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Microstructure, Mechanical Characteristics, and Wear Performance of Spark Plasma Sintered TiB$_2$–Si$_3$N$_4$ as Affected by B$_4$N Doping

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Abstract: This study aims to analyze the effect of boron nitride (B$_4$N) additive (3–6%) on the densification, microstructure, mechanical properties, and wear performance of TiB$_2$–15%Si$_3$N$_4$ and TiB$_2$–30%Si$_3$N$_4$ sintered composites. When the B$_4$N (3%) was added to the TiB$_2$–30%Si$_3$N$_4$ composite, the density increased to 99.5%, hardness increased to 25.2 MPa, and the fracture toughness increased to 4.62 MPa m$^{1/2}$. Microstructural analysis shows that in situ phases such as TiB$_2$ help to improve the relative mechanical characteristics. However, raising the B$_4$N additive to 6% in the above-sintered composite reduces the composites’ relative density and hardness. The tested sintered composites demonstrated that their superior wear resistance can be attributed to their increased density and hardness.

Keywords: spark plasma sintering; mechanical characteristics; wear; microstructure; densification

1. Introduction

Due to its high melting point, ultra-high temperature ceramics (UHTCs) are the best materials to use in hard environments [1–7]. TiB$_2$ is one of the UHTC that has received a lot of attention due to its impressive list of properties, which includes a higher hardness (33 MPa), higher melting point (3225 °C), abrasive resistance, less heat expansion coefficient, and higher Young’s modulus (530 MPa) [5]. TiB$_2$’s unique properties have made it useful in a variety of contexts, such as a component in armor, cutting tools, and as components that are resistant to corrosion and wear [8]. The covalent solid bonding and melting point of TiB$_2$, the surface oxide layer of B$_2$O$_3$ and TiO$_2$ that serves as impurities, and the weak self-diffusing coefficient of undoped TiB$_2$ all make its consolidation difficult. This indicates that TiB$_2$ consolidation requires severe densification conditions, including a long dwelling time, a high sintering temperature, and high pressure [9]. The mechanical characteristics of TiB$_2$ degrade rapidly due to the material’s property for developing aberrant microcracking, grain development due to the material’s crystallinity structure, and high heat expansion.
anisotropy. Therefore, it is essential to reduce the granular size of titanium diboride to improve its mechanical qualities.

AlN, Si$_3$N$_4$, TiN, TiC, etc., have all been utilized as reinforcements to improve TiB$_2$’s characteristics. With its great thermal strength, outstanding chemical stability, and remarkable oxidation resistance, silicon nitride (Si$_3$N$_4$) stands out among these numerous reinforcements. Sinterability, mechanical characteristics, and high-temperature oxidation resistance of the composites are improved by including Si$_3$N$_4$ in the TiB$_2$ matrix [10,11].

Since then, the number of studies conducted on TiB$_2$–Si$_3$N$_4$ composites has grown alongside its increased production. They show superior properties compared with other materials [12–16]. Experiments have shown that the addition of Si$_3$N$_4$ to TiB$_2$ starting powder can change the sintering mechanism, resulting in the development of stubborn titanium diboride and liquid SiO$_2$ phases by interacting with the TiO$_2$ impurity. The liquid phase sintering (LPS) method is largely used to enhance the density of TiB$_2$-based ceramic materials [17–19].

Hot-isostatic pressing, hot pressing, pressure-less sintering, and spark plasma sintering (SPS) are some of the methods used to produce TiB$_2$ materials [4,20–22]. Pressure-less sintering has emerged as the most effective and cost-efficient method for creating objects with intricate geometries and massive dimensions. Although the method relies on high sintering temperatures to achieve consolidation of the TiB$_2$–Si$_3$N$_4$ ceramic composite, this frequently results in uncontrolled grain formation [23,24]. The most popular process for producing dense UHTC-based materials is hot-pressing, which is also an effective sintering process. This method, while effective, is quite expensive and limits the final product’s size and shape [25]. SPS is a new consolidation method which is favored over the traditional sintering method owing to its shorter processing time and lesser sintering temperature. Here, a pulsed direct current is concurrently used to heat both the compressed powder and the die. The rapid sintering rate of this technique makes it possible to manipulate the characteristics and final microstructure of the sintered component [26–28]. A literature review has demonstrated that the usage of the SPS approach to synthesize TiB$_2$–AlN, TiB$_2$–B$_4$N, and TiB$_2$–Si$_3$N$_4$ composites resulted in improved mechanical properties and microstructure [29,30].

