Wire Electrical Discharge Machining, Mechanical and Tribological Performance of TiN Reinforced Multiscale SiAlON Ceramic Composites Fabricated by Spark Plasma Sintering

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Abstract: An effective approach for preparing electrically conductive multiscale SiAlON-based nanocomposites with 10 wt.% and 20 wt.% of titanium nitride was developed. Fully dense samples were obtained by spark plasma sintering (SPS) at 1700 °C and 80 MPa for 30 min. The morphology of nanocomposites was observed using scanning electron microscopy and the effects of TiN particles on the mechanical properties and electrical resistivity were studied. It was found that the addition of 20 wt.% TiN increased the hardness and fracture toughness compared to the commercial ceramic analogue TC3030. Meanwhile, the presence of TiN particles reduced the flexural strength of the nanocomposites due to the shrinkage difference between TiN particles and ceramic matrix during cooling, which led to tensile residual stresses and, consequently, to changes in strength values. In addition, the electrical resistivity of nanocomposites decreased with the increase of TiN content and reached 1.6 × 10^-4 Ω·m for 20 wt.% amount of second phase, which consequently made them suitable for electrical discharge machining. In addition to enhanced mechanical and electrical properties, under identical conditions, SPS-sintered multiscale nanocomposites exhibited a higher wear resistance (more than about 1.5-times) compared to the commercial sample due to their higher toughness and hardness.

Keywords: ceramic nanocomposites; spark plasma sintering (SPS); mechanical properties; electrical conductivity; wire electrical discharge machining (WEDM); wear resistance

1. Introduction

Because of their exceptional thermomechanical and tribological characteristics, silicon nitride-based materials are used in several applications such as cutting tools, ball bearings, sealing elements, and engine components [1–4]. However, the covalent bonds between silicon (Si) and nitrogen (N) atoms cause difficulties in obtaining a fully dense material during sintering. Moreover, the decomposition of silicon nitride (Si₃N₄) starts at high temperatures (>1850 °C), when atomic particles become more mobile for material densification due to their low self-diffusion coefficient. Therefore, the hot-pressing method is commonly used for the fabrication of Si₃N₄ with high density. Thus, a new class of ceramics, SiAlONs, a more cost-effective alternative to hot-pressed silicon nitride, was developed. SiAlONs are formed when silicon nitride, aluminum oxide (Al₂O₃), and aluminum nitride (AlN) react together. α-SiAlON and β-SiAlON represent the two main phases of this type of ceramic. Each phase features its own properties, which can be contributed to the composites. The α-SiAlON has increased hardness, whereas the β-SiAlON has a high level
of fracture toughness. The content of these phases in composites can be adjusted and, consequently, this makes it possible to vary the properties of $\alpha$/\$\beta$-SiAlON for different applications. However, the usage of ceramics on the basis of Si$_3$N$_4$ is often limited by their poor toughness (3–5 MPa$\cdot$m$^{1/2}$) [5]. The fracture toughness of the ceramic materials can be improved by designing composite materials reinforced with carbides, nitrides, borides, silicides, etc. [6–13]. In particular, Acikbas et al. found that the addition of titanium nitride (TiN) into SiAlON matrix can enhance fracture toughness at room temperature. They also reported that TiN presence did not have any adverse effect on densification, phase assemblage, or solid solution parameter [14–16].

In addition, the processing of ceramics is a very costly, labor-intensive, and complicated process. Only diamond tools are used for machining of this kind of materials. Based on this, it is advisable to use electrochemical processing methods, such as electrical discharge machining (EDM). This method makes it possible to avoid labor-intensive and expensive operations while maintaining high workpiece quality.

However, EDM works only for electrically conductive materials. In order to be machinable by EDM, the workpieces should have an electrical resistivity of less than 1–3 $\Omega\cdot$m [9,11,17]. The presence of reinforcement phases, which also have good electrical conductivity, can enhance the electrical conductivity of SiAlON-based composites as well, but these phases must be uniformly distributed in the ceramic matrix. Apart from the problem of distribution of the second phase, densification of these composites is very difficult due to the thermodynamic incompatibility of the incorporated phases.

