

# Microstructure and Wear Properties of Laser-Cladded cBN/Ti<sub>3</sub>Al on Pure Titanium

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**Abstract** In order to improve the tribological properties of titanium alloys at high temperature, the possibility of producing Ti<sub>3</sub>Al intermetallic with the addition of ceramic (cBN) coatings on titanium substrate using laser technique cladding was investigated. cBN is generally known for its high hot hardness, wear resistance and chemical stability. Laser cladding is an emerging material processing technique which is an efficient and cost-effective technique for improving the surface properties of general metallic materials. This paper presents the effects of laser cladding on the phase combination, microstructure, hardness and wear resistance of titanium aluminide/cBN IMC composites at different variations in quantity of cBN in the composite. Optical microscopy, X-ray diffraction and scanning electron microscopy with EDX were used for characterising the microstructure of the coating. In addition, the composite coating was subjected to wear testing using the ball-on-disc, friction and wear apparatus. The XRD results revealed phases with small cBN, Ti<sub>2</sub>N and TiB<sub>2</sub> peaks in addition to the rich  $\gamma$ -Ti,  $\alpha$ 2-Ti<sub>3</sub>Al and TiAl intermetallics phases in the coatings after cladding process, whereas the microstructure of the cBN cladded layer contained partially melted cBN grains evenly dispersed within the laths of alpha Widmanstätten phase in the form of dendrites, precipitate of  $\alpha$ 2-Ti<sub>3</sub>Al and spherical-shaped pure titanium. The effect of the addition of cBN into Ti<sub>3</sub>Al alloy at increasing weight percentages showed an improvement in the hardness and wear resistance of the coatings. The increase is attributed to reacted boron

nitrides particles with titanium, being distributed uniformly in the intermetallic matrix.

**Keywords** Titanium · Intermetallics · Laser cladding · cBN · Wear

## 1 Introduction

Laser cladding is an efficient, cost-effective and environmentally friendly surface modification technology, which has been extensively used for providing thermal barrier (TBC) coatings for engineering components [1]. During laser cladding process, a high-power laser beam is utilised to locally melt the coating materials and a thin surface layer of a substrate material; thus, a new layer of material with desired properties is formed after solidification [2].

Titanium alloys are widely used in the aerospace, chemical and medical industries owing to the excellent combinations of specific mechanical properties and corrosion resistance. However, the poor tribological properties of these alloys have limited its use in severe wear applications [1, 3, 4]. Single and multiple ceramic materials are commonly used as coatings in improving the surface properties of titanium. However, these materials are highly susceptible to cracking, thus restricting their industrial applications [5, 6]. TiAl intermetallics are normally used as a binding phase between titanium alloys matrix and the ceramic reinforced phase to reduce the cracking and residual stress associated with ceramic coatings [6]. Furthermore, TiAl present several advantages such as low density, high melting point, good structural stability, high strength at elevated temperature, good corrosion resistance, high elastic modulus and good creep resistance [2, 7, 8]. cBN is found to be a suitable material for improving wear resistance of metal

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composites because of its high hardness, wear resistance and excellent chemical stability at high temperatures [9–11].

Laser cladding of combination of Ti, Al and ceramics such as Ti–Al–B<sub>4</sub>C, TiN, TiC and SiC reinforced Ti<sub>3</sub>Al IMC coatings on titanium and its alloys has been extensively studied [12–14]. The authors reported improvement in the hardness and wear resistance of the resulting surface after laser cladding. Selvan et al. [15] investigated CO<sub>2</sub> laser alloying of pre-placed BN coating on Ti–6Al–4V alloy. They observed the formation of titanium nitride and titanium boride in the coating, and hardness up to 1700 was recorded. Although the effects of ceramic addition to TiAl intermetallics have been reported, there are no reports on laser cladding of the combination of cBN with Ti<sub>3</sub>Al intermetallics.

This study presents the effects of laser cladding on the phase combination, microstructure, hardness and wear resistance of titanium aluminide/cBN IMC composites at different variations in quantity of cBN in the composite.

## 2 Experimental Procedure

Commercial cBN powder (average particle size of 100 μm), titanium powder (average particle size of 60 μm) and aluminium powder (average particle size of 200 μm) were cladded on the surface of 99.6% pure titanium substrate (75 mm × 75 mm). Before the laser cladding experiments, the titanium and aluminium powders were premixed in nominal chemical constituents (atomic fraction, %) of 70Ti–30Al ratio of 70:30, respectively, in order to produce Ti<sub>3</sub>Al intermetallics alloy, through the reaction of Ti and Al during the laser cladding process. cBN powders were fed in volume ratio of 5, 10 and 15% using a dual powder feeding system hoppers. Before the cladding process, the substrate was sand-blasted and then rinsed in acetone to clean the surface.

