Characterization of TiB2-AlN composites for application as cutting tool

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Densified ceramic TiB2-AlN composites were prepared by spark plasma sintering and samples were subjected to short time machining tests. The maximum relative density and toughness were 96.9% and 16.2 GPa, respectively. Composites with 70 wt% AlN and 70 wt% TiB2 were applied as cutting tools and tested for cutting parameters. Scanning electron microscopy (SEM) allowed the analysis of the microstructure and the wear surfaces after cutting tests. The 70% TiB2 composites revealed unsatisfactory performance while the 70 wt% AlN disclosed satisfactory cutting performance.

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1. Introduction

Ceramic composites based on titanium diboride (TiB2) and aluminum nitride (AlN) have important properties for thermo-mechanical applications. In addition to high thermal conductivity, AlN ceramic is known for its good electrical insulation and low thermal expansion coefficient [1]. Indeed, AlN thermal conductivity values can range from 80 to 260 W m⁻¹ K⁻¹, depending on their chemical composition and microstructure [2]. Besides that, a combination of high melting point and high Young’s modulus makes TiB2 an important material for high performance applications [3]. However, the applications of TiB2 are limited, mainly due to its low fracture toughness, as well as low self-diffusion coefficient, which makes its densification difficult [4].

The combination of TiB2 and AlN provides, among other advantages, an increase in the impact resistance owing to especial toughening mechanisms. These mechanisms are of great importance for improving the toughness of ceramic
materials. Indeed, low fracture toughness, as indicated by $K_{IC}$ measurements, is the main limitation for thermo-mechanical applications of ceramic materials [2]. Among these applications, the use of ceramics as cutting tools is indispensable in the fabrication of high-hardness components, such as those made of tempered steels. This work aims to evaluate two TiB$_2$ - AlN composite consolidated by spark plasma sintering (SPS) and applied as a cutting tool subjected to machining tests.

2. Materials and Methods

Aluminum nitride (AlN) and titanium diboride (TiB$_2$) were used as starting materials for sintered ceramic compound. Both were commercially purchased as powders (Alfa-Aesar/Sigma-Aldrich) with purity greater than 98% and 99.5%, respectively, with 10 µm as average particle size. Two different mixtures were prepared, one with 70 wt% of TiB$_2$ and 30 wt% of AlN and the other containing 30 wt% of TiB$_2$ and 70 wt% AlN. Both mixtures were obtained by manual process using a mortar and a pestle during 30 min at 25°C. After mixing, the mixtures were dried at 60°C for 8 h in a stove.

The densified composites were fabricated by spark plasma sintering (SPS), using “Dr. Sinter Lab Jr.” model SPS 211 LX equipment. All samples were sintered using a constant pressure of 80 MPa under 10$^{-2}$ Torr vacuum. Different sintering temperatures were used (1600°C; 1700°C; and 1800°C) resulting in three samples for each mixture. Samples were heated using 200°C/min as heating rate, and kept at the peak temperature for 10 min. The precursor mixture used in each sintering procedure was charged in a graphite mold with 10.0 mm inner diameter. The composite samples obtained had 10.0 mm diameter, 2.82–3.05 mm in length and mass varying from 0.668 to 0.698 g.

The relative density of the sintered composites was determined using the Archimedes principle technique. After the density tests, the samples were transversally sectioned and observed in a model SSX-550 Shimadzu scanning electron microscope (SEM), for microstructure evaluation and analysis of the exposed surface. As preparation for the SEM analysis, the samples were ultrasonic cleaned for 10 min and platinum metalized at 23°C, under a current of 40 mA for 100 s.

Vickers hardness tests were performed under a load of 294 N in a Pantec tester, model RBSN. The load was slowly applied and held for 10 s, and then, removed as per ASTM E92-16 [5].

Samples that reached the highest relative density and hardness were subjected to short machining tests, based on the ISO 3685 standard recommendation [6], which is intended to unify experiments for cutting tools. Machining tests were performed in lathe-CNC Romi, model Centur 35D and CLP-Siemens 802D using quenched and tempered steel SAE 4140 with hardness of 52 Rc.

The tool lifespan curves were determined in association with parameters of depth (0.1 mm) and feed rate (0.2 mm) during the machining process, which uses 100, 200 and 300 m/min as cutting speeds. After machining tests, each wear surface of cutting tools was qualitative and semi-qualitative analyzed using SEM and energy-dispersive X-ray spectroscopy (EDS).

This study considered maximum flank wear (VB$_\text{max}$) equal to 0.30 mm or catastrophic failure of the tool as criteria of the end of the lifespan for sintered samples used as cutting tools [6].

