

Effect of Increasing Duty Time and Target Voltage on Adhesion and Tribological Properties of B4C Films Coated with HiPIMS at Constant Nitrogen Flow

Ayşenur KELEŞ-DAYAUÇ^{1*}, İhsan EFEOĞLU²

¹Hitit University Engineering Faculty Mechanical Engineering Department, Çorum, Turkey

²Atatürk University, Faculty of Engineering, Erzurum, Turkey

Received: 25/03/2025, Revised: 19/05/2025, Accepted: 29/05/2025, Published: 31/08/2025

Abstract

The mechanical, tribological, and chemical characteristics of nitride-based coatings make them crucial for industrial applications. C-BN films have better qualities than other nitride-based films. However, when ultra-high hardness values have been achieved, the adhesion characteristics of c-BN films produced by magnetic field sputtering with various power sources are often inadequate. High Power Impulse Magnetic Field Sputtering (HiPIMS) power sources have been applied recently to improve coating adhesion. In this study, c-BN films were coated on 4140 steels at increasing duty time and target voltage at constant N₂ flow using HiPIMS technique. SEM and FT-IR analyses were performed to investigate the structural properties of c-BN films; microhardness and scratch tests were performed to investigate their mechanical properties; pin-on-disk tribo test was performed to determine their tribological properties. As a result of the tests, the film thickness increased with increasing duty time and target voltage, while the hardness value and critical load value decreased. In addition, friction coefficient decreased with increasing duty time and target voltage.

Keywords: c-BN, Duty cycle, Target voltage, HiPIMS

Sabit Azot Akışında HiPIMS ile Kaplanmış B4C Filmlerin Adezyon ve Tribolojik Özellikleri Üzerinde Artan Görev Süresi ve Hedef Voltajın Etkisi

Öz

Mekanik, tribolojik ve kimyasal özellikleri nedeniyle nitrür bazlı filmler endüstriyel uygulamalar için çok önemlidir. Nitrür bazlı filmler arasında c-BN filmler çok iyidir. Bununla birlikte, farklı güç kaynakları ile manyetik alan sıçratma kullanılarak sentezlenen c-BN filmlerin adezyon özellikleri, ultra yüksek sertlik değerlerine ulaşıldığında sıklıkla yetersiz kalmaktadır. Kaplamaların adezyonunu artırmak için son zamanlarda Yüksek Güçlü İmpuls Manyetik Alan Sıçratma (HiPIMS) güç kaynakları kullanılmaktadır. Bu çalışmada 4140 çelik üzerine c-BN filmi kaplamak için HiPIMS tekniği kullanılarak sabit N₂ akışında hedef voltaj ve görev süresi artırılmıştır. c-BN filmlerin yapısal özelliklerini incelemek için SEM ve FT-IR analizleri, mekanik özelliklerini incelemek için mikrosertlik ile çizik testleri, tribolojik özelliklerini belirlemek için ise pin-on-disk tribo testleri kullanılmıştır. Testler sonucunda, artan görev süresi ve hedef voltajda film kalınlığı artarken, sertlik değeri ve kritik yük değeri azalmıştır. Ayrıca sürtünme katsayısı artan görev süresi ve hedef gerilim ile azalmıştır.

Anahtar Kelimeler: c-BN, Görev süresi, Hedef gerilim, HiPIMS

*Corresponding Author: aysenurkeles@hitit.edu.tr

Ayşenur KELEŞ-DAYAUÇ, <https://orcid.org/0000-0001-7026-8893>

İhsan EFEOĞLU, <https://orcid.org/0000-0002-9147-2104>

1. Introduction

There is a lot of technical promises for thin film applications in the cubic phase of boron nitride (c-BN). Cubic boron nitride (c-BN) is a superhard material second only to diamond, distinguished by its exceptional thermal stability, chemical inertness, and mechanical robustness. These properties make c-BN coatings particularly attractive for demanding applications such as cutting tools, wear-resistant surfaces, and microelectromechanical systems (MEMS). The mechanical and tribological properties of c-BN films—such as hardness, elastic modulus, adhesion strength, friction coefficient, and wear resistance—are critical parameters influencing their performance in practical applications [1, 2]. These properties are highly sensitive to the film's microstructure, phase composition, internal stress, and interface quality, all of which are influenced by deposition conditions including substrate bias, temperature, gas composition, and ion energy. Because of its characteristics, c-BN is sometimes likened to diamond. While diamonds occur in nature, c-BN is synthetic. All these extraordinary microstructural-mechanical-chemical properties make c-BN indispensable for defense (mechanical and electro-mechanical system components) and industrial (coating of primary cutting tools) applications. Although c-BN shows many valuable properties, its adhesion with most substrate is poor. The reason for the poor adhesion is the high compressive internal stress reaching 25GPa. The first step to improve adhesion is to minimize residual stress. Various methods can be used to eliminate this undesirable effect. Nowadays, intensive efforts are being made to improve the adhesion between coatings and substrates [3].