Laser cladding was carried out by a Kuka robot, 4.4 KW Nd-YAG laser machine. The deposition was performed using a laser focal length of 195 mm and beam spot size of 4 mm. Laser power and scanning speed were kept constant at 1500 W and 1.0 m/min, respectively. These parameters were fixed at optimum parameters, based on previous experiments in order to produce claddings with good metallurgical bonding. The cladding powders were shielded by argon gas at a flow rate of 2 l/min with the oxygen level being kept in the 10 ppm level (to avoid oxidation of the substrate and as well as the cladded layer, through reaction with oxygen) using a glove box.

X-ray diffraction analysis was performed using Bruker D8 Venture Photon CMOS. Microscopic examination was performed by means of an optical microscope (Olympus BX51 M) and scanning electron microscope (ZEISS) equipped with EDAX Oxford energy-dispersive spectroscopy (EDS). Microhardness was measured using a Vickers micro-

hardness testing machine (EMCOTEST®). The test load of 500 g/f with a dwell time of 10 s was used. Ten indentations were applied per sample, and the average diagonal values of the samples were recorded.

Dry sliding wear test was performed on the cladded surfaces using the ball-on-disc mode on friction and wear apparatus. The sample was preloaded with counterbody balls made of tungsten carbide with diameter of 10 mm. The applied load was 25 N with stroke length of 2 mm, sliding distance of 1000 m and sliding time of 1000 s at a frequency of 5 Hz. The coefficient of friction was plotted against time for 1000 s and recorded by the computer attached to the tribometer. Wear resistance testing was performed according to ASTM G133-05 standards [16]. Wear volume loss of the substrate and coating was calculated from Eq. (1) [16, 17].

$$V_{\text{total}} = V_A + V_B = L \left\{ r^2 \sin^{-1} \left( \frac{W}{2r} \right) - \frac{W}{2} \left( r^2 - \frac{W^2}{4} \right)^{1/2} \right\} + \frac{\pi}{3} \left\{ 2r^3 - 2r^2 \left( r^2 - \frac{W^2}{4} \right)^{1/2} - \frac{W^2}{4} \left( r^2 - \frac{W^2}{4} \right)^{1/2} \right\} \quad (1)$$

where ‘r’ is the radius of the sphere used, ‘w’ the wear track width and ‘L’ the length of the wear track. The micrograph of the wear surface morphology and wear debris of the coatings was observed under the SEM.

## 3 Results and Discussion

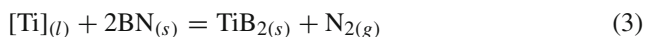
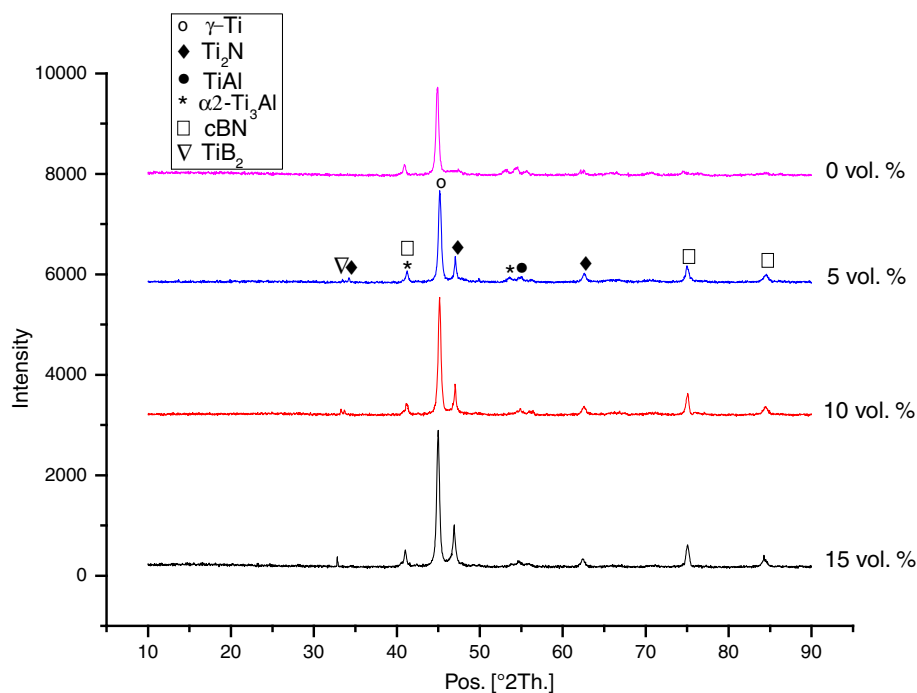
### 3.1 Microstructure Characterisation

The X-ray diffraction pattern of the cBN/Ti<sub>3</sub>Al composite coatings on the pure titanium substrate is shown in Fig. 1. The intermetallic coating with 0 vol% cBN has a phase composite consisting of mainly α2-Ti<sub>3</sub>Al intermetallic and γ-Ti solid solution. During the reaction between the individual elements of Ti and Al, it is considered that the ordered stoichiometric phase of α2-Ti<sub>3</sub>Al is preferred to form. However, the high peaks of pure titanium (γ-Ti) in the coating showed the presence of incomplete reaction of the titanium with the aluminium powders.