Diverse reinforcement additives have been studied in numerous investigations to determine their impact on the density and process of diboride ceramics [31,32]. Densification of 98% was achieved when AlN was used as sintering assistance in the hot press link of TiB$_2$ ceramic at 1800 °C and 30 MPa for one hour. This is attributed to the realization of a polished microstructure by removing grain growth. B$_4$N was used as a sintering aid on TiB$_2$ particles to remove surface oxide impurities (such as TiO$_2$), resulting in higher relative density to the formation of TiN, SiO$_2$, and BN [33]. Samples of TiB$_2$ reinforced with B$_4$N were tested for their consolidation performance and microstructure [34]. To lower the porosity of the TiB$_2$ ceramic matrix from 3% in the undoped state to 0.3%, an investigation was carried out. As a finding, adding B$_4$N at a weight percentage of 5% produced the highest density. The development of titanium nitride and boron nitride in situ matrix phases was studied by microstructural analysis. Using experimental methods, it was established that including Si$_3$N$_4$ into a B$_4$C matrix composite decreased wear and friction. Interfacial bonding in composites between additives and the matrix plays a key role in wear rates.

While some studies have investigated the effects of adding B$_4$N as a sintering additive on the densification and mechanical properties of TiB$_2$–Si$_3$N$_4$, no one has investigated how the B$_4$N sintering additive affects and controls the TiB$_2$–Si$_3$N$_4$ composites’ microstructure and density, which in turn control the composites’ desired mechanical properties for obtaining improved wear resistance.

2. Experimentation

The following materials with a purity level of 99.5% were obtained: titanium diboride (TiB$_2$) with a mean particle size of 5.2 µm; and boron nitride (B$_4$N) and silicon nitride (Si$_3$N$_4$)
with a mean particle size of 44 μm. Characteristics of the powder in its as-received form are listed in Table 1. By using a turbulent mixer, the powders were mixed at 49 rpm for 8 h.

Table 1. Characteristics of the powder elements in their as-received states.

| Resources  | Density | Particle Size (μm) | Purity (%) | Particle Shape |
|------------|---------|--------------------|------------|----------------|
|            |         | D10    | D50    | D90    |                |
| TiB₂       | 4.53    | 2.2    | 5.2    | 6.8    | 99.5           | Regular          |
| Si₃N₄      | 3.21    | 28     | 44     | 52     | 99.5           | Irregular        |
| B₄N        | 3.17    | 32     | 44     | 50     | 99.5           | Irregular        |

The microstructure of the admixed powders was examined with the help of a 7600F JEOL SEM (Merlin Compact, ZEISS, Oberkochen, Germany) equipped with EDX. The phase was analyzed via X-ray diffraction (XRD) using CuKα radiation at 30 kV and 40 mA. The powder mixture of varying compositions was poured between the walls of the graphite die, which had an internal radius of 10 mm. After the consolidation process, samples were removed from the die arrangement with the help of a foil. The sintering process was performed at a vacuum pressure of 10⁻² Torr. The 1850 °C sintering temperature was obtained using an AC with 50 Hz and at a heating rate of 150 °C/min. For the sintering process, 50 MPa of pressure and 10 min of dwell time were used. The fabricated specimens were cooled to room temperature before being collected from the SPS machine. Following collection, the samples were sandblasted to remove any traces of foil. TiB₂-15 Si₃N₄-(3-6) B₄N and TiB₂-30 Si₃N₄-(3-6) B₄N are the fabricated specimens using the above process.

