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Effect of graphite addition on the tribological properties of pure titanium carbonitride prepared by spark plasma sintering

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Abstract. Titanium carbonitride (TiCN) based cermet has received extensive attention as important components substantially utilized in cutting tools, milling operations and in sliding bearings. Recently, conventional WC-Co based hard alloys are being replaced with TiCN based cermets accompanied with the trend of high speed machining. These materials are considered potential candidate for a variety of tribological applications. In this study, the effects of graphite additions on titanium carbonitride (TiCN) based cermet were investigated. This involved consolidation of TiC_{0.7}TiN_{0.3} composition of pure TiCN based cermet and/or with 0.5, 1.0 and 1.5 wt % graphite using spark plasma sintering (SPS). The comparative studies on the tribological behaviours of the TiCN based cermets with graphite additions were performed using ball on disc set up at ambient temperature. Results show that the presence of different composition of graphite influences the microstructures of TiCN. In addition, a change in wear response of the sintered compacts was observed.

1. Introduction

Metal cutting industries persistently require materials with high temperature and good wear properties for high performance at extreme temperatures, in order to maximize productivity and reliability of the equipment. Titanium carbonitride (TiCN) based cermet as a ceramic-metal composite, gained enormous attention owing to its unique combination of properties such as high hardness, chemical inertness, low coefficient of friction, high temperature resistance and better wear resistance [1-10]. Compared to the performance of conventional cemented carbide (WC-Co) cutting tools, TiCN based cermet grant improved surface finishing, excellent chip/ tolerance control and allow geometrical accuracy of the work piece. Furthermore, TiCN based cermet, compares favourably to WC-Co in terms of performance and price. All these phenomenal mechanical properties are attributes of the hard phase composed within the cermet system [5-7].

Lately, there have been numerous attempts by various researchers to develop TiCN based cermet in accordance to enhance properties of the cermet by adding metallic binders, iron based alloys and



distinct carbides using conventional sintering methods [11-14]. However, binders draw on some challenges of poor wettability, health issues, and costs, they also affect mechanical properties (hardness and toughness) of the material. This will compromise the quality of the material and it is unsought, since hardness and toughness are the primal properties required for cutting tools [1,3,13,15,16]. Drawbacks of involving binders in the cermet system encourage the significance to develop binder-less TiCN based cermet. Although, TiCN based cermet is widely recognized as a wear resistant material in some applications e.g. cutting tools, sliding bearings, compaction or forming dies, it is crucial to further investigate and enhance their tribological properties in order to widen their applications. Therefore, additives and solid lubricants can be used to improve the tribological properties of the cermet to also increase the tool life.

Solid lubricants are promising material for high performance machinery and mechanical systems. Graphite which is known as a normal solid lubricant possesses the best effect of lubrication and can be used to develop materials with self-lubricating properties [26-28]. Verma and Kumar [23,25] studied the tribological behaviour of TiCN based cermet against cemented carbide but thus far there is limited work reported on binder-less/pure TiCN based cermet. The present study is aimed at investigating the effect of graphite on the tribological properties of pure TiCN based cermet prepared by SPS. TiC_{0.7}TiN_{0.3} composition of TiCN based cermet was consolidated by SPS and room temperature tribological behaviour of sintered samples was studied using ball on disc tribometer. To understand the wear mechanism and to demonstrate the effect of graphite, wear tracks and wear mechanisms were studied using SEM.

2. Materials and method

2.1. Characterization of TiCN – based cermet

Titanium carbonitride (TiCN) based cermet and graphite powders were used as the starting powders. TiC_{0.7}TiN_{0.3} composition of TiCN powder was used as a matrix and graphite powder as reinforcement with 1.0 –1.3 and 1–2 μm particle sizes respectively. Mixing of the powders (TiCN and with 0.5; 1.0; 1.5 % by weight of graphite) was done using T2F Tubular mixer with the addition of tungsten carbide balls, at a ball to powder ratio of 10:1 for 4 h at a speed of 101 rpm. Qualitative phase and morphology analysis was done on the starting powders using SEM. The mixed powders were then consolidated to discs of 40 mm in diameter using spark plasma sintering system model HHPD25, FCT Germany. Archimedes principle was used to measure the densification of the sintered compacts using a weighing balance, OHAUS FCF System. The micro-hardness was measured using Vickers indentation method at a load of 1kg/f with a dwell indentation time of 10s and the test results of each sample was the arithmetic mean of 5 successive indentations with standard deviation.