The addition of cBN powders in the coatings showed phases with small cBN, Ti<sub>2</sub>N and TiB<sub>2</sub> peaks in addition to the rich γ-Ti, α2-Ti<sub>3</sub>Al and TiAl intermetallics phases. The reaction of pure titanium with the boron nitride leads to the formation of Ti<sub>2</sub>N and TiB<sub>2</sub> compound in the coatings. The degree of formation of Ti<sub>2</sub>N and TiB<sub>2</sub> compound during the exothermic reaction between Ti and BN could be described as follows:



**Fig. 1** XRD pattern of the composite coatings on the pure titanium substrate



In the reaction described above, the dominating factor in the formation of Ti–N and Ti–B compounds is titanium.

The diffraction intensities of cBN,  $\text{Ti}_2\text{N}$  and  $\text{TiB}_2$  peaks, however, increases as the percentages and the amount of reaction product increase in the composites coatings. A more noticeable broader intensity of the  $\text{Ti}_2\text{N}$  peaks is visible at increasing cBN content from 10 to 15% due to the dissolution of nitrogen atoms in the hcp Ti lattice; the broader peaks indicate a more amorphous state of the titanium nitride.

Figure 2a–d shows the scanning electron micrograph (SEM) of the deposits on the substrate after laser cladding of the titanium aluminide powders. The coatings on all the samples produced were observed to be metallurgically bonded with the substrates and the microstructure relatively homogeneous. The microstructure of the intermetallic coatings with 0 vol% boron nitride (cBN) content appeared to be free of cracks, but few pores are visible within the coatings. However, the microstructure of the samples containing 15 vol% cBN in the composite coatings has few cracks (see Fig. 2e), usually towards the end of the heat-affected zone. The cracks are due to the surface tension gradient and high convective flow between the molten intermetallic liquid and the partially molten cBN powder particles during the rapid solidification of the coatings during laser cladding, thus leading to stresses

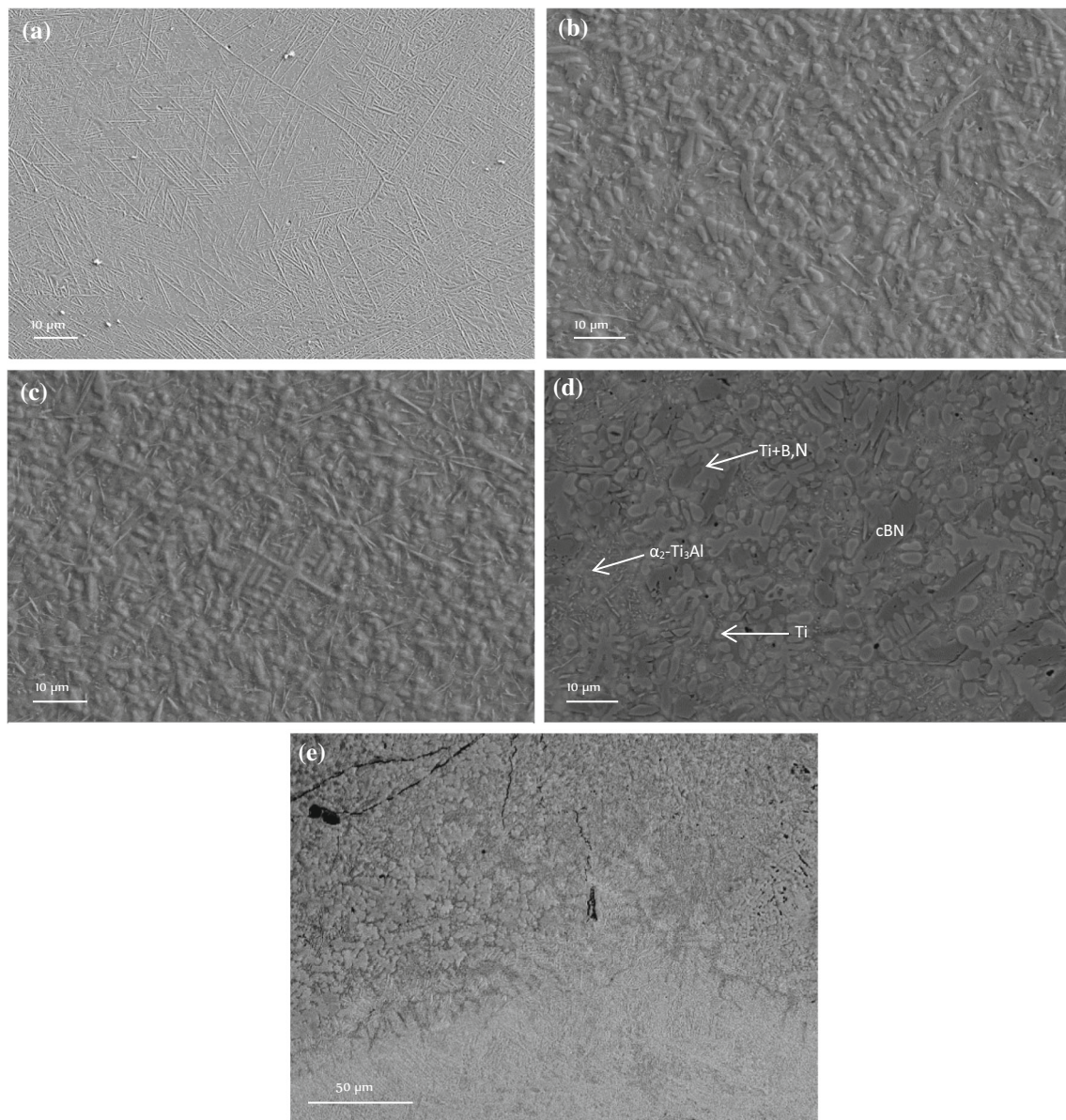
in the coatings [15, 18]. It can be noted that the number of partially melted cBN particles within the microstructure is more as compared at optimum cBN composition.