Based on this, the use of spark plasma sintering (SPS) appears promising. This sintering method produces fully dense ceramic composites. At the same time, the SPS temperature is lower than traditional sintering methods. This helps to avoid grain growth during the densification of materials [18–23].

The study of tribological performance of SiAlON composites is critical because friction and wear are inherent in the machining process. There have been many reports of the wear performance of Si$_3$N$_4$-based composites [24–31]. It was also noted that the addition of TiN phase reduces the friction due to the formation of a titanium oxide solid lubricant film and leads to a decreased wear rate of composites [32–35]. So far, however, there has been little discussion about tribological properties of SiAlON-TiN composites [36,37]. Sun et al. reported that a SiAlON composite with 30 wt.% addition of TiN showed a decrease in wear rate and a lower friction coefficient compared to the material without TiN due to the enhancement of heat-conducting properties, mechanical properties, and the tribo-chemical reaction [38,39].

Moreover, development of ceramic composites combining various phases at different length scales (i.e., micro or nano) would enable improvement of mechanical, electrical, and tribological properties, which are not available in traditional composites.

The purpose of this work, therefore, was to describe and analyze the behavior of the electromechanical and tribological properties of three multi-material composites, namely pure $\alpha$/\$\beta$ SiAlON ceramics, as well as the same ceramic with 10 wt.% and 20 wt.% TiN as reinforcement and as the electroconductive phase.

2. Materials and Methods
2.1. Composites Production

For this work, commercial $\alpha$-SiAlON and $\beta$-SiAlON ($d_{50} = 360 \pm 0.05$ nm and $d_{50} = 530 \pm 0.05$ nm, respectively, made by Plasmotherm, Moscow, Russia) and TiN ($d_{50} = 0.6$ $\mu$m, purity 99.9%, Plasmotherm, Moscow, Russia) powders were used. Powders were wet-mixed for 24 h in a plastic container using a multidirectional mixer Turbula (Tomasova Lea s.r.o., Pardubice, Czech Republic) with isopropyl as liquid media and Si$_3$N$_4$ grinding balls. The ratio in the starting powders between alfa- and beta-SiAlON was 90 wt.% and 10 wt.%, respectively. Obtained slurries were freeze-dried using LabConco FreeZone2.5 (Kansas, MO, USA) freeze dry system. This method of drying contributes to homogeneous distribution of powder mixture while avoiding sieving stage. More detailed
information about the operating mode has been presented by the authors of [10]. The obtained powder was placed in a die-punch setup made from isostatic graphite (grade C4, DonCarb Graphite, Rostov, Russia). Disks of 20 mm diameter and 3 mm height were produced using H-HP D-25 SD spark plasma sintering (SPS, FCT Systeme GmbH, Rauenstein, Germany) furnace with variation of temperatures (1600, 1650 and 1700 °C) with a heating rate of 200 °C/min under vacuum at 80 MPa axial pressure for a dwelling time of 30 min at the final temperature. Figure 1 shows the temperature-time diagram recorded in SPS apparatus during the process.

![Temperature-Time Diagram](image)

**Figure 1.** The temperature-time diagram recorded in the spark plasma sintering (SPS) apparatus during the process.

The temperature was controlled during sintering by a pyrometer situated at the top of the machine and focused at the center of the blank (3 mm over the top surface). The mixture of α- and β-SiAlON powders was used as a ceramic matrix to produce 3 compositions containing 0 wt.%, 10 wt.%, and 20 wt.% TiN. The sintered nanocomposites were labelled α-β0T, α-β10T, and α-β20T, where the numbers show the TiN weight content. Besides the SPS samples, commercial TC3030 (TaeguTec Ltd., Daegu, Korea) cutting inserts with cylindrical shape (Ø 19 mm) were used as a reference sample.

### 2.2. Microstructural and Mechanical Characterization

The phase composition was determined using X-ray diffraction (PANalytical, Almelo, Netherlands). The diffraction data were collected over a 2θ range of 20–44° with a step with of 0.02° and Cu-Kα (λ = 1.5405981 Å) radiation. Diffractometer was anode supplied with 60 kV and a current of 30 mA.

