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Research article

Materials Science

TRIBOLOGICAL AND MECHANICAL PERFORMANCE OF HYBRID TISIN/DLC COATINGS DEPOSITED USING THE FILTERED CATHODIC ARC TECHNIQUE

使用过滤阴极电弧技术沉积的混合钛辛/DLC涂层的摩擦学和机械性能

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Abstract

This study focuses on fabricating and enhancing the mechanical and tribological properties of novel hybrid TiSiN/DLC coatings deposited on tungsten carbide cutting tools using the filtered cathodic arc technique. The methodology involves incorporating DLC on the top surface and TiSiN as an interlayer, with a titanium interlayer used to ensure good adhesion of the coatings. XRD analyses indicate a preferred orientation of (111) and (200) with a high hardness phase. Raman spectroscopy confirms the presence of DLC nature through characteristic G and D bands. SEM and AFM analyses revealed reduced surface roughness and enhanced structural properties characterized by smooth and dense structures. The TiSiN/DLC coatings demonstrate a significant increase in hardness reaching up to 36.2 GPa and an elastic modulus of 320 GPa, along with excellent adhesion strength compared with individual DLC coatings. Furthermore, these hybrid coatings exhibit superior tribological properties, including remarkable wear resistance and a low coefficient of friction, surpassing TiSiN and DLC coatings. This research highlights the promising potential of TiSiN/DLC hybrid coatings for various industrial applications, such as cutting tools, automotive components, and medical implants, where superhard coatings and robust tribological performance are crucial. The comprehensive evaluation and characterization of these hybrid coatings, tailored specifically for tungsten carbide cutting tools, underscores their superiority over conventional coatings.

Keywords: tribological performance, titanium silicon nitride coatings, diamond-like carbon coatings, titanium silicon nitride/diamond-like carbon coatings, tungsten carbide, hardness, wear, friction

摘要 本研究的重点是使用过滤阴极电弧技术沉积在碳化钨切削刀具上的新型混合氮化钛/DLC涂层并增强其机械和摩擦学性能。该方法包括在顶表面加入DLC，将氮化钛作为中间层，并使用钛中间层来确保涂层具有良好的附着力。X射线衍射分析表明（111）和（200）具有高硬度相的择优取向。拉曼光谱通过特征G和D波段证实了DLC性质的存在。扫描电镜和原子力显微镜分析表明，表面粗糙度降低，结构性能增强，其特点是结构光滑致密。与单独的DLC涂层相比，氮化钛/DLC涂层的硬度显着提高，达到36.2 GPa，弹性模量达到320 GPa，并且具有优异的附着强度。此外，这些混合涂层表现出优异的摩擦学性能，包括卓越的耐磨性和低摩擦系数，超过了氮化钛和DLC涂层。这项研究强调了氮化钛/DLC混合涂层在各种工业应用中的巨大潜力，例如切削工具、汽车零部件和医疗植入物，其中超硬涂层和强大的摩擦学性能至关重要。这些专为碳化钨切削刀具定制的混合涂层的综合评估和表征强调了它们相对于传统涂层的优越性。

关键词:摩擦学性能、氮化硅钛涂层、类金刚石碳涂层、氮化钛硅/类金刚石碳涂层、碳化钨、硬度、磨损、摩擦

I. INTRODUCTION

Tribological properties are essential factors in evaluating the suitability of these coatings for various industrial applications. Understanding their tribological performance is crucial because it directly influences their practical utility, especially in cutting tools, automotive components, and medical implants. The use of superhard coatings is not only limited to enhancing hardness but also extends to increasing wear resistance, reducing friction, and ensuring long-term durability in various industrial applications. These coatings, owing to their mechanical properties, significantly contribute to the improved performance and lifespan of critical components through multiple sectors [1-3].

