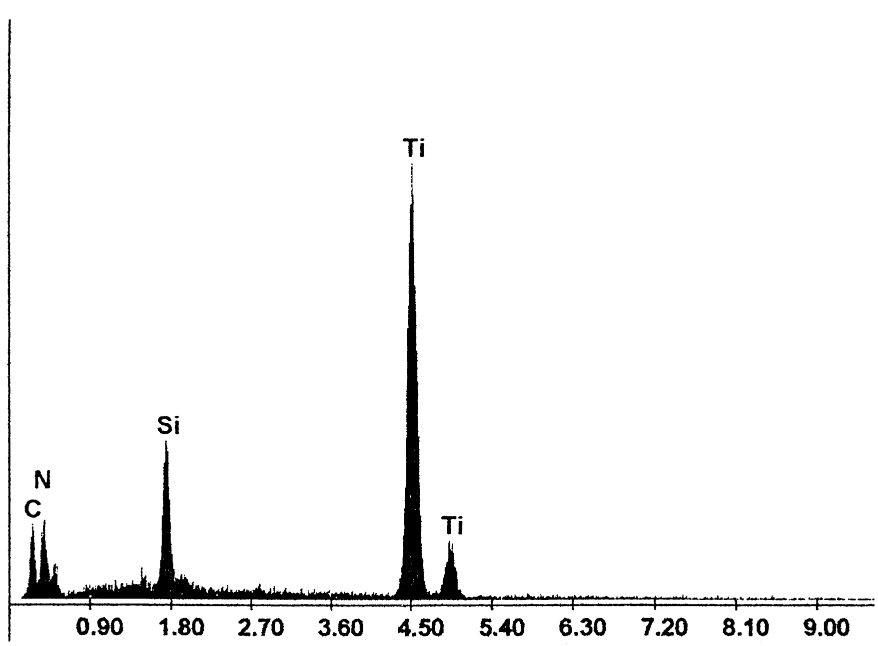

Fig. 6. EDS analysis of the resolidified melt from a resolidified droplets.