In addition, the titanium aluminide coatings with 5, 10 and 15 vol% cBN content have few visible pores within the microstructure. The pores increase with the increase in the cBN content in the deposits. The pores are caused by the gas [possibly  $\text{N}_2$  that escaped during the exothermic reaction of Ti with BN as indicated in Eq. (2)], produced from the in situ synthesised  $\text{TiB}_2/\text{cBN}/\text{Ti}/\text{Ti}_2\text{N}/\text{Ti}_3\text{Al}$  IMC coating, entrapped within the molten pool as a result of large viscosity induced by the material particles during the laser cladding process [3, 17, 19].

The microstructure of the layer cladded with 0 vol% cBN consisted mainly of Widmanstätten alpha phase in beta matrix (see Fig. 2a). The microstructure of the titanium aluminium cladded layer with 5, 10 and 15 vol% cBN content as shown in Fig. 2b–d, contained cBN grains (partially reacted with titanium particle) evenly dispersed within the laths of alpha Widmanstätten phase in form of dendrites and as well as precipitate of  $\alpha_2\text{-Ti}_3\text{Al}$ , and spherical-shaped pure titanium. As the percentage of the cBN increases in the cladded layer, the structure of the cBN grains is more defined. Partially melted cBN particles dispersed within the cladded layer was observed mostly visible in the 15% cBN cladded layer.

Figure 3a shows the microstructure of the intermetallic coatings with 15 vol% boron nitride (cBN), showing the deposit zone, bond zone, heat-affected zone and the substrate. Partially reacted cBN particles with fine equiaxed precipi-





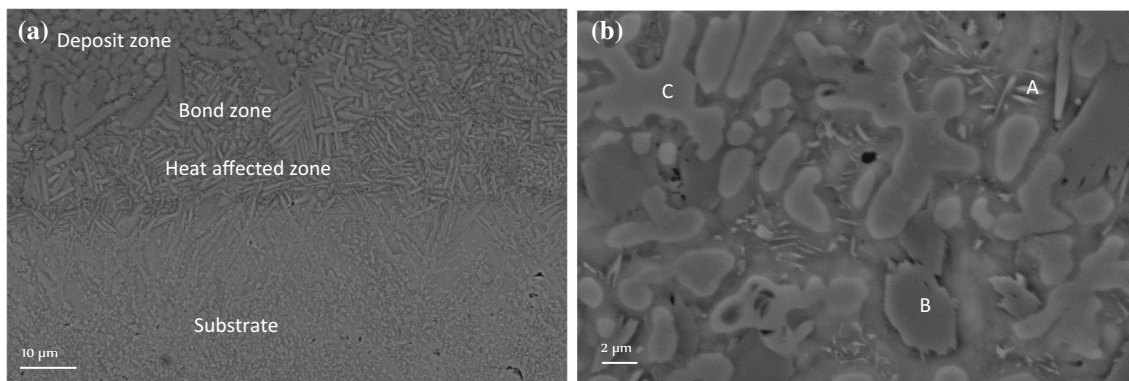
**Fig. 2** Microstructure of the laser-cladded composite coatings on the pure titanium substrate at varying quantity of cBN **a** 0 vol%, **b** 5 vol%, **c** 10 vol%, **d** 15 vol% and **e** crack formation (15 vol%)