With the advancement of research in the field of surface engineering, the focus has shifted toward achieving the delicate balance between hardness and toughness in coatings. The quest for superhard coatings with optimal tribological properties, including exceptional wear resistance and low friction, has stimulated numerous investigations and innovations in coating technologies. The family of Ti-based coatings, such as titanium nitride (TiN) [4-6], titanium aluminum nitride (TiAlN) [7], and titanium chromium nitride (TiCrN) [4, 5], has long held standing in surface engineering, contributing a diverse range of choices for enhanced functionality. These coatings, including titanium silicon nitride (TiSiN) [2], diamond-like carbon (DLC) [8, 9], and novel hybrid coatings [10-12], are indicated by their outstanding properties, including exceptional hardness, making them of great interest in various industrial applications.

This work explores the fabrication and characterization of tribological and mechanical properties, particularly wear resistance and low friction, of these superhard coatings, i.e., TiSiN, DLC, and TiSiN/DLC thin coatings.

II. MATERIALS AND METHODS

A. Substrate Material and Preparation

The substrate material used in this study was tungsten carbide (WC-Co). Tungsten carbide is a widely used material in the production of cutting tools and various industrial applications. A commercial-grade RSK15X tungsten carbide substrate with dimensions of 1.2 x 1.2 x 0.3 cm was chosen for this research. The chemical composition of the RSK15X tungsten carbide substrate, as determined by energy-dispersive X-ray spectroscopy (EDS), is as follows: W (tungsten) 78.8 wt.%, C (carbon) 7.8 wt.%, Co (cobalt) 11.1 wt.%, Si (silicon) 1.7 wt.%, and Cr (chromium) 0.6 wt.%. The specimen surfaces were thoroughly ground and polished to ensure uniformity and cleanliness. Before entering the deposition chamber, the WC-Co substrates also underwent ultrasonic cleaning with acetone and ethanol to eliminate any contaminants.

B. Coating Deposition Process

All coatings' deposition in this paper was performed using a filtered cathodic arc system supplied by Diacoat Technologies Company.

1) TiSiN Coating Deposition

TiSiN coatings were deposited on the WC-Co substrates using a multi-cathodic arc system. The deposition targets contained pure titanium (99.95% purity) and a TiSi alloy (containing 80 at% titanium and 20 at% silicon) with a diameter of 10 cm. In the deposition chamber, the Ti and TiSi cathodes were positioned on opposite sides. The substrate coating temperature was maintained at 480°C. The deposition process began with a 5-min deposition of pure titanium. During this phase, the substrate bias voltage was set at -200 V, and the Ti cathode current ranged from 120 to 140 A. Subsequently, the top TiSiN layer was deposited for 90 min. The TiSi cathode arc current varied from 120 to 140 A, and the bias voltage varied from 8 to 130 A. Throughout all deposition steps, to maintain homogenous coatings, the substrate was constantly rotated at a consistent speed of 20 revolutions per minute (rpm).

2) Diamond-Like Carbon (DLC) Coating Deposition

For DLC coatings, a titanium (Ti) interlayer was first sputtered onto the substrate using DC magnetron sputtering as a glue layer. The substrate temperature was set at 170°C, and the distance between the substrate and the Ti target was maintained at 80 mm. The vacuum chamber was evacuated to a base pressure of 3.0 x 10^{-3} Pa, and the substrates were subjected to a 20-min argon ion etching process to remove possible contaminations. During the DLC deposition process, the cathodes operated with a DC arc current ranging from 60 to 100 A. Three different DC bias voltages were applied in the range of -1500 to -1700 V. The deposition time for the DLC coatings was 200 min.

3) TiSiN/DLC Coating Deposition

In the case of the TiSiN/DLC hybrid coatings, a titanium (Ti) interlayer was deposited first, followed by the TiSiN and DLC layers. A schematic of these studied coatings is shown in Figure 1.