Bulk densities of the obtained samples were determined by the Archimedes method in distilled water. Prior to the microstructure study by scanning electron microscopy (SEM; VEGA 3 LMH, Tescan, Brno, Czech Republic), the samples were polished to a 1 μm finish. The Vickers hardness (HV) and fracture toughness (Kt) were calculated using the indenter load 19.6 N and 10 indents of each sample. The diagonal sizes of corresponding indentations and indentation-induced crack lengths were determined by SEM. A more detailed description of the methods used in this research has been presented in previous works [11,40]. The Young’s modulus values were calculated from indentation load-displacement curves after micro-indentation obtained with the Anton Paar Micro Indentation Tester (Graz, Austria). The fracture toughness was evaluated according to the method proposed by P. Miranzo and J.S. Moya [41].

In obtain the average matrix grain size, the SPS samples were thermally etched for 60 min at a temperature of 1600 °C.

The biaxial flexural strength (σf) was measured using the piston-on-3-ball method. The obtained specimens were placed, with the polished surface down, on 3 balls located 120° apart on a circle with a diameter of 10 mm as the tensile side. A piston positioned above the center of the 3-ball support directly applied the load to the unpolished side, producing a biaxial flexural loading condition. Twelve specimens were tested at room temperature with a 5 kN testing machine applying a piston speed of 1 mm/min until failure occurred.
2.3. Tribological Behavior

Tribological behavior was evaluated using Calotest equipment (CSM Instruments, Needham, MA, USA). A rotating steel ball with a diameter of 20 mm was pressed downward on the sample surface with a 0.2 N load. The ball was made of 100Cr6 (SAE52100, UNS G52986) steel (Table 1). The hardness, mean roughness, and rotating speed of the ball was 210 HB, 1.25 µm, and 949 r/min, respectively.

Table 1. Chemical composition of the steel ball.

| Chemical Composition, % |
|-------------------------|
| C  | Si  | Mn  | Cr  | S   | P   | Fe   |
| 1.00 | 0.25 | 0.35 | 1.50 | 0.008 | 0.025 | balance |

The rotating sphere was impressed at the center of the samples and the contact load always remained constant. A spherical depression was created in the contact zone of the material using the drop method of Calotest superfine water base diamond suspension with an average particle size of 0.2 µm. The suspension wore down the sample in a controlled manner and provided reproducible test results. For each composite, 5 experiments with different run times were carried out at room temperature.

The wear rate \( W \) was given by the Equation (1):

\[
W = \frac{\Delta V}{F_N \cdot S}
\]

where \( \Delta V \) is the volume loss after the wear tests (mm\(^3\)), \( F_N \) is the applied load (N), and \( S \) is the sliding distance (m). The Calotester unit was equipped with a cycle counter, which showed the number of revolutions of the steel sphere. Therefore, the sliding distance was calculated using this number and circumference of the ball. Since the size of the contact area was much smaller than sphere diameter, the contact area was considered as the point of contact during the whole test. The depth and volume changes after the friction tests were estimated by 3D Taylor Hobson’s surface profilometer Talysurf CLI 500 (Leicester, UK), which mapped the measured area by tip-sample analysis of the contact side. Wear scars were examined under a SEM.

2.4. Electrical Properties and Wire-Electroerosion Machining (WEDM)

To obtain an indication of electrical properties of materials, the 4-probe method was applied using a current sourced model, Keithly 6220 current source (Cleveland, OH, USA) and a Keithly 2182 nanovoltmeter (Cleveland, OH, USA).

The samples were processed using a wire electrical discharge machine (WEDM) (Seibu M500SG, Seibu Electric & Machinery Co., Koga, Japan) machine. Deionized water was used as a dielectric conductor (0.1 µS/cm). Commercial brass wire (Osaka Brasscut A500, 0.25 mm diameter) was used as WEDM electrode tool. The wire was fed at a rate of 80 mm/s and wire tension was maintained at 1000 g. The average machining voltage, average working current, and pulse-off time were set at 72 volts, 1.9 amperes, and 2 µs, respectively.