To improve the adhesion of c-BN film: 1) multilayer coating such as (W, WC, TiAlN, Ti(B,C)) after the interlayer such as B₄C or BCN, 2) h-BN/c-BN multilayer coating [4]. In this direction, Lattemann et al. (2006) found that by increasing the voltage applied to the substrate to -700V, the basal planes in the h-BN crystal lattice grew parallel to the surface of the substrate and a -9GPa decrease in stress occurred [5].

PVD and CVD techniques are widely used to synthesize c-BN films. The most widely used PVD method is magnetic field sputtering. Among various deposition methods, magnetron sputtering has emerged as a promising technique for fabricating high-quality c-BN thin films due to its controllability, scalability, and compatibility with industrial processes [1, 2, 6]. However, achieving the metastable c-BN phase is challenging, typically requiring ion-assisted deposition or high-energy ion bombardment during sputtering to stabilize the cubic phase over the thermodynamically favored hexagonal phase. Several types of power sources are used in the magnetron sputtering technique. These are dc, rf, pulsed dc, and HiPIMS (High Power Impulse Magnetron Sputtering) [7]. Compared to all power sources, HiPIMS, which has been used for the last 10 years, has a significant technological advantage since films with superior properties can be coated [8].

Therefore, in this study, the effect of increasing duty time and target voltage using constant N₂ flow was investigated to improve the adhesion and tribological properties of c-BN film.

2. Material and Methods

Silicon was used as the substrate for the microstructural analysis of c-BN films in the present study. 4140 tempered steels with a diameter of 30 mm and a thickness of 5 mm was chosen as the substrate in order to analyze the mechanical and tribological properties of the films. The substrates were ultrasonically bath cleaned with ethyl alcohol, polished to Ra≈0.06 μm, and then etched in Nital solution. The HiPIMS power supply was used to coat the c-BN films on the substrates. Table 1 provides the test parameter values for the coatings applied using the HiPIMS technique. One Ti, one TiB₂, and two B₄C targets were employed in the procedure. Prior to coating, contaminants were removed by 20 minutes of ion cleaning at -850V. Prior to c-BN film, Ti, TiN, TiB₂, and TiBN interlayers were composite-graded to improve the film's adherence to the substrate. The constant parameters are given in the previous article [9].

Table 1. The test parameters

	N ₂ content [MKS]	Duty time (μs)	B ₄ C target voltage (V)	Duty cycle (%)
D1	4,5	80	650	4
D2	4,5	90	750	4,5

FT-IR (Fourier Transform Infrared) Spectroscopy using a Bruker VERTEX 70v was used to characterize the silicon-coated c-BN film in order to percentage amount of film compression. With a resolution of 4 cm⁻¹, the FT-IR test was conducted in the wavenumber range of 500–4000 cm⁻¹. Using a scanning electron microscope (FeI Quanta FEG-450 SEM), the microstructure and thickness of the c-BN layer deposited on the silicon substrate were investigated.

To determine the critical load, the adhesion test was performed using a Rockwell-C diamond tip (tip radius 200 microns) at a feed rate of 10 mm/min and under increasing loading up to 100 N/min. The microhardness values of the c-BN films were determined with a Buehler Micromet 2001 microhardness tester using a Knoop tip. Microhardness measurements were performed under 10gf load and 15s indentation time. Wear tests were performed under air atmosphere using CSM tribo-tester. Wear tests were carried out with 6.25 mm diameter alumina (Al₂O₃) spheres at the sliding contact under a load of 1 N at a speed of 10 cm/s and a travel time of 1400 laps.

3. Results and Discussion

Surface and cross-sectional images of c-BN films are given in Figure 1. Thanks to HiPIMS technology, Zone 3 “fully dense” structure was obtained in two films coated at high energy level [10]. The film thickness increased with increasing duty cycle and target voltage.

