

# THE EFFECT OF "B" TARGET VOLTAGE ON WEAR PROPERTIES OF TICrNb-hBN COATINGS

## Yaşar SERT\*<sup>®</sup>, Tevfik KÜÇÜKÖMEROĞLU\*<sup>®</sup>, Hojjat GHAHRAMANZADEH ASL\*<sup>®</sup>, Levent KARA\*\*<sup>®</sup>

\*Engineering Faculty, Mechanical Engineering Department, Karadeniz Technical University, 61080, Trabzon, Turkey \*\*Engineering Faculty, Mechanical Engineering Department, Erzincan Binali Yıldırım University, 24002 Erzincan, Turkey

yasarsert@ktu.edu.tr, tkomer@ktu.edu.tr, h.kahramanzade@ktu.edu.tr, lkara@erzincan.edu.tr

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**Abstract:** The present study aims to determine the effect of target voltage of boron on elevated temperature wear behaviour of newly designed (Ti, Cr, Nb)-hBN PVD coatings. For this purpose, this layer is grown on the AISI L6 (55NiCrMoV7) at various target voltages (600 V, 700 V) using a high-power impulse magnetron sputtering setup. The coating layer has a graded design and has been coated on the substrate surface in adherence with the following order: Cr – CrN – TiCrN – TiCrNbN and finally TiCrNb-hBN (constituting the working layer). The surface properties of the layer were determined using SEM and an optical profilometer. It is seen that the coatings were deposited on the surface in a granular structure pattern away from the deposition defect (such as a droplet or hole), and the roughness values increase as the target voltage increases. Phase analysis is determined using XRD, and average grain size calculations are performed using the XRD data. The coating layer has grown on the surface at TiN (112), CrN (311), NbN (111) and h-BN (001) orientations. Then, mechanical tests including microhardness and scratch tests were conducted on the specimens. Although the layer that is produced with both different parameters improves the hardness of the substrate (4.7 GPa), the hardness of the coating layer at the voltage of 700 V (24.67 GPa) is higher than that of others. Based on scratch tests, scratch crack propagation resistance (CPR) values were determined as 40 N2 and 1,650 N2 for coatings produced at 600 V and 700 V, respectively. The wear behaviours of specimens are specified using a ball-on-disc type tribometer at 450°C. It is seen that the coating with high hardness and scratch resistance offers unique contributions to the wear performance of the substrate. The optimum value of the target voltage to be used in the production of this innovative coating has been introduced into the literature.

Key words: TiCrNb-hBN, target voltage, elevated temperature, scratch, wear

#### 1. INTRODUCTION

The surfaces of the mechanical parts used in industrial applications are damaged owing to friction and wear, and as a result of relative contact, these eventually undergo enough wear so as to require being put out of use [1]. Wear in unsatisfactory service times increases the costs of manufacturing. For industrial companies, it has become an important need to improve the wear performance of the mechanical parts. From past to present, various researches have been carried out to meet this requirement [2]. It has been determined that the service life of the material can be increased by improving the wear performance attributable to some surface treatment methods [3]. Boronising, carburising, nitriding and especially films produced from thin hard coating with vacuum technology are some of the most widely used surface treatments [4, 5]. Among them, PVD coatings stand out with their effective use and successful results [6]. The binary, ternary and quaternary layers in the composite structure are produced from transition metals (Ti, Cr, Zr, Al, Nb, V, Y etc.), and these nitrides are widely used [7-9]. TiN is the first coating produced to improve the hardness, friction and wear properties of components [10]. Afterwards, with the addition of other transition metals to the TiN structure, ternary and quaternary coatings were produced, thus improving the performance of the TiN layer under harsh working conditions [11]. TiCrNb-hBN coating layer, which constitutes the novelty of this study, was produced to further the usage performance of TiN coatings for the above-mentioned purpose. Various techniques are employed in the production of PVD coatings [12, 13]. Among these techniques, the magnetron sputtering method is effectively used in the production of dense coatings that are free from deposit defects. In the magnetron sputtering method, the coating production parameters are one of the most important factors that control the coating properties. Therefore, the performance of the deposited layer depends on the correct selection of the coating parameters, including deposition parameters such as working pressure, bias voltage, target material voltage, duty time and frequency. Bhaduri et al. [14] deposited TiN coatings on the M2 steel surface using different bias voltage and target frequency values. When the effects of the variable parameters were examined, it was determined that the microhardness value improved steadily with increasing the target frequency from 50 kHz to 300 kHz [14]. Also, the critical load increased up to the bias voltage of -70 V and then decreased with the further increase of the bias voltage. The reason attributed for this is that the coating becomes densely layered, and the grain size decreases with the increase of the bias voltage [14]. In another study, the effect of Mo target current on CrMoSiCN coatings was determined. By increasing the current value from 0.5 A to 2 A, the columnar structure in the coating layer became denser and blurred. The coatings produced with 2 A exhibited the superior hardness and wear performance [15].