3. Results and Discussion

Density evaluation using theoretical density values for \( \alpha \)-SiAlON, \( \beta \)-SiAlON, and TiN (3.30 g/cm\(^3\), 3.09 g/cm\(^3\), and 5.43 g/cm\(^3\), respectively) revealed that only samples sintered at 1700 °C reached nearly full density. Therefore, for further research, these compositions were selected. The densities of the SPSed composites are listed in Table 2.
Table 2. Densities of SPSed composites.

| Temperature (°C) | Density \(\left(\frac{g}{cm^3}\right)\) | Density \(1\) (%\(\rho\)th) | Density \(\left(\frac{g}{cm^3}\right)\) | Density \(1\) (%\(\rho\)th) | Density \(\left(\frac{g}{cm^3}\right)\) | Density \(1\) (%\(\rho\)th) |
|------------------|-----------------------------------|-----------------------------|-----------------------------------|-----------------------------|-----------------------------------|-----------------------------|
| 1600 °C          | 3.20                              | 97.7                        | 3.39                              | 97.3                        | 3.92                              | 97.1                        |
| 1650 °C          | 3.24                              | 99                          | 3.45                              | 98.9                        | 3.99                              | 98.9                        |
| 1700 °C          | 3.27                              | 99.8                        | 3.48                              | 99.7                        | 4.02                              | 99.7                        |

\(^1\) Theoretical value.

Figure 2 shows the XRD spectra of the \(\alpha-\beta20T\) and TC3030 samples. Any other residual or newly formed phases after sintering, except \(\alpha\)-SiAlON, \(\beta\)-SiAlON, and TiN, were not detected.

The investigation of microstructure by analyzing of SEM images showed bright and gray phases corresponding to TiN particles and \(\alpha\)-\(\beta\) SiAlON ceramic, respectively (Figure 3A). Figure 3B exhibits energy-dispersive X-ray spectroscopy (EDS) maps of polished surface of sintered \(\alpha-\beta20T\) specimen, which confirms the uniform distribution of TiN particles.

It is also important to note that the mean particle size of ceramic matrix with and without TiN was similar, around 2.1 \(\mu\)m (Figure 4). Consequently, the presence of TiN did not affect the grain growth of SiAlON matrix.
The values of Vickers hardness ($H_V$), toughness ($K_{IC}$), Young modulus ($E$), and flexural strength ($\sigma_f$), together with electrical resistivity, are presented in Table 3.

**Table 3. Mechanical properties and electrical resistivity of studied materials.**

|                         | $\alpha$-0T | $\alpha$-10T | $\alpha$-20T | TC3030 |
|-------------------------|-------------|-------------|-------------|--------|
| Vickers hardness ($H_V$) | 14.4 ± 0.3  | 15.3 ± 0.4  | 15.9 ± 0.7  | 13.8 ± 0.3 |
| Toughness $K_{IC}$ (MPa-m^{1/2}) | 3.3 ± 0.3  | 4.1 ± 0.4  | 4.7 ± 0.5  | 3.4 ± 0.3  |
| Flexural strength $\sigma_f$ (MPa) | 375 ± 9    | 343 ± 19   | 327 ± 26   | 360 ± 10   |
| Young modulus $E$ (GPa)  | 345         | 335         | 326         | 343      |
| Electrical resistivity ($\Omega\cdot m$) | $1 \times 10^{11}$ | $10.2 \times 10^1$ | $1.6 \times 10^{-4}$ | $1 \times 10^{11}$ |

Reinforced nanocomposites showed higher hardness than monolithic ceramics due to high HV of TiN (~18 GPa). The $K_{IC}$ also slightly increased with the increase of TiN.
loading. Samples without the additive had a fracture toughness between 3.4 MPa·m$^{1/2}$ and 3.7 MPa·m$^{1/2}$ for TC3030 and α-β0T, while with the addition of TiN, fracture toughness increased from 4.1 MPa·m$^{1/2}$ to 4.7 MPa·m$^{1/2}$ for α-β10T and α-β20T, respectively. Moreover, TiN particles had a higher ($\alpha_{0-1000}$ °C = 9.4 × 10$^{-6}$/°C) [42] coefficient of thermal expansion than SiAlON ($\alpha_{0-1000}$ °C = 3.1 × 10$^{-6}$/°C). This difference caused compressive stresses in the matrix that could also increase fracture toughness due to the crack deflection mechanism (Figure 3C). Results of strength parameters are presented in Table 3. As can be seen, the addition of TiN to the ceramic matrix reduced the strength of α/β-SiAlON-based composites. We assume that this may have been due to the shrinkage difference between TiN particles and ceramic matrix during cooling, which can lead to tensile residual stresses and consequently to changes in strength values.