2.2. Sliding wear test

Tribological behaviour of the sintered materials was studied under dry sliding conditions. The surface of each sample was prepared according to metallographic standards (grinding and polishing). Wear tests were then carried out on the standard tribometer using ball on disc technique. The counterpart for wear testing was Al₂O₃ with 6mm diameter. Sliding tests were conducted in unlubricated ambient temperature. The sintered cermet disc was rotated at 300 rpm and the ball was kept stationary. A load 10 N was applied on the ball. The coefficient of friction was also estimated during the tests and the dominant wear mechanisms were also identified using SEM analysis of surfaces of worn cermet discs.

3. Results and discussion

The morphology of the mixed and as received powders as observed by FESEM are shown in Figure 1. The as mixed powders in Figure 1 c and d show the clustering of graphite particles with sizes of several micrometers of $\text{TiC}_{0.7}\text{N}_{0.3}$. In 1.5 % Gr almost all of the carbon atoms in graphite dissolved or mixed completely in titanium carbonitride. No significant carbide phase can be observed in the microstructure as compared with 0.5% Gr and in Figure 1 c and d.

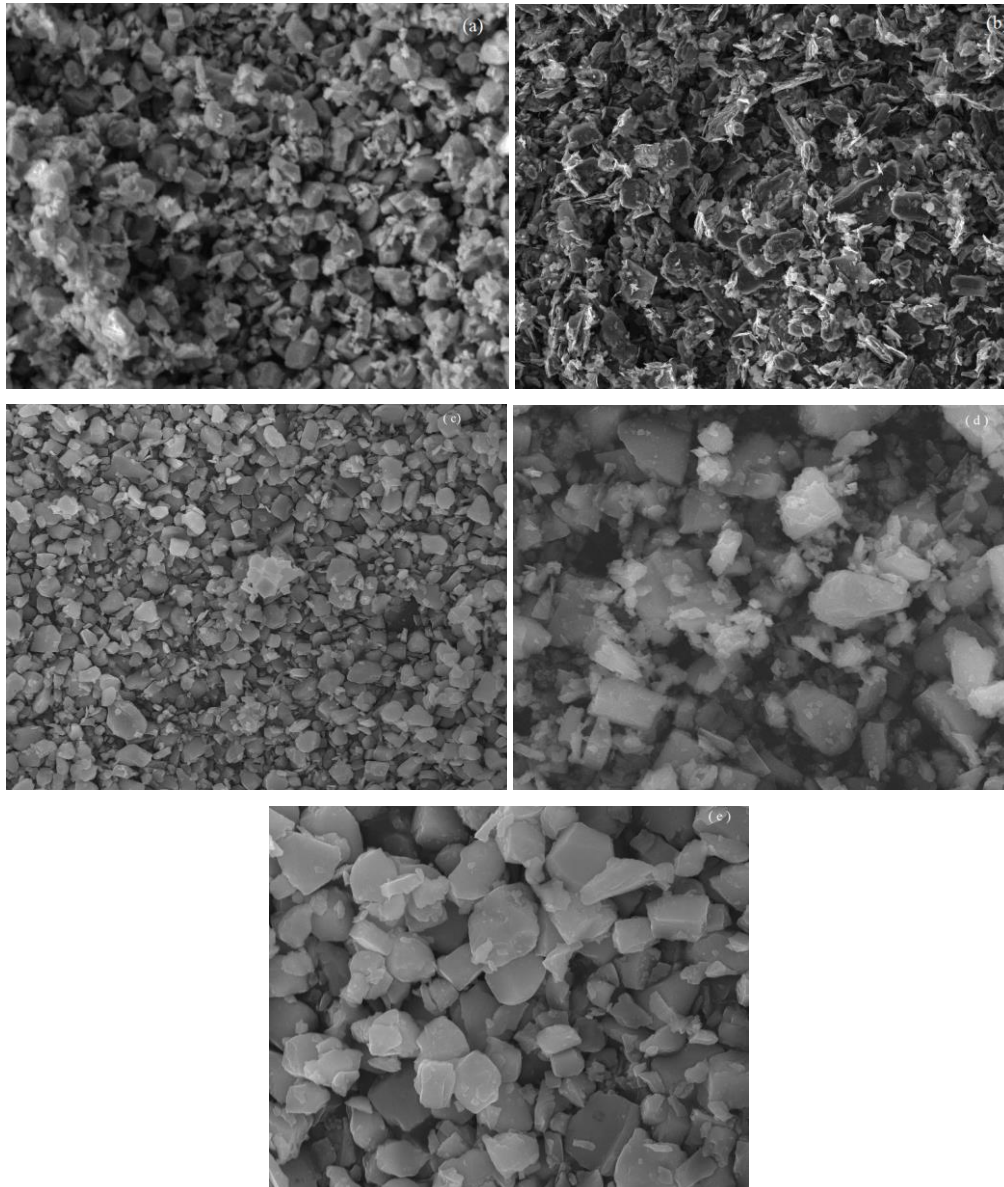


Figure 1. SEM microstructures of the powders: a). $\text{TiC}_{0.7}\text{N}_{0.3}$ and b). Graphite as received. c). 0.5 % Gr, d). 1.0 % Gr and e). 1.5 % Gr.

Figure 2 shows the SEM micrograph of sintered pure $\text{TiC}_{0.7}\text{N}_{0.3}$ with 0% Gr, 0.5% Gr, 1.0% Gr and 1.5% Gr respectively after swabbing in macro-etchant containing (50 ml Di water, 40ml HNO_3 and 10ml HF) for 5-8 minutes at 60 – 80 °C. The SEM images reveal differences in porosity among (0% Gr, 0.5% Gr, 1.0% Gr and 1.5% Gr) sintered samples at 2100 °C temperature. The liquid phase had disappeared at 2100 °C due to the powerful sintering driving force under SPS. The black spot in the image shows the graphite entrapped in the pore as a result of undissolved carbon.