tates of  $\alpha_2$ -Ti<sub>3</sub>Al were commonly observed in the deposit zone (Fig. 3b). The bond zone and heat-affected zone near the substrate contained acicular martensite with distributed lamellae phase (phase composition of 13.9 at.% Al and 86.1 at.% Ti), while the substrate contained recrystallised alpha grains, and particles of beta stabilised by impurities.

The microstructure of the titanium aluminide intermetallic coating with 15 vol% cBN content at the deposit zone (higher magnification) is shown in Fig. 3b. The figure shows a clearer representation of the morphological features of the deposit zone. The results of the EDS analysis of the phases with different morphological features are presented in Table 1. The darker dendritic phase showed a BN-rich region, whereas

the composition of BN is much lower in the lighter dendritic phase. The white needle-like particles observed in the alloyed layer is composed mainly of  $\alpha_2$ -Ti<sub>3</sub>Al and TiB<sub>2</sub>. The spherical particles contain mainly titanium, and the dendritic structure consists mainly of titanium nitride in atomic ratio approximately 2:1, identified as Ti<sub>2</sub>Ni, similarly observed by [15]. These elements were also confirmed with the XRD traces as shown in Fig. 1.

In summary, the microstructure of the cladded IMC coatings showed different morphological features from the surface (deposit zone) to the substrate with refined cBN grain particles as the vol% cBN content increases in the deposits. The formation of hard ceramic compounds in situ synthe-



**Fig. 3** Microstructure of titanium aluminide intermetallic coating with 15% cBN content. **a** Interface. **b** Deposit zone at 10,000×

**Table 1** Chemical composition (at.%) of the phases formed on the 15 vol% cBN content coating

Analysis zone	Ti	Al	BN
A	79.45	21.55	–
B	62.27	0.90	36.83
C	99.49	0.51	–

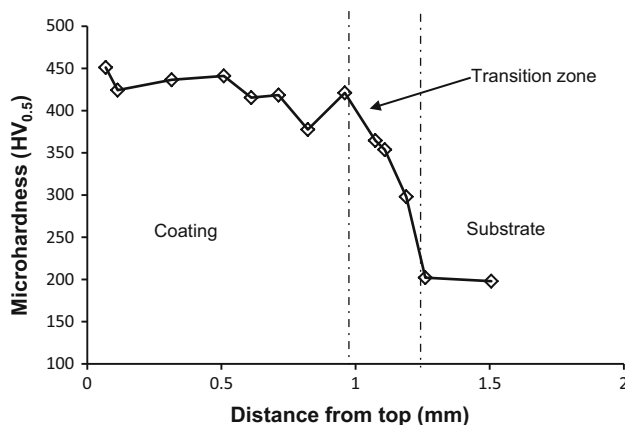
sised and Ti–Al intermetallic was more predominant in the coatings clad with higher vol% cBN content.

### 3.2 Microhardness

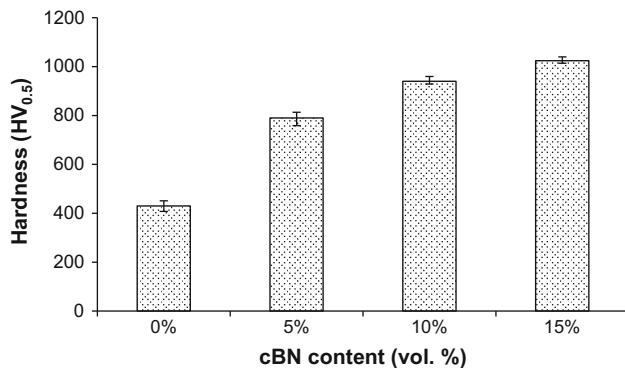
Figure 4 shows the graphical profile of the Vickers microhardness measured across the depth direction of the laser-clad sample with layer of titanium aluminide intermetallic with 0 vol% cBN from top to bottom. It was observed that the microhardness decreases towards the substrate along the profile. The hardness of the deposit zone was higher than that of the substrate as shown in Fig. 4. The average hardness at the deposit zone (coating) is approximately 430 HV<sub>0.5</sub> compared to the pure titanium substrate of 190 HV<sub>0.5</sub>. The higher hardness at the deposit zone is attributed to the presence of homogeneous dispersion of TiAl and α<sub>2</sub>-Ti<sub>3</sub>Al phases within the coating [19].