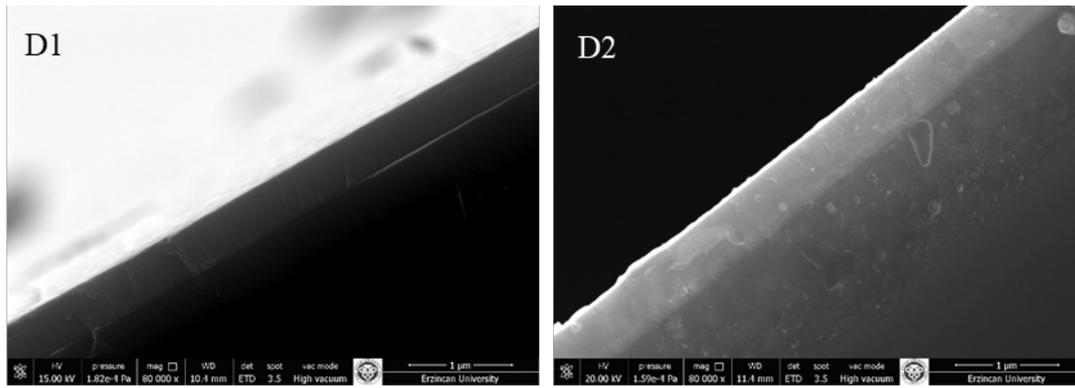


Figure 1. Cross section SEM images

The relative intensities of the c-BN (TO) peak and the h-BN peak at 1400 cm^{-1} may be used to directly measure the quantity of c-BN phase. Figure 2 shows the FT-IR graphs of c-BN films coated with a HiPIMS power source. The contents of c-BN in parameters D1 and D2 are 55 and 52, respectively.

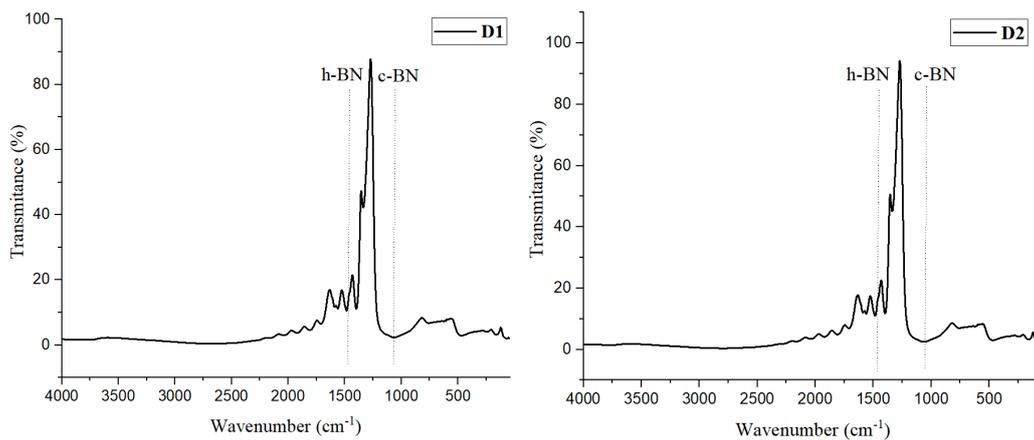


Figure 2. FT-IR graphs

The ion flux increases with increasing target voltage. Moreover, increasing target voltage also increases the ion-boron flux ratio. It can be interpreted that as the ion-boron flux ratio decreases, the h-BN interlayer increases, and this flux ratio is one of the parameters determining the nucleation and growth of c-BN [9].

The coefficient of friction -normal load graphs and light microscope images obtained as a result of the scratch test to determine the adhesion properties of c-BN films are given in Figure 3. No edge damage was observed in the D1 coating until 20N. In D2 coating, crack initiation and adhesive cracks occurred at 10N. Critical load values were determined as 38N and 36N for D1 and D2, respectively. Also, the hardness of the D1 coating was 2792 Hv_K and the D2 coating was 2585 Hv_K . Since D1 is harder, it is more resistant to plastic deformation. Therefore, the critical load value is higher [11].