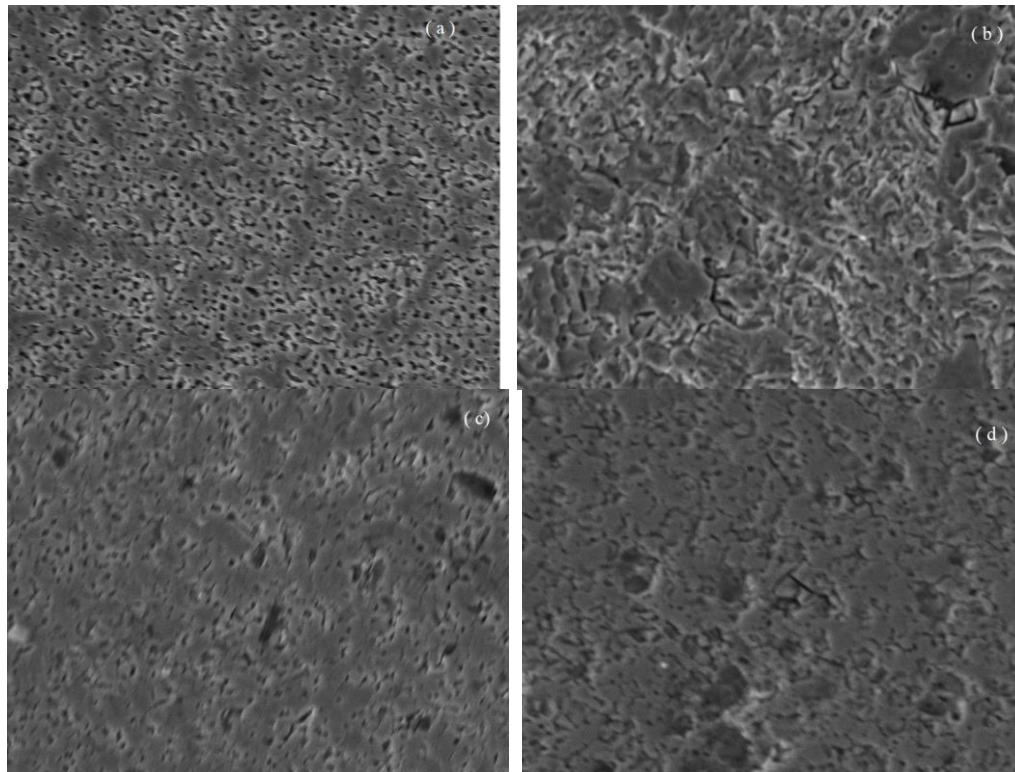


Figure 2. SEM analysis of the sintered TiCN cermet: a). Pure $\text{TiC}_{0.7}\text{N}_{0.3}$ (0% Gr), b). 0.5 % Gr, c). 1.0 % Gr and d). 1.5 % Gr.

The variations in the relative density and the sintered $\text{TiC}_{0.7}\text{N}_{0.3}$ based cermet with graphite additions are presented in Table 1. It can be observed that the addition of graphite has a noticeable improvement on the densification of the cermet. In Table 1, composition B and C obtained the best densification while there is a slight reduction in hardness 2192 Hv. Further increase in amount of carbon resulted in a decrease in relative density. Similar trend was also observed on the hardness value. Furthermore, it is observed that hardness decreases due to a decrease in densification.

Table 1. Composition and properties of the sintered materials.

| Composition wt % Graphite | Relative Density (%) | Hardness Hv ₁ |
|---------------------------|----------------------|--------------------------|
| a. Pure TiCN 0% Gr | 98 | 2168 |
| b. 0.5 wt % Gr | 99.5 | 2297 |
| c. 1.0 wt % Gr | 99 | 2277 |