The density of the sintered sample was measured using a densitometer, a device based on Archimedes’ principle. The elemental composition and microstructure of the polished surface were analyzed by using SEM coupled with EDX. The phases of the specimen were analyzed by using the XRD technique. Vickers microhardness testing was carried out on the specimens using a 2 kg force and a 15 s dwell period. Each sample’s microhardness was determined by taking an average of 15 measurements (HV). The fracture toughness of the samples was calculated by using the crack length which is generated by the indenter (Figure 1) under a force of 2 kg. The crack length was determined under an optical microscope and then the fracture toughness was evaluated by using Equation (1), derived by refs [35–37].

\[ K_{ic} = 0.203HV \frac{a^{1/2}}{c/a}^{-3/2} \]  

(1)

where,

- \( c \) crack span of the indentation.
- \( a \) diagonal of the indenter.
Fifteen indentation tests were performed per specimen, and the average was calculated. Tribology analysis was performed utilizing a tribometer (RTec make) with reciprocating wear drive due to the significance of loads in defining the wear properties of the materials under investigation. An appropriate linear slider, attached to a rod, was used to hold the sample and initiate the process. An external motor was used to introduce motion into the operational platform. The impact of a 6 mm stainless steel ball with different weights (6 N, 12N, 18 N, 24 N) was measured over 900 s at a linear speed of 0.07 cm/s. A square specimen measuring 10 mm² subjected to grinding and polishing before the investigation was subjected to a wear test through SEM.

3. Results and Discussions
3.1. Characteristics of As-Received and Sintered Additives Powders

Figures 2 and 3 show SEM images and XRD patterns of the titanium diboride and its reinforcements (Si₃N₄ and B₄N), respectively. Additionally, the EDX method was used to analyze and visualize the elemental composition (Figure 4). Visual inspection with XRD pattern and SEM images of the mixed powders (TiB₂-15Si₃N₄/30Si₃N₄+3/6 B₄N) are shown in Figures 5 and 6, respectively.

![Figure 2](image-url) SEM images of the powders (a) TiB₂, (b) Si₃N₄, and (c) B₄N.

![Figure 3](image-url) XRD of the as-received powder.
Figure 4. EDX analysis of (a) TiB$_2$, (b) B$_4$N, and (c) Si$_3$N$_4$.

Figure 5. Relative intensity of mixed powders as given by XRD: (a) TiB$_2$-15Si$_3$N$_4$-3B$_4$N, (b) TiB$_2$-15Si$_3$N$_4$-6B$_4$N, (c) TiB$_2$-30Si$_3$N$_4$-3B$_4$N, and (d) TiB$_2$-30Si$_3$N$_4$-6B$_4$N.
which prevented pores from forming on the sintered composites’ surface, had made a considerable impact on the composite’s density. The relative density of the as-sintered specimens, as shown in Figure 7, reveals the densification effect of B₄N percentage (3% to 6%) on the composite specimen’s density. For each specimen, five readings were taken, and the average is presented in Figure 7. The relative density was 98.2% in a TiB₂-15Si₃N₄-3B₄N composite, but only 97% when B₄N is raised to 6%. TiB₂-30Si₃N₄-3B₄N and TiB₂-30Si₃N₄-6B₄N both followed the same densification pattern as the composites above, having relative densities of 99.5% and 98.8%, respectively. The density of TiB₂-30Si₃N₄-6B₄N is slightly lower than that of TiB₂-30Si₃N₄-3B₄N, indicating that B₄N has a negligible effect on the composite’s density. The microstructural evolution also revealed that the sintering factors (1850 °C/50 MPa/10 min), which prevented pores from forming on the sintered composites’ surface, had made a considerable impact on the consolidation of the composites.

Figure 7. Sintered composites’ relative densities.