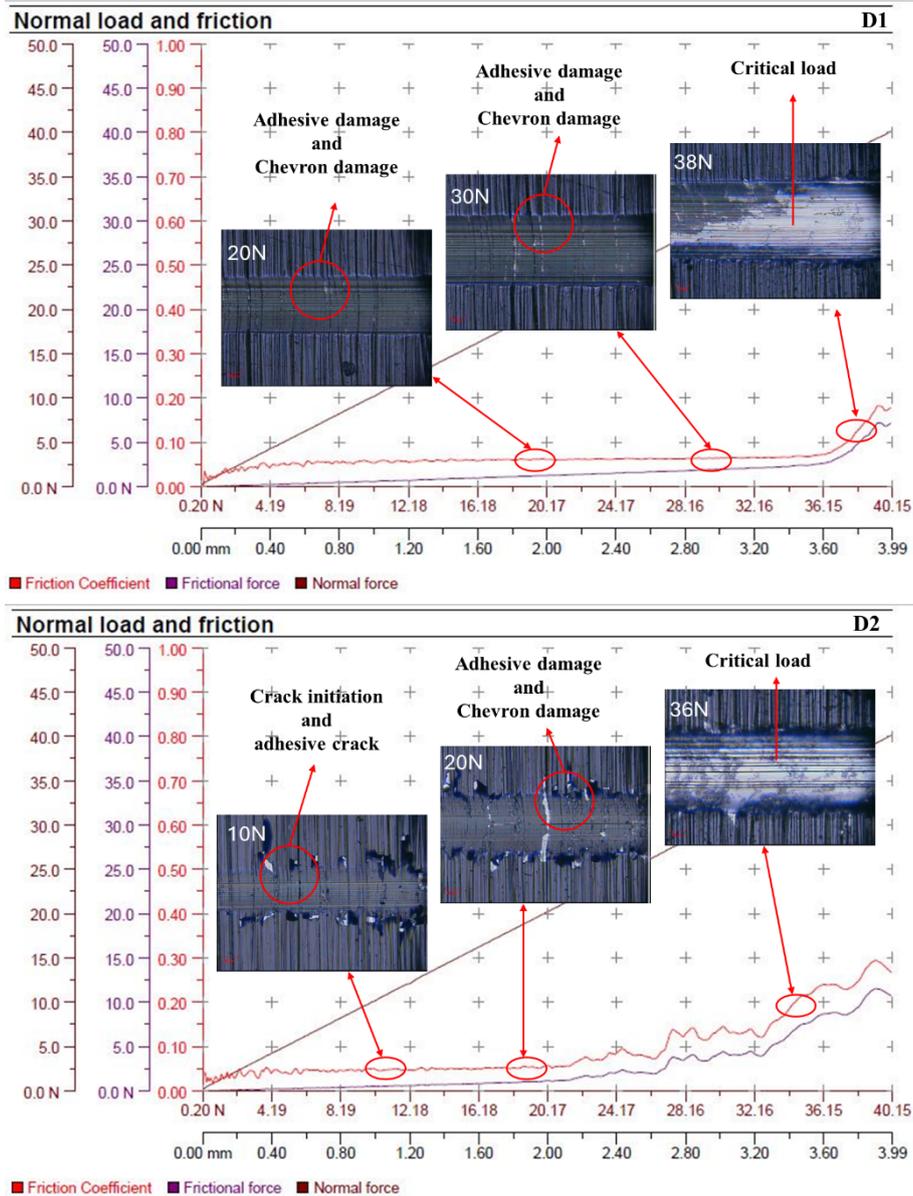


Figure 3. Friction coefficient - normal load graphs and light microscope images obtained as a result of scratch (adhesion) test

The friction coefficient lap graph is given in Figure 4. Friction coefficients were obtained as 0.191 and 0.295 for D1 and D2, respectively. In addition, wear rates were obtained as $2.781 \times 10^{-6} \text{ mm}^3/\text{Nm}$ and $3.459 \times 10^{-6} \text{ mm}^3/\text{Nm}$ for D1 and D2, respectively. The D1 coating with higher hardness has a lower coefficient of friction [12, 13].