| d. 1.5 wt % Gr | 98 | 2192 |

The evolution of coefficient of friction during ball on disk tribotests at room temperature is summarised in Figure 3. Due to the solid lubricating behaviour of graphite in the composite, the friction of coefficient significantly reduced with an increase in carbon content. It was observed in Figure 3 that COF of composites containing high amount of graphite decreases as compared with the one without graphite. Although a sample with 0.5% Gr composition had higher densification and hardness, sample with 1.0 % Gr composition can be more preferred for wear resistance application. The wear tracks are of the $\text{TiC}_{0.7}\text{N}_{0.3}$ based cermet sintered compacts at room temperature are shown in Figure 4. The main wear damage observed was abrasion. It can be observed that composite containing 1.0 % Gr has good wear resistance as compared to other samples used for this work. For wear resistance application, results obtained from this study, suggest that composition with 1.0 % Gr addition will be more suitable.

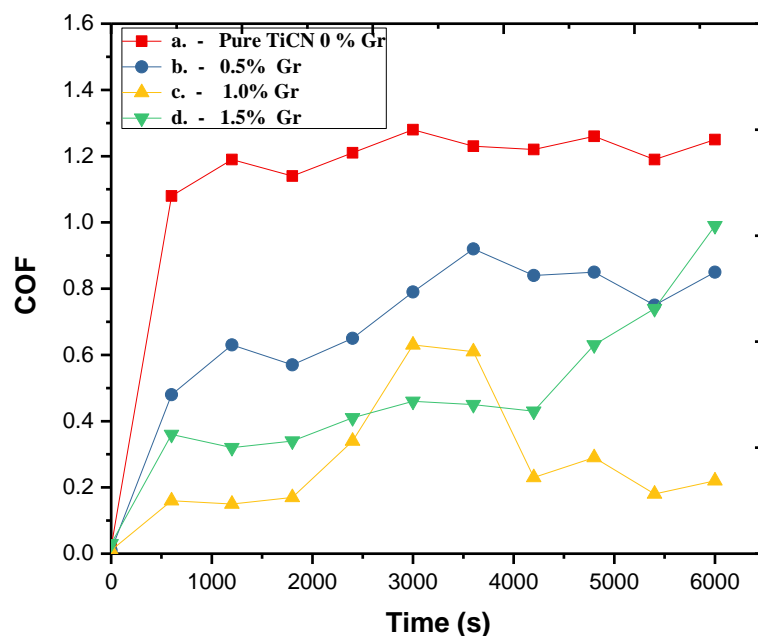
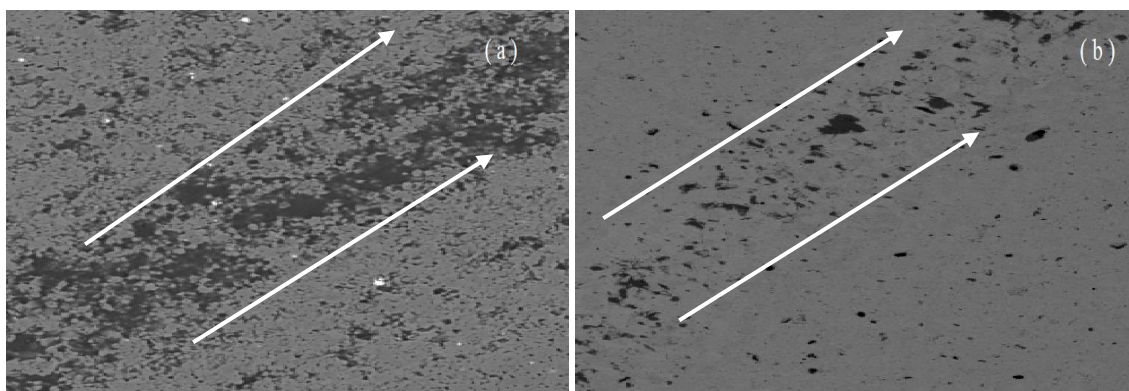