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## 2. MATERIAL AND METHODS

In this study, 1.2714 (55NiCrMoV7) hot work tool steels were prepared based on in-demand dimensions for the coating process and laboratory tests with the use of machining. Before the deposition processes, the substrates were ground via 400, 800, 1,200 and 2,000 mesh SiC papers and then polished with a 1- $\mu$ m Al<sub>2</sub>O<sub>3</sub> solution until surface roughness reached the value of Ra  $\cong$  50 nm. The polished substrates were cleaned with ethanol for the deposition process. Then, steel samples were coated with the TiCrNb-hBN layer using the high-power impulse closed field unbalanced magnetron sputtering method. Ti, Cr, Nb and LaB<sub>6</sub> targets were used to create the desired structure on the substrate surface. In the coating process, Ti current of 6 A, Cr current of 2 A and Nb voltage of 500 V were applied at constant values. In addition, the coating deposition time was 90 min. The coating assembly and the placement of the target materials in the system are shown in Fig. 1. The parameters used in the coating processes are given in Tab. 1.

Tab. '	I. Deposition	parameters

Coating No.	Bias voltage (V)	Working pres- sure (Torr)	"B" target voltage (V)
B1	100	2.5 x 10 <sup>−</sup> 3	600
B2	100	2.5 x 10⁻₃	700



Fig. 1. Coating unit and placement of targets in the system

The microstructure and coatings' thickness were examined using a scanning electron microscope (Zeiss EVO LS10, Germany). The average surface roughness of samples was determined using an optical profilometer (Nanofocus, Germany). The phase analyses of coatings were carried out using Bragg-Brentano mode XRD (Panalytical X'Pert3, UK). The hardness and adhesion strength of the coatings were determined using nanohardness (Hysitron Ti 950, USA) and scratch tests (CSM Revetest RST, USA). The wear behaviours of the samples were investigated using a ball-on-disc type tribometer (UTS, Turkey) at 450°C, as shown in Fig. 2. The wear tests were carried out using the parameters of a constant load of 2 N, wear track diameter of 10 mm, cycles of 2,000 and velocity of 60 mm/s. An Al<sub>2</sub>O<sub>3</sub> ball with a diameter of 6 mm was used for the counter body. Al<sub>2</sub>O<sub>3</sub> counter bodies were preferred owing to their high hardness and toughness. In this way, it is possible to determine the wear resistance of the produced coatings under more difficult challenges. The wear rates of the coatings were determined using the Archard equation: W = V/(P\*d), where W is the wear rate (mm<sup>3</sup>/Nm), V is the wear volume (mm<sup>3</sup>), P is applied load (N) and d is the distance of sliding (m). The wear mechanism was characterised as being a result of SEM investigations of the wear tracks.



Fig. 2. The ball-on-disc type tribometer (UTS, Turkey)

## 3. RESULTS AND DISCUSSION

The surface and cross-sectional SEM images of the produced coating layers are shown in Figs 3 and 4. As seen in the figures, the coating layer became denser and fine-grained with increasing "B" target material voltage (600–700 V). It was determined that by increasing the target material voltage, the mobility of the ions sputtered from the target material increased and better absorbed to the substrate surface. On the other hand, the coating thickness values decreased with the increase of the voltage value. While a coating thickness of 1.33  $\mu$ m is obtained in the B1 coating produced at 600 V, this value decreased to 967 nm at 700 V. Tab. 2 confirms that the obtained elemental proportions of the coatings are consistent with the coating structures.