The fracture surfaces of all studied composites are presented in Figure 5. In all cases, brittle fractures were observed. Some pits on the surface of the samples, especially the α-β0T composite, were found because some of the grains fell. On the other hand, the occurrence of microcracks or pores was not observed.

Electrical resistivity values of the nanocomposites as a function of TiN loading are presented in Table 3. Simultaneously, together with the addition of TiN, the resistivity value of SPS composites dropped to $10.2 \times 10^1$ Ω·m and $1.6 \times 10^{-4}$ Ω·m for α-β10T and α-β20T, respectively. This number is lower than the EDM machinable limit of 1–3 Ω·m. Consequently, this makes the material suitable for nontraditional machining processes [9,11,17].

Figure 6 shows the SEM cross-sectional view after WEDM for α-β20T ceramic. Multi-component ceramic compositions with a high melting point, in which there is a thermal expansion mismatch between constituents, are more susceptible to spalling due to increased stress concentrations at the matrix-particle interphase. Thus, the mechanism of erosion is thermal spalling, in contrast to metals, where the mechanism of material removal is melting and evaporation. It should be pointed out that the width of the heat-affected zone was 120–125 μm, although some cracks were observed in the parent material (Figure 6B). Clearly, the presence of this type of damage in the material directly affects the flexural strength of composites.
decomposition of Si\textsubscript{3}N\textsubscript{4} and TiN induced during the WEDM process, as illustrated by the pores on the WEDMed surface.

As can be seen, various oxidation reactions can occur. For example, Equations (2) and (3) show simultaneously oxidation of both Si\textsubscript{3}N\textsubscript{4} and TiN, while Equations (4) and (5) occur in dielectric liquid. Equation (6) demonstrates decomposition of silicon nitride into nitrogen and silicon, which, in turn, oxidizes further in the deionized water (Equations (7) and (8)). This leads to a large volume of the gases and, consequently, to the formation of pores on the WEDMed surface.

\begin{align*}
\text{Si}_3\text{N}_4 + 3\text{O}_2 & \rightarrow 3\text{SiO}_2 + 2\text{N}_2 \uparrow \quad (2) \\
2\text{TiN} + 2\text{O}_2 & \rightarrow 2\text{TiO}_2 + \text{N}_2 \uparrow \quad (3) \\
\text{Si}_3\text{N}_4 + 6\text{H}_2\text{O} & \rightarrow 3\text{SiO}_2 + 2\text{N}_2 \uparrow + 6\text{H}_2 \uparrow \quad (4) \\
2\text{TiN} + 4\text{H}_2\text{O} & \rightarrow 2\text{TiO}_2 + 2\text{NH}_3 \uparrow + \text{H}_2 \uparrow \quad (5) \\
\text{Si}_3\text{N}_4 & \rightarrow 3\text{Si} + 2\text{N}_2 \uparrow \quad (6)
\end{align*}

Figure 6. SEM cross-sectional view of α-β20T ceramic after wire electrical discharge machining (WEDM) at different magnifications. (A) low magnification; (B) high magnification.

Topography of the machined surface of α-β20T ceramic is presented in Figure 7.

Figure 7. Topography of WEDM surface of α-β20T ceramic at different magnifications.

We assume that such porous and foamy structure was formed due to expansion of nitrogen (N\textsubscript{2}) and hydrogen (H\textsubscript{2}) gases from the surface as a result of oxidation and/or decomposition of Si\textsubscript{3}N\textsubscript{4} and TiN induced during the WEDM process, as illustrated by the following equations:

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2\text{TiN} + 2\text{O}_2 & \rightarrow 2\text{TiO}_2 + \text{N}_2 \uparrow \quad (3) \\
\text{Si}_3\text{N}_4 + 6\text{H}_2\text{O} & \rightarrow 3\text{SiO}_2 + 2\text{N}_2 \uparrow + 6\text{H}_2 \uparrow \quad (4) \\
2\text{TiN} + 4\text{H}_2\text{O} & \rightarrow 2\text{TiO}_2 + 2\text{NH}_3 \uparrow + \text{H}_2 \uparrow \quad (5) \\
\text{Si}_3\text{N}_4 & \rightarrow 3\text{Si} + 2\text{N}_2 \uparrow \quad (6)
\end{align*}
Figure 8 represents depth of the wear scar, depending on the time, observed for α-β20T and TC3030 ceramics.