3.2. Densification

The relative density of the as-sintered specimens, as shown in Figure 7, reveals the influence of B₄N percentage (3% to 6%) on the composite specimen’s density. For each specimen, five readings were taken, and the average is presented in Figure 7. The relative density was 98.2% in a TiB₂-15Si₃N₄-3B₄N composite, but only 97% when B₄N is raised to 6%. TiB₂-30Si₃N₄-3B₄N and TiB₂-30Si₃N₄-6B₄N both followed the same densification pattern as the composites above, having relative densities of 99.5% and 98.8%, respectively. The density of TiB₂-30Si₃N₄-6B₄N is slightly lower than that of TiB₂-30Si₃N₄-3B₄N, indicating that B₄N has a negligible effect on the composite’s density. The microstructural evolution also revealed that the sintering factors (1850 °C/50 MPa/10 min), which prevented pores from forming on the sintered composites’ surface, had made a considerable impact on the consolidation of the composites.
3.3. Microstructural Analysis of Sintered Composites

The adhesion between the as-sintered composite grains (Figure 8) indicates the effect of sintering between the reinforcement and the matrix. However, composites with a higher amount of additive show a wider variety of in situ phases than those with a lower amount of additive. Similarly, researchers investigated ZrB$_2$’s consolidation with Si$_3$N$_4$ as reinforcement and found that utilizing lower sintering temperature and Si$_3$N$_4$ were the primary determinants in reducing grain growth [38–41].

![Figure 8. SEM images of the sintered composites: (a) TiB$_2$-15Si$_3$N$_4$-3B$_4$N, (b) TiB$_2$-15Si$_3$N$_4$-6B$_4$N, (c) TiB$_2$-30Si$_3$N$_4$-3B$_4$N, and (d) TiB$_2$-30Si$_3$N$_4$-6B$_4$N.](image-url)

The XRD pattern (Figure 9) shows the presence of TiC and SiO$_2$ layers on the sintered composite specimens. Similar findings were reported by Hosseini Vajargah et al. [42]. They noted the presence of TiO$_2$ and B$_2$O$_3$ layers on the TiB$_2$ powder in its as-received form. Because of the chemical processes’ influence on the sintering behavior, the oxygen percentage reduces. The densification enhancement of the sintered sample can be initiated by the mechanism of a liquid phase, and the achieved progress of SiO$_2$ through the sintering operation can commence this process. Last but not least, the interaction of Si$_3$N$_4$ with the oxide layer (B$_2$O$_3$) triggered the formation of B$_4$C and various gaseous byproduct phases, and the thermodynamic fulfillment of these materials occurred at 1760 °C. At room temperature, the boiling and melting points of B$_2$O$_3$ are 1870 °C and 460 °C, while at temperatures lower than 1600 °C, B$_2$O$_3$ evaporates.

The EDX analysis (Figure 10) of the sintered composite specimen, TiB$_2$-30Si$_3$N$_4$-6B$_4$N, reveals that the ceramic matrix and the Si$_3$N$_4$ or B$_4$N reinforcement are excellent grey phases. Figure 8 shows what could be either pores or pulled-out grains caused by the polishing process. Because of holes and surface pits, lower densification results in the composites. The above is in accordance with previous findings [43,44], which claimed the reduction of densification is due to grain pull-out of extracts or matrix while specimen refining. Consolidation efforts for boride materials, including ZrB$_2$, HfB$_2$, and TiB$_2$, have revealed the occurrence of oxide impurities on the powder surface. Densification and grain expansion are encouraged due to metallic impurities that are found in the precursor powders [45,46]. The densification of investigated composite specimens has been improved.
because of sintering additives which removed oxide impurities from the sintered surface of TiB$_2$ and induced some secondary phases, primarily TiC.