3. Results and Discussion

Table 1 shows the relative density and hardness, as a function of temperature, for the sintered samples. Samples with 70 wt%

<table>
<thead>
<tr>
<th>Sintering temperature (°C)</th>
<th>Relative density (%)</th>
<th>Hardness (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1600</td>
<td>85.0 ± 2.15</td>
<td>10.1 ± 0.4</td>
</tr>
<tr>
<td>1700</td>
<td>93.2 ± 1.22</td>
<td>13.8 ± 0.3</td>
</tr>
<tr>
<td>1800</td>
<td>96.9 ± 0.65</td>
<td>15.1 ± 0.3</td>
</tr>
<tr>
<td>1600</td>
<td>82.4 ± 1.30</td>
<td>8.6 ± 0.6</td>
</tr>
<tr>
<td>1700</td>
<td>87.5 ± 1.33</td>
<td>16.0 ± 0.5</td>
</tr>
<tr>
<td>1800</td>
<td>92.0 ± 1.20</td>
<td>16.2 ± 0.9</td>
</tr>
</tbody>
</table>

Fig. 1 – SEM micrograph of 70 wt% of AlN samples sintered in different temperatures: (a) 1600°C; (b) 1700°C; and (c) 1800°C.
AlN achieved higher densification at all temperatures when compared to samples with 70 wt% TiB₂. However, composites with 70 wt% TiB₂ reached higher hardness. This behavior is justified by the low sinterability and high hardness of monolithic TiB₂ [4]. Both sintered composites reached hardness greater than the monolithic AlN (10.6 MPa), however, they remained below that of the TiB₂ monolithic ceramic (25 MPa) [7].

Fig. 1 presents the microstructure of the sintered samples with 70 wt% of AlN. It is possible to observe the elongated morphology of TiB₂. Elongated grains of TiB₂ and the difference in thermal expansion coefficient between TiB₂ and AlN provide both deflection and propagation of cracks through contact interface. These are the most common toughening mechanisms in ceramic composites that are responsible for the composites fracture toughness (Kᵢc) [8]. By contrast, the 70 wt% TiB₂ composite has a mostly brittle microstructure with corresponding lower fracture toughness.

In Fig. 1 it is also possible to observe porosity reduction when sintering temperature was increased, confirming the results presented in Table 1. The presence of sharp-edges pores, mainly seen at the sintering temperature of 1600 °C, demonstrates the difficulty that AlN finds to wet TiB₂ at the lower temperatures investigated.

After microstructural analysis together with evaluation of the fracture toughness and densification, samples sintered at 1800 °C were chosen for short time machining tests [6]. Tests were performed in severe conditions of machining to accelerate the wear of cutting tool.

![Fig. 2 - Cutting speed vs. tool life of the 70% AlN sintered at 1800 °C.](image)

![Fig. 3 - Machined surface using 70 wt% TiB₂ composite as cutting tool.](image)

![Fig. 4 - SEM (a) and EDS (b) of a 70 wt% AlN wear crater (Vc = 300 m/min).](image)
Lifespan of tools with 70 wt% of AlN are presented in Fig. 2. As expected, lifespan decreased when a greater cutting speed was used.

Using cutting speed of 100 m/min the samples reached average lifespan of 4.17 min. The samples tested at speed of 200 m/min reached end of lifespan after 3.8 min. However, the samples subjected to machining tests with a speed of 300 m/min achieved end of lifespan after about 2.0 min.

Tools with 70 wt% of AlN showed satisfactory results during machining tests when compared to the results obtained by Ezugwu et al. [9] who performed tests of machining using tools based on CBN (cubic boron nitride) and found a maximum lifespan of approximately 4.0 min, using cutting speed of 150 m/min and liquid flow for cooling.

The composites with 70 wt% of TiB₂ did not present satisfactory behavior in machining tests. For all the speeds used, the tool presented premature wear. The excessive heating during machining with this tool caused plastic flow in the work piece (Fig. 3), and, consequently, a very irregular machined surface. Low performance of tools with 70 wt% of TiB₂ can be explained by low thermal conductivity, which was responsible for excessive heating.

All composites used as cutting tool in the three speeds presented crater and flank wear (Fig 4a). The flank wear is observed as irregular machining gaps between the cutting tool and the working piece. This wear is caused by loss of tool relief angle, which increases friction in the contact with the working piece. Regarding the wear of the crater, which occurs in the rake face, it is one of the reasons for both the significant increase in the cutting forces and difficult in removing metal chips.

Fig. 4b presents EDS chemical microanalysis performed in the region of the composite wear after the process of machining. The EDS results indicate the predominant presence of aluminum (Al) and titanium (Ti), which are the basic elements of this composite. In addition, the presence of carbon (C) was observed, which could be justified by the carbon sheets used in SPS. EDS was fundamental to check the chemical stability of this composite in thermo-mechanical applications. The absence of external elements in the cutting tool demonstrated that there were no significant chemical reaction between the composite and the working piece. The chemical stability is essential to guarantee the maintenance of composite mechanical properties during machining.

4. Conclusions

- Densification and hardness of TiB₂–AlN composites sintered by spark plasma sintering (SPS) were evaluated, as well as their performance as cutting tools in standard machining tests. Both specimens, TiB₂–30wt%AlN and AlN–30wt%TiB₂, reached satisfactory densification and hardness values.
- Composites with 70 wt% of TiB₂ presented unsatisfactory performance when used as cutting tools.
- Composites with 70 wt% of AlN presented a microstructure of confined elongated TiB₂ grains, which contribute to deflect cracks and improve the toughness of the composite. This is responsible for its better performance as cutting tool and satisfactory wear resistance.

Conflicts of interest

The authors declare no conflicts of interest.

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