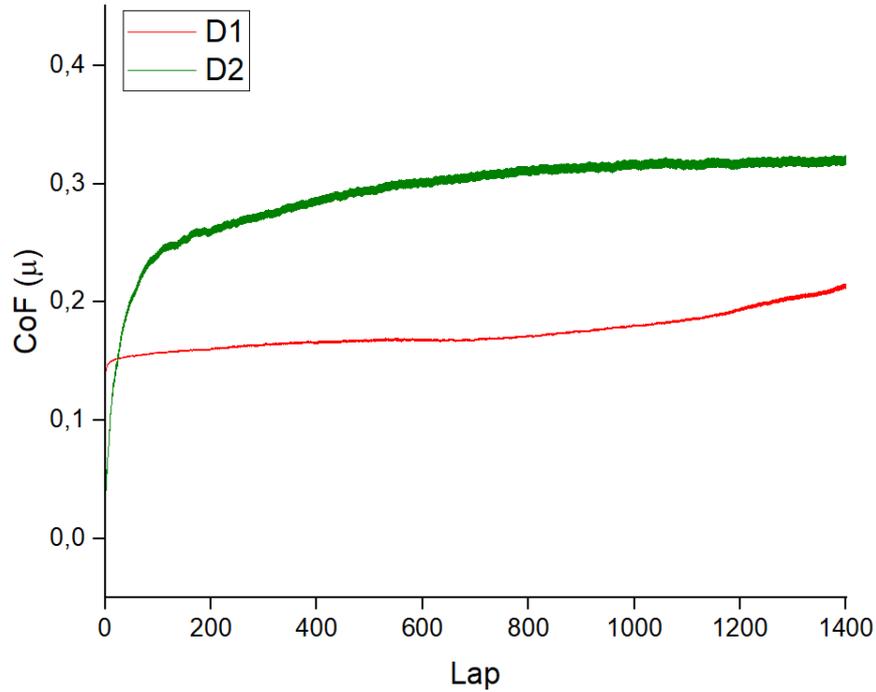


Figure 4. The CoF-Lap graphs of c-BN films

Figure 5 shows SEM and 3D profilometer pictures of the worn c-BN films. As the friction coefficient value rose, the degree of wear on c-BN films increased as well.

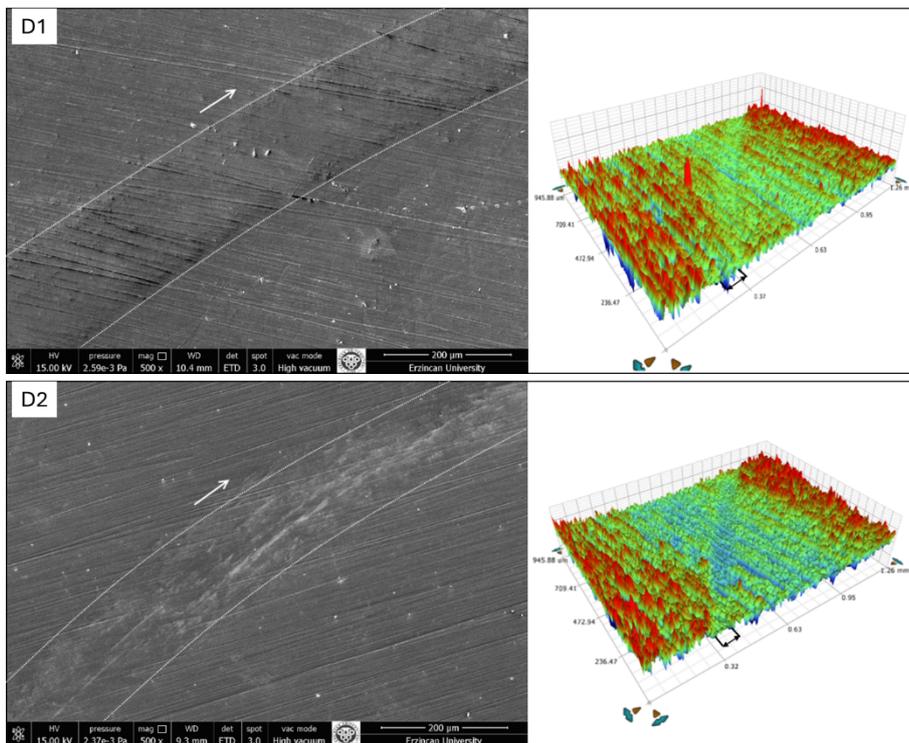


Figure 5. SEM images and 3D profilometer images of worn c-BN films

4. Conclusion

In this study, c-BN films were successfully coated on 4140 steels at increasing duty time and target voltage at constant N₂ flow using HiPIMS technique. The following lists the results that this article provided.

- Structural analysis showed that the c-BN films exhibited a fully dense structure.
- Increasing duty cycle and target voltage (D2) resulted in increased film thickness and decreased c-BN content.
- Hardness value and adhesion increased with increasing amount of c-BN.
- Increasing hardness value (2792 Hv_K) also caused a decrease in friction coefficient. Also, increasing the duty time and target stress led to an increase in hardness and a decrease in the coefficient of friction. The lowest CoF of 0.192 was achieved.