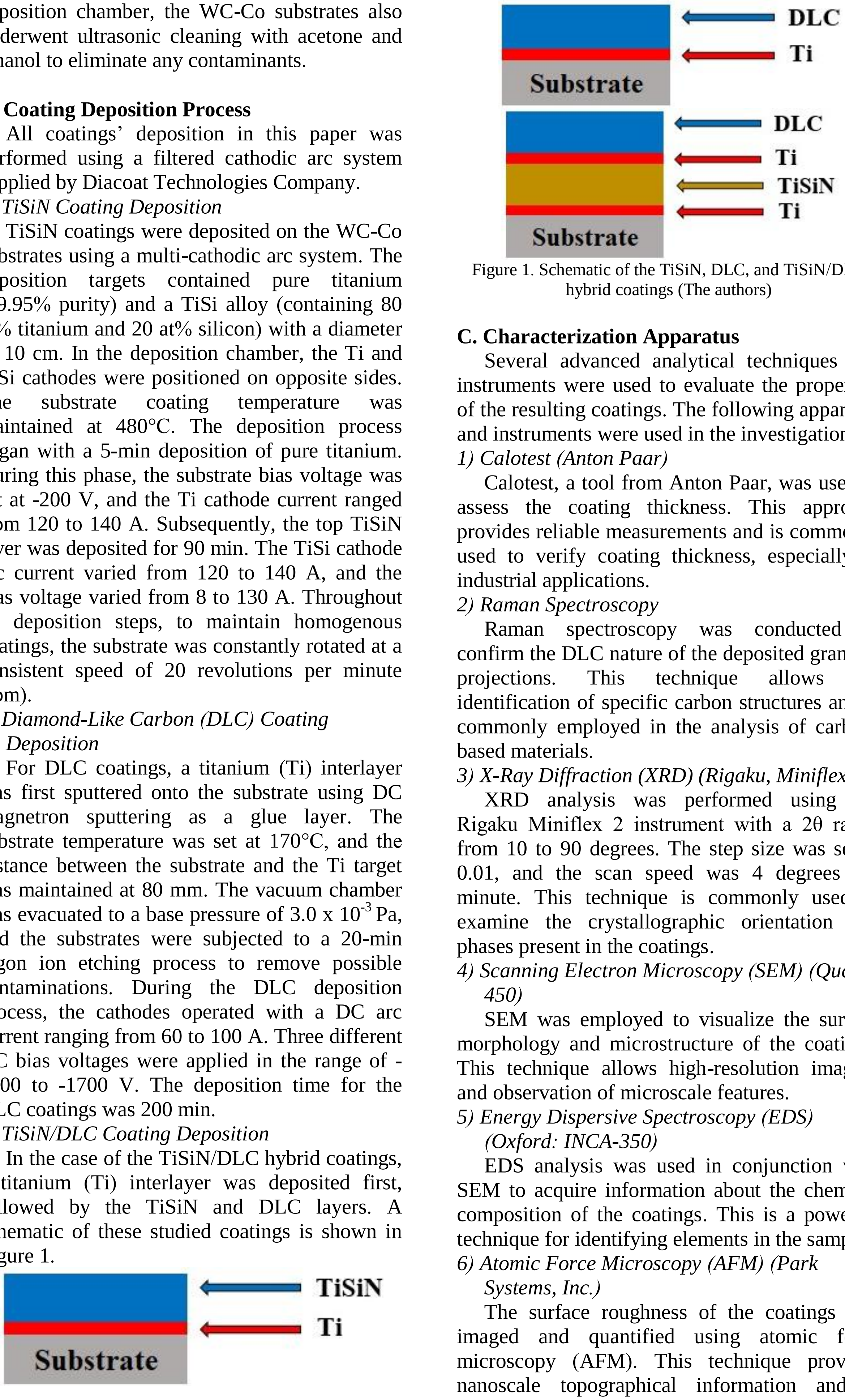


Figure 1. Schematic of the TiSiN, DLC, and TiSiN/DLC hybrid coatings (The authors)

C. Characterization Apparatus

Several advanced analytical techniques and instruments were used to evaluate the properties of the resulting coatings. The following apparatus and instruments were used in the investigation:

1) Calotest (Anton Paar)

Calotest, a tool from Anton Paar, was used to assess the coating thickness. This approach provides reliable measurements and is commonly used to verify coating thickness, especially in industrial applications.

2) Raman Spectroscopy

Raman spectroscopy was conducted to confirm the DLC nature of the deposited granular projections. This technique allows the identification of specific carbon structures and is commonly employed in the analysis of carbon-based materials.