The wear curve of the commercial sample increased rapidly throughout the first minutes and then became flatter. We believe that this behavior can be explained by the polishing process during testing and removal of surface irregularities or roughness peaks. Meanwhile, the curve of SPS ceramic increased monotonically from the beginning of experiments. Figure 9 shows the 3D surface topography of the wear scar for tested materials. Upon completion of the tribotests, the smallest wear scar depth (∼62 μm) was measured for the α-β20T nanocomposites, which was less than half (∼144 μm) of the TC3030 ceramic.

2Si + O₂ → 2SiO ↑  
Si + O₂ → SiO₂

Figure 9. Three-dimensional wear scar topographies corresponding to (A) α-β20T and (B) TC3030 ceramics after the 900 s tribotest.

The wear volume loss (W) value was determined using Equation (1) and three-dimensional surface topography within the wear scar. The smallest W (2.7 × 10⁻³ mm³/N∙m) was calculated for the α-β20T SPS nanocomposites. Meanwhile, for TC3030 ceramic, the W was 4.4 × 10⁻³ mm³/N∙m. Therefore, under identical conditions, the α-β20T nanocomposites are approximately more than 1.5-times more wear-resistant than the commercial sample.
SEM analysis of the worn surfaces confirmed that the observed changes in tribological behavior were caused by a fundamental change in the wear process. Microstructure plays a vital role on the tribological properties of SiAlONs. However, since the average grain size of the ceramic matrix is similar, greater attention should be paid to the mechanical characteristics. From the point of view of material properties, the higher hardness and toughness, the better wear resistance [37–39].

The worn surface of SPS ceramic nanocomposite was relatively smooth (Figure 10A). Only a small number of microcracks were found.

![Figure 10. Scanning electron microscope photos of the friction surface of (A) α-β20T and (B) TC3030 ceramics. The dotted line indicates the area where the close-up was taken.](image)

The friction surface of TC3030 sample was generally rough (Figure 10B). We suggest that the grain boundary broke first, and after more severe damage, the grains were pulled out. Therefore, the dominant mechanism of material removal in the TC3030 sample was determined as an intragranular fracture (Figure 10B close-up).

Meanwhile, in the previous works, it has been proven that TiN can effectively improve the thermal conductivity of SiAlON ceramic composites [14,37], thereby leading to a lower friction force.

Besides the enhancement in hardness and fracture toughness, TiN particles involved in composites can play an effective role in reducing friction and, consequently, decrease the wear rate of composites.

In general, it can be concluded that SiAlON-20 wt.% TiN ceramic composites fabricated by spark plasma sintering have better properties than commercially available TC3030 ceramic.

4. Conclusions

Near fully dense and homogeneous SiAlON-based nanocomposites reinforced with 10 wt.% and 20 wt.% of TiN were manufactured by combining a colloidal processing route and spark plasma sintering (SPS) technique. The results obtained showed that the addition of a reinforced phase improved the hardness and fracture toughness of ceramic nanocomposites. However, the presence of this phase reduced the strength of ceramic nanocomposites. We assume this was due to the shrinkage difference between TiN particles and ceramic matrix, which can lead to tensile residual stresses and, consequently, to changes in strength values. The nanocomposites with 20 wt.% TiN also showed specific electrical resistivity lower than 1–3 Ω·m, demonstrating their suitability for electrical discharge machining. Besides their enhanced mechanical and electrical performance, SPS-sintered nanocomposites also showed high wear resistance.

Further study is needed for the research and theoretical modelling of not only particle/matrix interfaces and their impact on the reinforcing mechanism of nanocomposites, but also the different ratio of α- and β-SiAlON phases in the matrix. This understanding will enable the creation of ceramic nanocomposites with tailored properties using the most advantageous characteristics of each phase.
In addition, further research is needed to explore the ways and means to improve surface treatment and ceramic integrity. This is necessary in order to ensure the durability of components made of SiAlON.

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