Similarly, high hardness that is slightly lower than that of the deposit zone was observed at the transition between the deposit and heat-affected zone. This hardness can be attributed to possibly better crystallisation of the grains at this zone and also the lower heat experienced at the lateral inner part of the substrate [20].

Figure 5 presents the average Vickers microhardness of the coatings of the samples with 0, 5, 10 and 15 vol% cBN content. The hardness increases with an increase in the percentage of cBN addition to the clad layer. The average hardness for the 0, 5, 10 and 15 vol% cBN content is 430 HV<sub>0.5</sub>, 790 HV<sub>0.5</sub>, 940 HV<sub>0.5</sub> and 1024 HV<sub>0.5</sub>, respectively.



**Fig. 4** Microhardness profile of coatings containing 0% cBN content



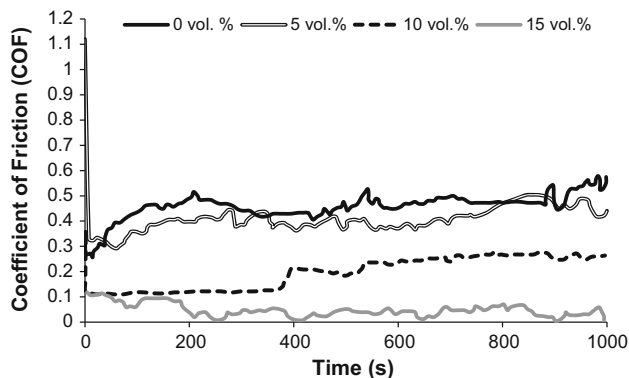
**Fig. 5** Hardness of the composite coatings clad on the pure titanium substrate

The small addition of cBN up to 5 vol% to the titanium aluminide in the coating increased the hardness to almost twice the value. The increase in the hardness at the clad layer with increasing cBN content is due to the presence of hard cBN particles (precipitates of titanium nitride and titanium boride) dispersed within the TiAl and α<sub>2</sub>-Ti<sub>3</sub>Al phases.

### 3.3 Wear Resistance Behaviour

Figure 6 presents the results of the experimental coefficient of friction of the dry sliding wear resistance tests plotted against time for the coatings cladded on the pure titanium substrate. The coefficient of friction of the coatings was generally observed to increase slightly with the increase in time. A high coefficient of friction was recorded for the coating containing 0 vol% cBN content. The coefficient of friction reduces as the cBN content is increased from 5, 10 and 15 vol%, respectively. This indicates that the coatings with higher percentage of cBN exhibited better frictional coefficient under the normal load. At optimum composition of cBN content (15 vol%), it can be seen that the coefficient of friction reduces with time. This can be attributed possibly to the low penetration of the sliding ball and the presence of more partially melted cBN particles within the matrix of the titanium alloy causing ore resistance to the tungsten carbide counterbody. The hard cBN particles embedded in the softer titanium aluminide matrix help increase the sliding resistance, thus decreasing the value of the frictional coefficient. The presence of the cBN improved the wear resistance performance of the substrate.

Figure 7 shows the wear volume of the pure titanium substrate and the titanium aluminium/cBN IMC coatings with different volume fractions of cBN sliding against WC ball under 25 N normal load. The volume of material removed from the substrate decreased by 2.3 times with the application of titanium aluminide as a coating on the substrate. In this light, less material was removed from the coating with addition of cBN particle to the titanium aluminide. The volume of material removed decreased by 3.2, 4.2 and 8.8 times for 5, 10 and 15 vol% addition of cBN to the titanium aluminide coating, respectively, as compared to pure Ti substrate. The coating with mainly  $Ti_3Al$  (0 vol% cBN) had poor wear resistance compared to the titanium aluminium/cBN IMC coatings containing 5, 10 and 15 vol% cBN content.



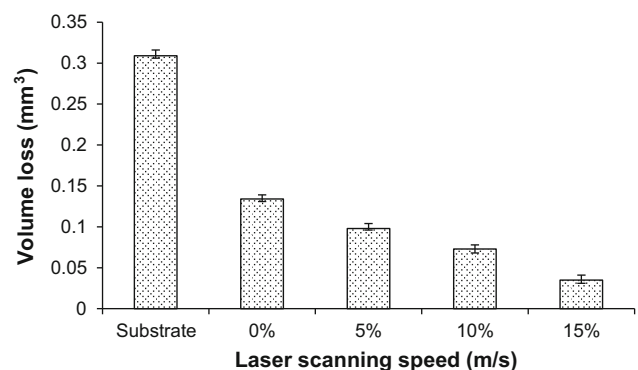
**Fig. 6** COF against time during dry sliding wear resistance tests of the titanium aluminium/cBN IMC coatings

The poor wear resistance results from its high coefficient of friction and higher chemical affinity with the WC balls [21]. As observed, the wear volume of the cBN/  $Ti_3Al$  IMC coatings decreases with the increase in the volume fraction of cBN content in the coatings. The coating with 15 vol% cBN had the highest wear resistance.