3) X-Ray Diffraction (XRD) (Rigaku, Miniflex 2)

XRD analysis was performed using the Rigaku Miniflex 2 instrument with a 2θ range from 10 to 90 degrees. The step size was set at 0.01, and the scan speed was 4 degrees per minute. This technique is commonly used to examine the crystallographic orientation and phases present in the coatings.

4) Scanning Electron Microscopy (SEM) (Quanta 450)

SEM was employed to visualize the surface morphology and microstructure of the coatings. This technique allows high-resolution imaging and observation of microscale features.

5) Energy Dispersive Spectroscopy (EDS) (Oxford: INCA-350)

EDS analysis was used in conjunction with SEM to acquire information about the chemical composition of the coatings. This is a powerful technique for identifying elements in the sample.

6) Atomic Force Microscopy (AFM) (Park Systems, Inc.)

The surface roughness of the coatings was imaged and quantified using atomic force microscopy (AFM). This technique provides nanoscale topographical information and is valuable for assessing surface appearance and quality.

7) Microhardness Tester (Fischer: HM2000)

The microhardness of the coatings was assessed using the Fischer HM2000 microhardness tester. This technique provides information about the coatings' resistance to indentation and is crucial for understanding their mechanical properties.

8) Micro-Scratch Tester (Fischer: HM2000)

A micro-scratch tester, also from Fischer's HM2000 series, was used to evaluate the coating scratch resistance and adhesion characteristics. It helps determine the coating' durability and ability to withstand wear.

9) Tribometer (Ball-on-Disk, Anton Paar's TRB3)

Tribological performance, including wear characteristics and coefficient of friction (COF), was assessed using a ball-on-disk tribometer. An alumina Al_2O_3 ball was used in this apparatus to investigate wear behavior under various conditions, providing insights into the coating performance under sliding contact conditions.

III. RESULTS AND DISCUSSION

A. Structure Characterization

1) XRD and Raman Spectrum Analysis

X-ray diffraction (XRD) analysis revealed that the TiSiN, DLC, and TiSiN/DLC coatings exhibited broad diffraction peaks corresponding to the TiSiN, DLC, and TiSiN/DLC phases, irrespective of the deposition temperature (Figure 2). Small peaks of Ti were also observed, as expected, originating from the Ti interlayer. The presence of a single peak suggests a fiber texture with preferred (111), (110), and (200) orientations in the TiSiN and TiSiN/DLC coatings. Most TiSiN coatings reported in the literature exhibit hexagonal phase patterns with preferred (111) and (200) orientations. These (111) and (200) oriented coatings often yield the highest hardness due to the higher packing factor on the (111) and (200) planes.

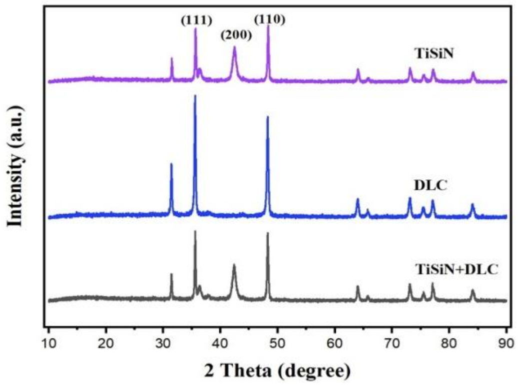


Figure 2. XRD patterns generated from the resulting coatings (The authors)

Raman spectroscopy patterns are displayed in Figure 3, with characteristic peaks near 1540 cm^{-1} (G band) and 1330 cm^{-1} (D band). Raman analysis confirmed the DLC nature of the coatings. These peaks indicate graphitic and disordered carbon structures commonly associated with DLC materials.