Figure 3. Wear tracks of all experimental materials at ambient temperature against alumina ball - SEM: a). Pure $\text{TiC}_{0.7}\text{N}_{0.3}$ (0% Gr), b). 0.5 % Gr, c). 1.0 % Gr and d). 1.5 % Gr



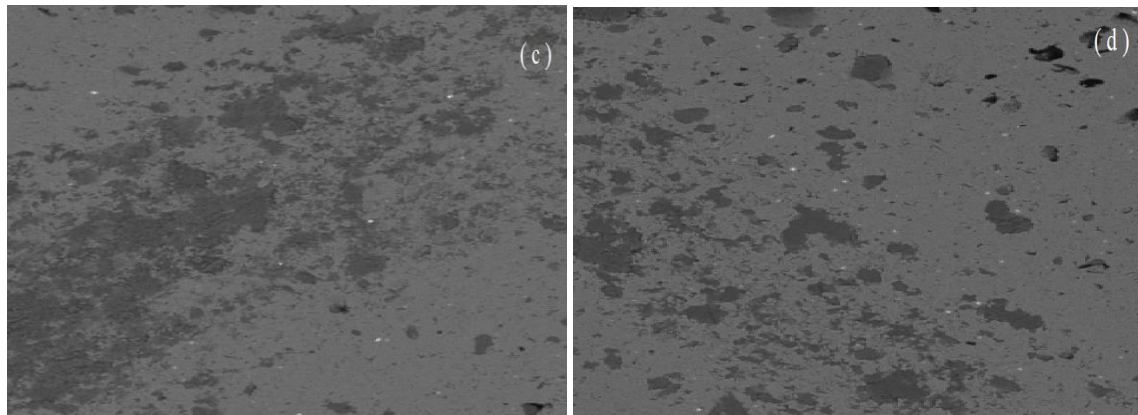


Figure 4 . Wear tracks of all experimental materials at ambient temperature against alumina ball - SEM. : a). Pure $\text{TiC}_{0.7}\text{N}_{0.3}$ (0% Gr), b). 0.5 % Gr, c). 1.0 % Gr and d). 1.5 % Gr.

4. Conclusion

In this study, the tribological behaviour of $\text{TiC}_{0.7}\text{N}_{0.3}$ composition based cermet reinforced with graphite additions was investigated. Based on the outcome reports, the following conclusions can be made:

- Pure $\text{TiC}_{0.7}\text{N}_{0.3}$ with or without can be successfully consolidated by spark plasma sintering with densification of 99.5%.
- It is observed that an increase in graphite addition of up to 0.5 % Gr and 1.0 % Gr improves densification and hardness.
- A cermet with reduced COF was produced and the worn surface of the samples revealed abrasive wear mechanism.
- Sample containing 1.0 % Gr was the most resistant to wear as compared to the other testing compacts.

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