The SEM micrograph of the worn cBN/  $Ti_3Al$  IMC coatings with different volume fractions of cBN powder after sliding under the WC ball is shown in Fig. 8. All the coatings showed loose debris on the worn surfaces containing W, C and O elements indicating the occurrence of oxidation wear [22]. For the  $Ti_3Al$  coating, the wear mechanism observed is mainly abrasive wear feature as evidenced by grooves on the worn surface. The wear tracks comprised abraded regions with continuous microgrooves parallel to the direction of sliding.

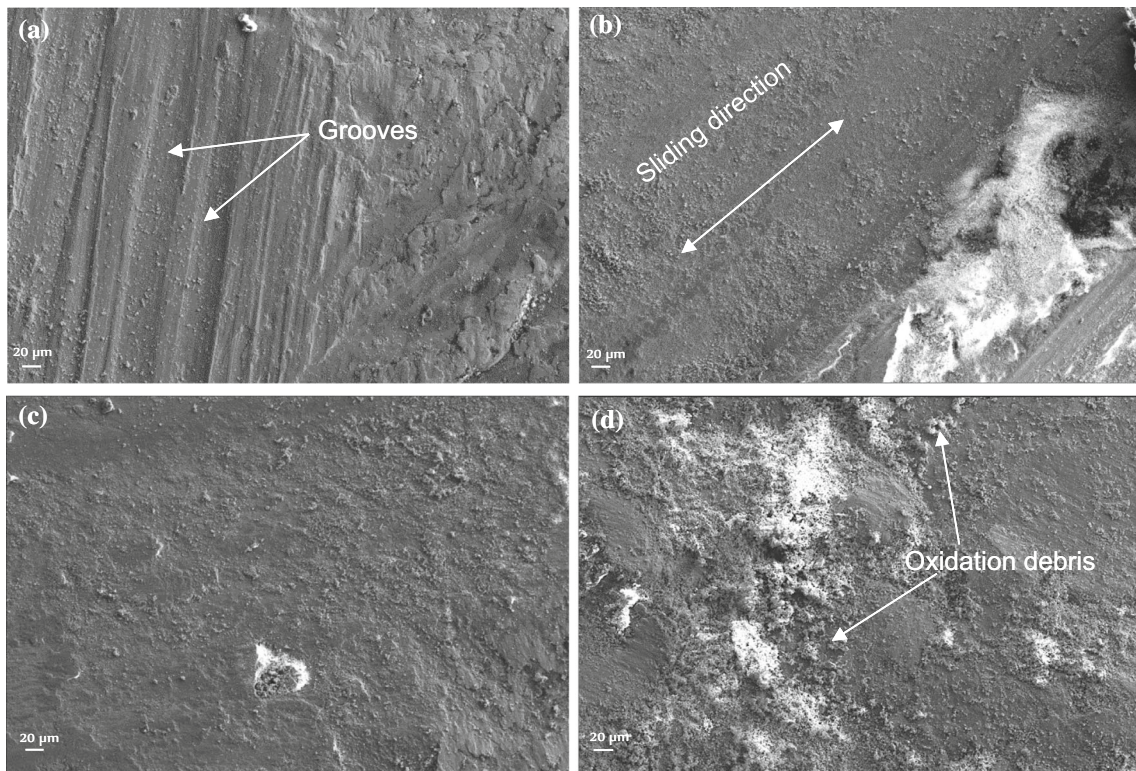
The cBN/ $Ti_3Al$  IMC coatings with different fractions of cBN powders showed different wear mechanism. For the coatings with 5 vol% cBN the worn surface showed evidence of plastic deformation as well as abrasive wear features, whereas the worn surface of coatings with higher cBN content is mostly covered with pits and transferred layer without the features of microgrooves, indicating a combination of severe plastic deformation, oxidation and adhesive wear (observed as adhesive craters on the wear surface) features. Evidence of wear debris consisting of small flakes and tiny particles was observed on the worn surfaces (see Fig. 8). The presence of plastic deformation in the sliding contact area is responsible for the surface morphology [23]. Evidence of pull out of harder phases (partially melted particles of cBN powder) was also observed on the worn surfaces of the coatings with 10 and 15 vol% cBN. The feature was more common with the coating with 15 vol% cBN. In addition, irregular wear debris and accumulated fine particles, micropatches and delaminated scars on the worn tracks were observed within the worn zone.