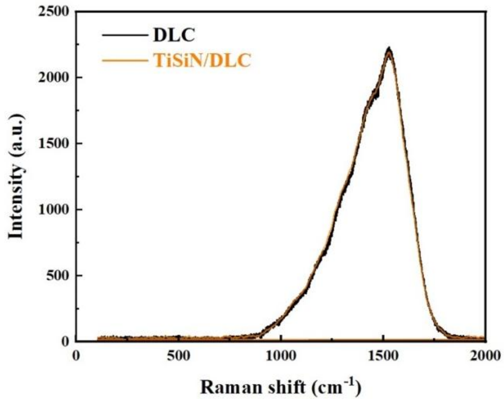
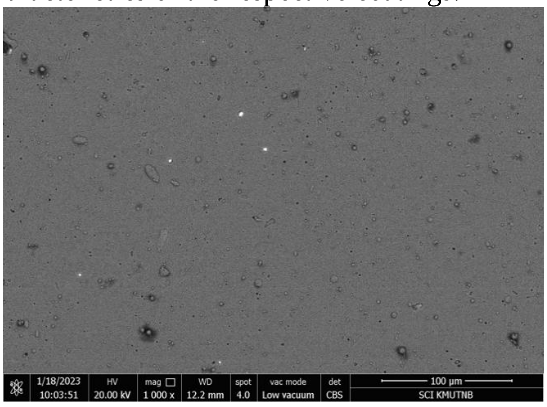


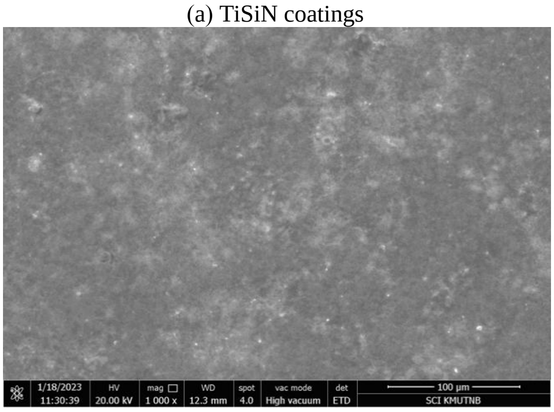
Figure 3. Patterns of Raman spectroscopy of the resulting coatings (The authors)

2) SEM Analysis

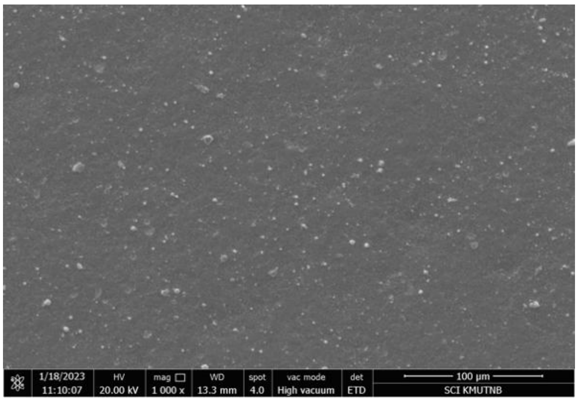
SEM analysis (Figure 4) revealed that the surface morphologies of the TiSiN coatings exhibited several macroparticles. In contrast, the surface morphologies of the DLC coatings appeared much smoother and dense with less porosity compared to the TiSiN coatings. This visual evidence reinforces the structural characteristics of the respective coatings.



(a) TiSiN coatings



(b) DLC coatings

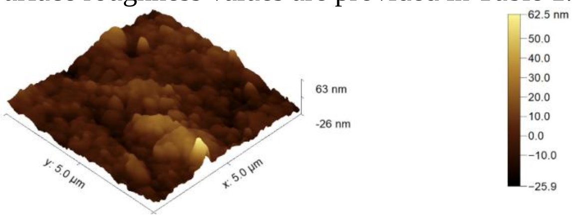


(c) TiSiN/DLC coatings

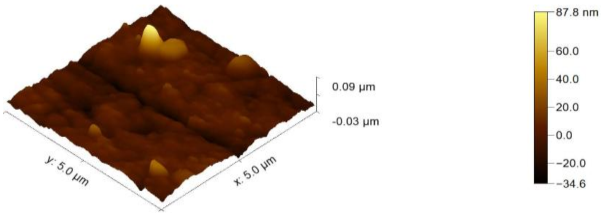
Figure 4. SEM images of the sample surfaces of (a) TiSiN, (b) DLC, and (c) TiSiN/DLC coatings (The authors)