Fig. 3. The surface and cross-sectional SEM image of B1 coatings



Fig. 4. The surface and cross-sectional SEM image of B2 coatings



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ab. 2.	Chemical	composition	of coatings	(wt.%)
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Coating No.	Ti	Cr	Nb	В	N
B1	28.88	21.77	1.9	12.78	29.11
B2	18.25	18.22	0.9	15.9	49.54



Fig. 5. XRD graph of coatings (A) TiCrNb-hBN (600 V) and (B) TiCrNbN-hBN (700 V)

Tab.	3.	The	mechanical	and	tribologi	cal pro	perties	of	samp	bles

Sam- ples	Grain size (µm)	Surface rough- ness (nm)	Nano- hard- ness (GPa)	Adhe- sion strengt h (N)	CPRs (N²)	CoF (µ)
Sub- strate	-	50	4.7	-	-	0.67
B1	37.8	55	12.87	13	40	0.62
B2	19.8	72	24.67	85	1,650	0.26

The XRD graphs of coatings are given in Fig. 5. According to this figure, h-BN, NbN, Fe, TiN and CrN phases are formed in these coatings. The lowest intensity of Fe peaks is determined in the B1 (TiCrNb-hBN/600 V) coating layer. It is perceptible on analysis that this finding demonstrates a good consistency with the changes occurring in coating thickness values, i.e. a higher intensity Fe peak, together with the substrate material effect, is observed to characterise coatings that have a lower thickness value. Among the phases forming the coating, CrN and NbN are dominant structures especially in the B2 coating. It is emphasised that the peak widths increase with the increase in the "B" target voltage value in the coatings, which is a sign of a decrease in

grain size. In calculations carried out with the Scherrer approach using XRD data, grain size values are determined as 37.8 nm and 19.8 nm in the B1 and B2 coatings, respectively, as seen in Table 2. The calculated grain size values are also compatible with the surface SEM images given in Fig. 2. In addition, the surface roughness values (Ra) of the coatings are given in Table 3. It is observed that the surface roughness of the coatings is higher than that of the substrate. However, as the B voltage value increased, the surface roughness value also increased.

The hardness, adhesion strength, friction coefficient (COF) and wear rate values of the coatings obtained as a result of mechanical and tribological tests are shown in Table 3. As can be seen from this table, the nanohardness values of the coatings produced under both conditions (600 V and 700 V) are superior in comparison with those of the 1.2714 steel substrate. While the hardness of the substrate is determined as 4.7 GPa, the hardness of the coatings is obtained as 12.87 GPa and 24.67 GPa for the B1 and B2 samples, respectively. It should not be overlooked that the hardness value of the coating increases by approximately 100% with the increase of the target material voltage. It is observed that the hardness value of the B2 coating is superior in comparison with that of the other samples owing to its low grain and dense structure. It is striking that the TiCrNb-hBN coating deposited at a target voltage of 700 V has excellent resistance to plastic deformation. Considering the adhesion strength values of the coatings, a high Lc<sub>2</sub> value of 85 N is obtained in the B2 coating with high nanohardness. On the other hand, a relatively low adhesion strength value of 13 N is obtained in the layer produced at the 600 V voltage. Therefore, the high adhesion strength of the B2 coatings may also be associated with high hardness. Besides, it was obvious that the coating layers were exposed to plastic deformation in the scratch test. The higher hardness also provides higher resistance to plastic deformation. Previous studies pointed out that the adhesion strength values were improved linearly with increasing the hardness, as in agreement with the findings in the present study [16]. The scratch crack propagation (CPR) is a qualitative indicator of coating toughness, which can be formulated as CPRs: Lc1 \* (Lc2-Lc1). It is determined that the strength of fracture toughness increased as the target voltage of boron in the coatings increased.



Fig. 6. The COF graph of samples. COF, coefficient of friction

The COF graphs obtained as a result of the wear tests of the samples are given in Fig. 6. In the first stages of the wear tests, the COF values suddenly increased. In the literature, this stage is referred to as the running-in, i.e. the acclimation of the friction caused by the roughness of the surfaces to each other. As the



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surface roughness of the samples is crushed, the graph becomes more stable. In the base material where COF waves are shown in black, the surface roughness that was crushed after the running-in phase was broken by the progress of the cycle and caused wear.