![XRD pattern](image)

**Figure 9.** Relative intensity of sintered composites as measured by XRD: (a) TiB$_2$-15Si$_3$N$_4$-3B$_4$N, (b) TiB$_2$-15 Si$_3$N$_4$-6B$_4$N, (c) TiB$_2$-30Si$_3$N$_4$-3B$_4$N, and (d) TiB$_2$-30 Si$_3$N$_4$-6B$_4$N.

![EDX](image)

**Figure 10.** EDX of sintered composite TiB$_2$-30Si$_3$N$_4$-6B$_4$N.

### 3.4. Mechanical Characteristics

A comparison of the sintered composites’ hardness is shown in Figure 11. The hardness values follow a similar pattern as the obtained relative density values. The hardness of TiB$_2$–15Si$_3$N$_4$ composites is 19.08 MPa when doped with 3% B$_4$N; when doped with 6% B$_4$N, the value drops by 1.58%. The hardness of TiB$_2$–30Si$_3$N$_4$ composite doped with 3% B$_4$N is 10% more than that of TiB$_2$–30Si$_3$N$_4$ composite doped with 6% B$_4$N. Data in Figure 11 show the highest fracture toughness in the composite with high reinforcing compositions. It is concluded that the development of the second phase is responsible for this improvement.
Figure 11. Mechanical properties of sintered composites: (a) hardness and (b) fracture toughness.

Improvement in mechanical features is due to the enhancement in microstructural qualities such as porosity, second phases, and grain size. Densification of ceramics is also a common factor in determining its fracture toughness and hardness. Increased densification and lesser penetrability in the composites leads to improved micro-hardness and fracture toughness. The relative densities and the grain sizes of ceramics contribute to their improved fracture toughness. Larger grain-size particles decrease fracture toughness by diluting other mechanical characteristics.

As grain size decreases, crack propagation and fracture energy consumption occur at grain borders and bends. Therefore, lessening the grain size improves the crack robustness of composites. Properties enhancement was achieved in ZrB$_2$–Si$_3$N$_4$-based composites by employing an effective sintering temperature of 1700 °C with an 8-min dwell period [47]. More importantly, raising the sintering parameters reduces the composite’s fracture toughness. Moreover, porosity is decreased by applying longer dwell times and higher temperatures [47].

Therefore, grain size and open pores essentially control the improvement in mechanical characteristics. Further, one more component affecting the toughening mechanism in sintered composites is crack deflection at the Si$_3$N$_4$ particle and TiB$_2$/Si$_3$N$_4$/B$_4$N contacts. Compressive stresses in the Si$_3$N$_4$ particulate phases cause fracture deflection, which in turn causes large stress to be applied at the crack’s tip, reducing the active force for crack propagation and increasing the toughness of the sample. Other mechanisms, such as fracture branching and crack bridging, contribute to the toughening impact of the composites (Figure 12). Microcracking is a toughening impact in TiB$_2$–Si$_3$N$_4$ samples [48,49]. Because of created interfacial stress at the grain boundaries of TiB$_2$/Si$_3$N$_4$, microcracking
occurs that requires more fracture energy to complete. The incompatibility among the heat expansion coefficients of TiB$_2$/Si$_3$N$_4$/B$_4$N grains induces interfacial stress.

![Crack deflection](image)

**Figure 12.** Scanning electron micrographs of indentation cracks in TiB$_2$-30Si$_3$N$_4$-6B$_4$N.

The inclusion of B$_4$N and Si$_3$N$_4$ changes the fracture mode from intergranular to mostly transgranular (as seen from the broken surface in Figure 13). When the toughening process is integrated into the fracturing mechanism, the material has sufficient energy to reduce the propagation of cracks.