The hard ceramic reinforced phases of  $TiB_2$ - $Ti_2N$  in the cBN/ $Ti_3Al$  IMC coatings (5, 10, 15 vol% cBN) is responsible



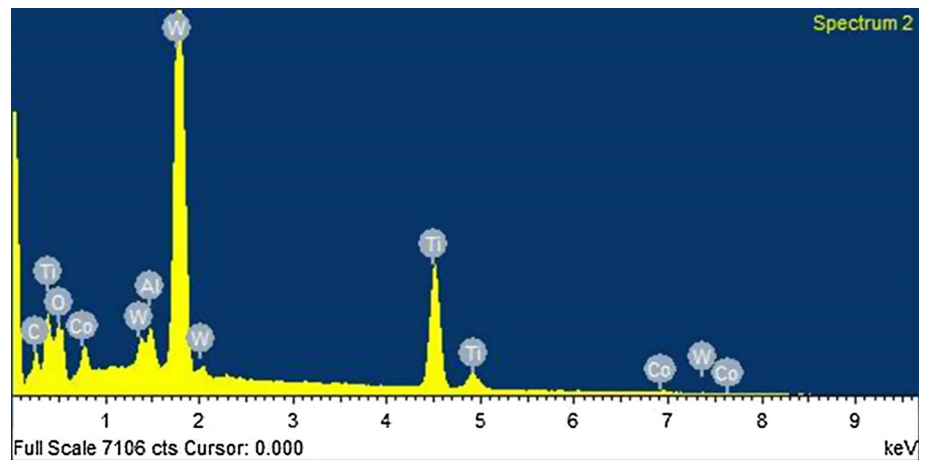
**Fig. 7** Wear volume loss for the titanium aluminium/cBN IMC coatings





**Fig. 8** SEM micrograph of the worn titanium aluminium/cBN IMC coatings **a** 0 vol%, **b** 5 vol%, **c** 10 vol%, **d** 15 vol%

**Fig. 9** EDS point analysis of wear debris



for the improvement in the wear resistance of these coatings by withstanding the external load applied by the sliding ball on the surfaces [24].

The EDS point analysis of the debris on the surface of the coatings containing 15 vol% cBN is shown in Fig. 9. The analysis showed the presence of elemental oxygen, aluminium, tungsten (originated from the contacting surfaces of the tungsten carbide counterpart), and titanium in atomic weight percentages of 27.3, 2.4, 34.3 and 14.9% respectively, leading to possible formation of titanium oxides and aluminium oxides. The presence of

oxygen confirms the considerable oxidation experienced at the contacting surfaces by the coatings and the counterbody.

#### 4 Conclusion

The study investigated the microstructure and wear resistance behaviour of laser-cladded cBN/Ti<sub>3</sub>Al composites on pure titanium substrate by varying the quantity of cBN in the composite. The following conclusions can be drawn.

The laser-cladded surfaces showed few pores and cracks which increased with the increase in the addition of boron nitride to the titanium aluminide intermetallic coating.

The coatings produced the formation of hard intermetallic compounds of titanium aluminide, titanium nitride and titanium boride as confirmed with XRD and EDS analysis.

The results obtained through microstructure of the intermetallic coatings with varying percentage of boron nitride (cBN) revealed that dendritic structure consists mainly of titanium nitride, white needle-like particles of  $\alpha_2$ -Ti<sub>3</sub>Al and TiB<sub>2</sub> and spherical particles of titanium.

Microhardness results showed that the hardness increases with an increase in the percentage of cBN addition to the cladded layer. The precipitates of titanium nitride and titanium boride as well as partially melted cBN were responsible for the increase in hardness of the cladded layer.

The results of the wear tests indicates that the wear volume of material removed from the surface of the substrate is higher than the wear volume of material removed from the deposit surface. The coefficient of friction of substrate was observed to be higher for 0 vol% cBN as compared to the 5, 10, 15 vol% cBN cladded layer. This is as a result of the hard ceramic reinforcement phases of cBN, Ti<sub>2</sub>N and TiB<sub>2</sub> formed during the laser cladding process. The addition of cBN to the titanium aluminide improved the wear properties of the alloyed cladded layer.

Thus, it can be concluded that improvement in the wear resistance, corrosion resistance, load-bearing capability and high temperature stability can be attained from the hardness level of the ceramic TiN and TiB phases formed during the laser cladding of pure Ti substrate surface. This makes the material more applicable in the aircraft propulsion systems and automotive industries.

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