Ethics in Publishing

There are no ethical issues regarding the publication of this study.

Author Contributions

Ayşenur KELEŞ-DAYAUÇ: Writing – review & editing, Visualization, Methodology, Investigation, Conceptualization

İhsan EFEOĞLU: Writing – review & editing, Methodology, Conceptualization, Supervision

Acknowledgements

This research is part of the TUBITAK (The Scientific and Technical Research Council of Turkey) project was supported by Grant no: MAG-215M213–215M217-215M218. The authors would like to thank TUBITAK for funding the project.

References

- [1] Wang, H., Meng, D., Wu, Z., Kang, J., She, D., Qin, W., & Yue, W. (2024). Tribological Behavior of Polycrystalline Cubic Boron Nitride Sliding against WC-Co Cemented Carbide in Vacuum Conditions. *Journal of Materials Engineering and Performance*, 33(4), 1975-1984.
- [2] Yang, F., Wu, Z., Meng, D., Qin, W., & She, D. (2022). Tribological Behaviors of Polycrystalline Cubic Boron Nitride Sliding against Bearing Steel in Vacuum Conditions. *Coatings*, 12(5), 693.
- [3] Sell, K., Holleck, H., Leiste, H., Stuber, M., Ulrich, S., & Yë, J. Y. (2002). Synthesis and characterization of cubic boron nitride films-investigations of growth and annealing processes. *Diamond and Related Materials*, 11(3-6), 1272-1275.

- [4] Soltani, A., Talbi, A., Mortetb, V., Benmoussa, A., Zhang, W. J., Gerbedoen, J. C., De Jaeger, J. C., et al. (2010). Diamond and cubic boron nitride: Properties, growth and applications. *AIP Conference Proceedings*, 1292, 191-196.
- [5] Lattemann, M., Helmersson, U., & Greene, J. E. (2010). Fully dense, non-faceted 111-textured high power impulse magnetron sputtering TiN films grown in the absence of substrate heating and bias. *Thin Solid Films*, 518(21), 5978-5980.
- [6] Wani, S. M., Ahmad, B., & Saleem, S. S. (2023). Nanomechanical and Nanotribological Characterization of Co-BN Nano-composite Coating on Al-Si Substrate. *Journal of Materials Engineering and Performance*, 180, 108281.
- [7] Semenov, V. A., Rabotkin, S. V., Grenadyorov, A. S., Solovyev, A. A., Oskirko, V. O., Zakharov, A. N., & Shipilova, A. V. (2024). Effect of Reactive Magnetron Sputtering Modes (DCMS, HIPIMS, and DC + HIPIMS) on the Properties of Copper Oxide Films. *Physics of Metals and Metallography*, 25, 1593-1600.
- [8] Raman, P., Cheng, M., Weberski, J., Xu, W., Houlahan, T., Rivera, J., Su, R., et al. (2018). Magnetic field influence on ionization zones in high-power impulse Magnetron Sputtering. *Vacuum*, 156, 9-19.
- [9] Keleş, A., & Efeoğlu, İ. (2019). Effect of Target Voltage on Tribological and Adhesive Properties of c-BN Films Coated with HiPIMS. 2019 IEEE Regional Symposium on Micro and Nanoelectronics (RSM).
- [10] Mukherjee, S., & Gall, D. (2013). Structure zone model for extreme shadowing conditions. *Thin Solid Films*, 527, 158-163.
- [11] Sert, Y., Küçükömeroğlu, T., Ghahramanzadeh Asl, H., & Efeoğlu, İ. (2021). Hardness, Adhesion, and Wear Performance of Duplex Treatment Coatings of Nitride/TiAlZrN with Different Zr Target Currents. *Journal of Materials Engineering and Performance*, 30(1), 638-651.
- [12] Grenadyorov, A. S., Solovyev, A. A., Oskomov, K. V., & Zhulkov, M. O. (2021). Dependence of Mechanical and Tribological Properties of a-C:H:SiO_x Films on the Bias Voltage Amplitude of the Substrate. *Technical Physics*, 66(10), 1111-1117.
- [13] Kvatchadze, V., Bairamashvili, I., Mikeladze, A., Gventsadze, D., Mestvirishvili, Z., & Chkhartishvili, L. (2023). Boron carbide based ceramics for dry friction units. *Solid State Sciences*, 142, 107244.