![Fractured surface](image)

**Figure 13.** SEM image showing fractured surface of: (a) TiB$_2$-15Si$_3$N$_4$-3B$_4$N, (b) TiB$_2$-15Si$_3$N$_4$-6B$_4$N, (c) TiB$_2$-30Si$_3$N$_4$-3B$_4$N, and (d) TiB$_2$-30Si$_3$N$_4$-6B$_4$N.
3.5. Coefficient of Friction of TiB$_2$–Si$_3$N$_4$ Doped with B$_4$N as a Load Function

Figure 14 displays the results of tests done on sintered samples without the use of any lubricant. It shows that, as the applied load increases from 6 N to 24 N, the mean coefficient of friction of these materials is assorted from 0.1 to 0.441. In some composites, the coefficient of friction (COF) reduces as the applied force grows stronger. TiB$_2$–15Si$_3$N$_4$ doped with 6B$_4$N has a COF of 0.439 at 6N load, falling to 0.16, 0.125, 0.19 at 12 N, 18 N, and 24 N loads, correspondingly. From Figure 14, it is evident that the composite specimen with less COF has a greater proportion of Si$_3$N$_4$ combined with a smaller proportion of B$_4$N. That is, TiB$_2$-30Si$_3$N$_4$-3B$_4$N composite has the lowest COF of 0.1 at 18 N applied load.

![Figure 14. Average coefficient of friction of sintered composites.](image)

Applying a load causes the matrix and sintering additives to come into contact, forming a thin film layer that decreases friction. The formed film acts as a barrier and prevents the load from slipping past the sample’s contact point. Results from this research show that high-hardness composites also have less COF.

3.6. Effect of the Load on Wear Rates of TiB$_2$–Si$_3$N$_4$ Doped with B$_4$N

The sintered sample’s wear rate, as shown in Figure 15, reveals that the wear rate increases with increasing load, except for TiB$_2$–15Si$_3$N$_4$–6B$_4$N, TiB$_2$–30Si$_3$N$_4$–3B$_4$N, and TiB$_2$–30Si$_3$N$_4$–6B$_4$N composites, where the wear rates fell most at a load of 12 N. When subjected to a load of 12N, the composite TiB$_2$–30Si$_3$N$_4$–3B$_4$N has the lowest wear rate at 2.9045 $\times$ 10$^{-6}$ mm$^3$/Nm. The composite specimen with higher wear resistance has higher relative density and hardness. Hence, the aforesaid characteristics account for the composite’s improved wear resistance.

![Figure 15. Wear rates of sintered samples.](image)
Thus, tribological film formation under increasing stress controls the wear rate. That is, the interface bond between the sintering reinforcements and the matrix affects wear rates in the composites. In numerous studies of self-lubricating ceramics’ wear and friction performances, the significance of tribological film on contact surfaces has been emphasized.

Several studies [50,51] have investigated the impact of tribological film and its characterization on wear behavior. By forming the film, wear rates and the coefficient of friction can be significantly reduced in the composites. It has been shown in prior research that when B$_4$N and Si$_3$N$_4$ (a covalently bonded form of ceramic) rub against one another, a film, including hydrated SiO$_2$, may be generated on the abrasive surface due to chemical consequences. A hydrated silicon dioxide film is formed on the surface of the worn B$_4$N when it moves along in the water. Some researchers have found that connecting ceramics initiates film relocations on its wearing surfaces with the same ionic bond (such as TiB$_2$, Al$_2$O$_3$, and ZrO$_2$). Thus, the tribological film acts as a friction-reducing force which leads to a lower coefficient of friction. Sintering additives (Si$_3$N$_4$ and B$_4$N) provide load-bearing capabilities that reduce breakage and plastic distortion in the composite matrix, and therefore they are the driving force behind increasing the wear resistance of doped Titanium diboride when compared to the undoped one.

### 3.7. Morphological Analysis of Worn-Out Surfaces

The wear morphology of synthesized materials (TiB$_2$-15/30Si$_3$N$_4$-3/6B$_4$N) is shown in Figures 16–19. The microstructure of wear tracks shows debris, scratches, and grooves. As seen in Figures 16 and 17, the composites made up of 15% Si$_3$N$_4$ and 3/6% of B$_4$N exhibited significant delamination when the load raised from 18 N to 24 N. As seen in Figures 18 and 19, the composites made up of 30% Si$_3$N$_4$ and 3/6% of B$_4$N exhibited little grooves and scratches that show their excellent wear resistance. The composites’ wear resistance is enhanced by the presence of phases, especially TiC, that prevent the composite’s surface from wearing out. Similarly, previous research [52] found that reinforcing the B$_4$C ceramic matrix with ZrO$_2$, led to the in situ formation of ZrB$_2$, which greatly improved the tribological performance of the composite material.