Fig. 7. Optical profilometer image of wear paths of samples

As can be seen from the graph, this wear occurred in the form of third body wear when the particles that were trapped between the mating surfaces underwent detachment from the surface. The wear process undergone by the B1 coating is highly similar to that by the substrate, as demonstrated clearly by the fluctuations apparent in Fig. 6. After the running-in phase, the coating particles that were broken off in the continuation of the cycle caused abrasive wear here as well. Therefore, the coating layer lost its protection, and the wear continued on the 1.2714 steel. The situation is completely different in the B2 coating. After the sudden increase in the COF value, the graph progressed in a steady state up to 400 s. Subsequently, as manifest in the fluctuations visible in Fig. 6's graph, the partially ruptured par-ticles caused an increase in the COF, and this is attributed to the high hardness of the B2 coating layer. However, the wear seen after the 400th second did not separate the coating layer from the surface until the end of the cycle, thus ensuring that the COF was lower than that of the other samples. The optical profilometer images of the wear paths and the depth profile are shown in Fig. 7. The wear rate values of the coatings are also in a consistent correlation with the COF graph and values. The B2 coating significantly increased the wear performance of the steel (1.065e-3 mm3/Nm), with a wear rate of 2.79e-5 mm3/Nm.



Fig. 8. The SEM image of wear tracks



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The SEM images of the wear tracks of samples are given in Fig. 8. As can be seen from this figure, the particles broken off owing to oxidation in the substrate have caused deep grooves and abrasive scratches in 1.2714 steel. It is clearly visible of the wear debris settled in the wear track. This appearance in the wear track of the substrate confirms the statements made in accordance with the COF graph in Fig. 4. On the B1 coating, it was determined that the wear track was similar to that of the substrate. Here, too, the ruptured wear particles entered between the friction surfaces and caused abrasive scratches. Besides, the irregularities in the coating layer are evident. As stated above, local abrasive scratches occurred in the edge of the wear track on the B2 coating. Except for the upper corner of the wear track, no significant damage has occurred. By maintaining its protective coating layer, it has made unique contributions to the wear performance of the substrate.

Tab. 4. Chemical composition of wear paths of coatings under high temperature conditions

Coating No.	0	Fe	Ti	Cr	Nb	В	N
B1	31.3	35.3	16.4	12.4	0.9	0.8	2.9
B2	20.7	10.1	15.6	17.3	0.9	7.3	20.7

Table 4 shows the result of the Energy-dispersive spectroscopy (EDS) analysis of the wear tracks of the coatings. As can be seen from the table, it is evident from the "Fe" value that the layer is separated from the surface in the B1 coating layer. It is also obvious that the "O" value in this coating layer is higher than that in the B2 coating. However, when the coating composition values given in Table 2 are compared with the values taken from the wear path, it can be observed that the element content in the coating layer within the wear path has decreased, and that, as a result of friction heat and relative contact as well as owing to oxidation, O2 formation has taken place in the structure. The decrease in composition values is less in the B2 coating. This indicates that the protection of the coating layer is superior.

## 4. CONCLUSIONS

TiCrNb-hBN coatings with various target voltages of boron have been successfully implemented using the closed field unbalanced magnetron sputtering method. The influence of target voltage on microstructure and mechanical and tribological properties of TiCrNb-hBN coatings has been investigated. The results are summarised as follows:

- The TiCrNb-hBN coating layer was deposited on the 1.2714 steel samples in a columnar structure.
- With the increase in the target voltage (600 V to 700 V), the coating structure became more compact and fine-grained. A coating thickness of 1,329 µm was obtained in the B1 coating produced at 600 V. When the voltage value was increased to 700 V, the thickness value decreased to 967 nm.
- In the grain size values obtained from the XRD data, this value is 37.8 nm for the B1 coating and 19.8 nm for the B2 coating.
- While both coating conditions significantly increased the hardness of the substrate, an ultra-hardness value of 24.67 GPa was obtained in the B2 coating layer.
- The adhesion strength values (Lc2) of the B1 and B2 coatings

were determined as 13 N and 85 N, respectively.

- The crack propagation strengths of the B1 and B2 coatings were calculated as 40 N2 and 1650 N2, respectively.
- While the COF value in the B1 coating was similar to that of the substrate, a low COF value of 0.26 was obtained in the B2 coating.
- The friction feature of the steel at high temperature has been improved owing to the h-BN structure in the coatings produced at the voltage of 700 V. The wear performance of the B2 coating is also striking, as is the COF.

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Yaşar Sert: 10 https://orcid.org/0000-0001-7742-0335

Tevfik Küçükömeroğlu: 🔟 https://orcid.org/0000-0002-4392-9966

Hojjat Ghahramazadeh Asl: Dhttps://orcid.org/0000-0002-9078-1933

Levent Kara: Up https://orcid.org/0000-0002-6982-859X



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