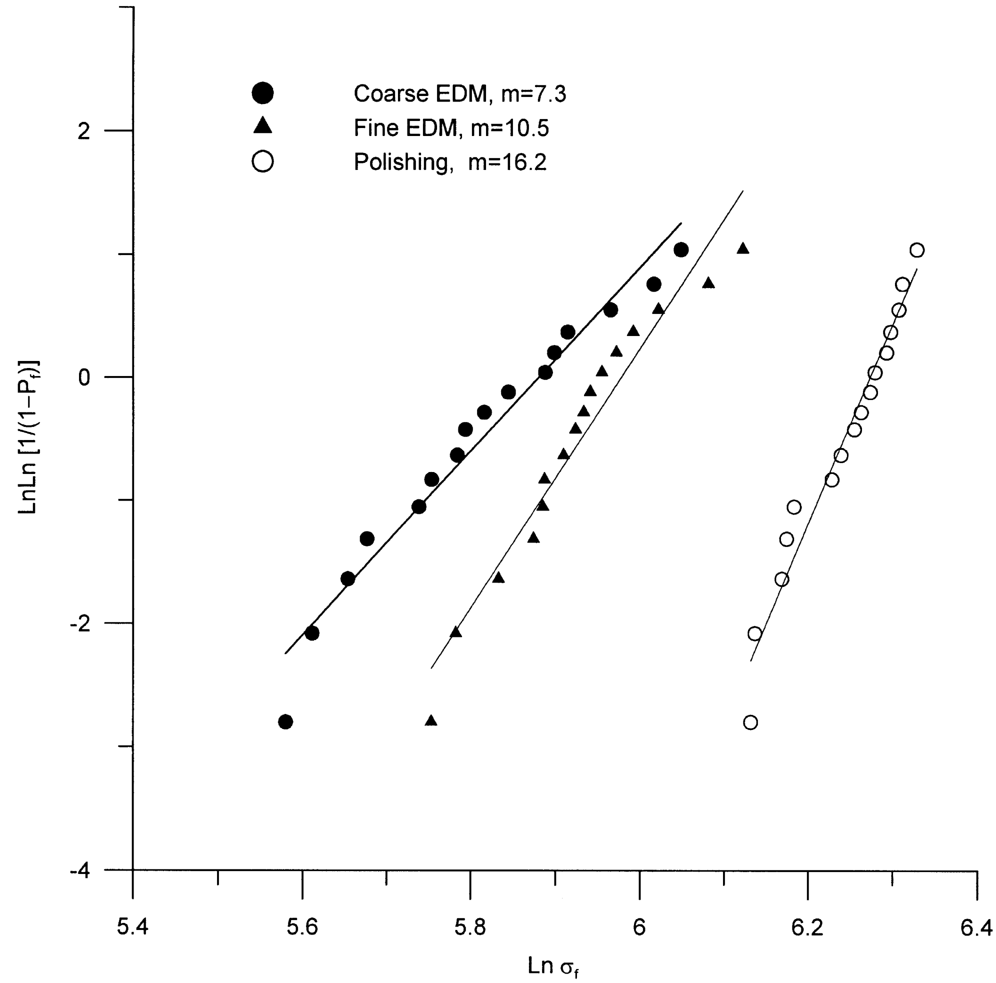

Fig. 7. Weibull plot of the strength distribution of the wire-EDMed and polishing of 40 vol.% TiN/Si_3N_4 composite.

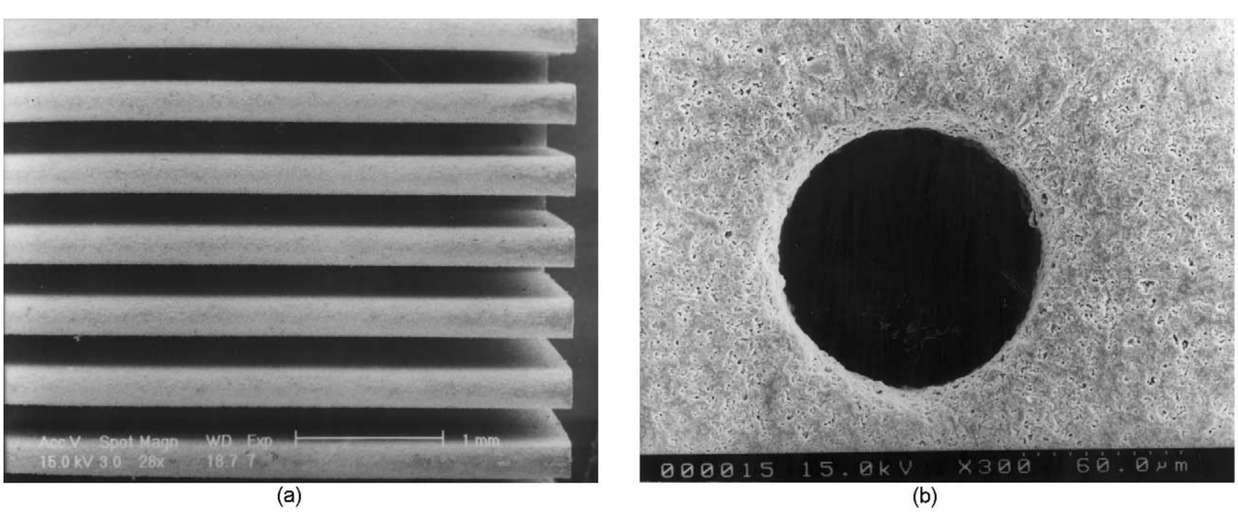

Fig. 8. (a) Vertical walls of TiN/Si_3N_4 composite material machined by wire-EDMed with brass wires of 0.1 mm dia, with width of 220 μm and spacing of 217 μm; (b) micro-hole machined by micro-EDM.

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