![Figure 16. Micrographs showing worn-out surfaces of TiB$_2$-15Si$_3$N$_4$-3B$_4$N at: (a) 6 N, (b) 12 N, (c) 18 N, and (d) 24 N.](image-url)
Figure 16. Micrographs showing worn-out surfaces of TiB$_2$-15Si$_3$N$_4$-3B$_4$N at: (a) 6 N, (b) 12 N, (c) 18 N, and (d) 24 N.

Figure 17. Micrographs showing worn-out surfaces of TiB$_2$-15Si$_3$N$_4$-6B$_4$N at: (a) 6 N, (b) 12 N, (c) 18 N, and (d) 24 N.

Figure 18. Micrographs showing worn-out surfaces of TiB$_2$-30Si$_3$N$_4$-3B$_4$N at: (a) 6 N, (b) 12 N, (c) 18 N, and (d) 24 N.
4. Wear Performance of TiB$_2$-Si$_3$N$_4$-B$_4$N Composites and Their Relation with Microstructure Characterization, Densification, and Mechanical Characteristics

The microstructure of TiB$_2$-Si$_3$N$_4$-B$_4$N hybrid composites shows that the additives have distributed uniformly, which improved the composite’s wear resistance. When there is a disparity between the strength and the material, either due to insufficient consolidation of the resulting composite or unequal dispersion of the additives in the interstices of the matrix, achieving the desired enhanced features will be more difficult. In advanced ceramic composites, sintering additives subsidizes the overall characteristics of sintered ceramic composites; as a result, the amount and appropriate choice of sintering reinforcement are critical to obtain enhanced qualities of the ceramic composites. A material’s flexural strength, elastic modulus, fracture toughness, and hardness contribute to its wear resistance under sliding conditions.

The wear performance of composite material is affected by the number of reinforcements used in its manufacture, which also affects the fracture toughness, crack deflection, and crack bridging. The tribology of the composite is affected by its hardness, which is its resistance to plastic deformation when subjected to indenter or mechanical scratch. Various properties such as elasticity, ductility, robustness, stiffness, viscoelasticity and strain change the material’s hardness. Any flaw in the abovesaid characteristics could reduce the material’s hardness. For a better wear-resistant material, it is crucial to have a good microstructure and excellent mechanical characteristics.

5. Conclusions

TiB$_2$-15/30 Si$_3$N$_4$-3/6 B$_4$N composites were fabricated by the spark plasma sintering method at 1850°C and under 50 MPa for 10 min. The findings from the aforesaid composites are as follows:

1. TiB$_2$ composites with greater Si$_3$N$_4$ reinforcement exhibit in situ phases leading to higher densification (98.8–99.5%). Reduction in surface oxide impurities and porosity is the cause for grain development and thereby enhancement in densification.
2. A decrease in density and hardness occurs when B₄N content increases in the TiB₂–Si₃N₄ composite.
3. Hardness values for the composites followed the same pattern as that of densification and were inversely proportional to fracture toughness values. Microstructural analysis revealed that both the diffusion of lattice atoms and the emission of an in situ phase played important role in improving the composites’ characteristics.
4. Composites with a superior microstructure and improved mechanical characteristics have an improved wear behavior. The higher densification (99.5%) and modest hardness (25.2 MPa) of the TiB₂–30Si₃N₄–3B₄N composite account for its excellent wear resistance.
5. This study established the significance of sintering additives and their suitable proportions in improving the mechanical characteristics, and, by extension, the wear behavior of the resultant composite.

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