3) Surface Roughness

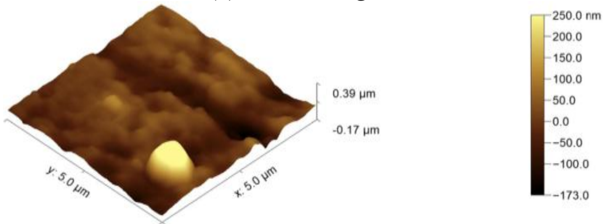
Surface roughness was determined using AFM analysis shown in Figure 5. Although the TiSiN/DLC hybrid coatings displayed a higher surface roughness value, the AFM image showed a denser morphology. This denseness contributed to the super high hardness of the TiSiN/DLC coatings compared to the DLC coatings. The surface roughness values are provided in Table 1.



(a) TiSiN coatings



(b) DLC coatings



(c) TiSiN/DLC coatings

Figure 5. Surface roughness images of the sample surfaces of (a) TiSiN, (b) DLC, and (c) TiSiN/DLC coatings (The authors)

Table 1.
Surface roughness of the resulting coatings (The authors)

Coatings		Surface Roughness	
		Mean (nm)	RMS (nm)
1	TiSiN	35.96	55.45
2	DLC	5.65	7.75
3	TiSiN/DLC	40.48	64.48

B. Coating Property Characterization

1) Calotest

Calotest analysis showed that the DLC coatings were very thin, with a thickness of approximately 1 μm. In contrast, the novel TiSiN/DLC hybrid coatings had a thickness of 1.77 μm. Additional mechanical properties and thickness values are presented in Table 2.

Table 2.
Thickness and mechanical properties of the resulting coatings (The authors)

Coatings		Thickness (μm)	Hardness (GPa)	Elastic Modulus (GPa)	Critical Load (mN)
1	TiSiN	1.56	40.9	362	80.66
2	DLC	1.03	36.0	333	41.27
3	TiSiN/DLC	1.77	36.2	320	55.8

2) Nanoindentation Test

A nanoindentation test with an applied load of 8 mN provided load-displacement curves shown in Figure 6. The TiSiN coatings demonstrated exceptional hardness exceeding 40 GPa with an elastic modulus of 362 GPa. In contrast, the TiSiN/DLC hybrid coatings showed an improvement in hardness, further enhancing its hardness, reaching 36.2 GPa, with an elastic modulus of 320 GPa. This makes TiSiN/DLC hybrid coatings a promising alternative for future applications. The hardness and modulus values are detailed in Figure 7 and Table 2.

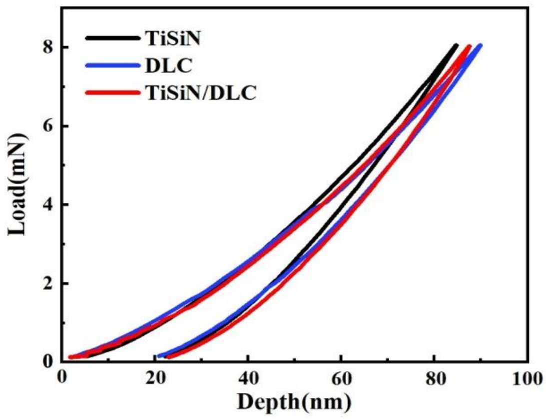


Figure 6. Load-displacement curves of the resultant coatings (The authors)

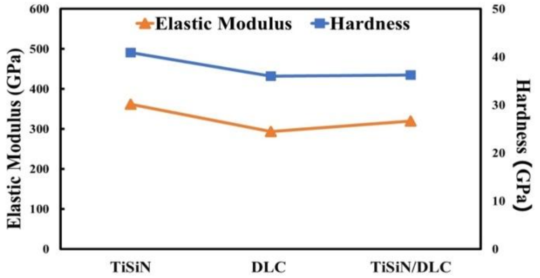
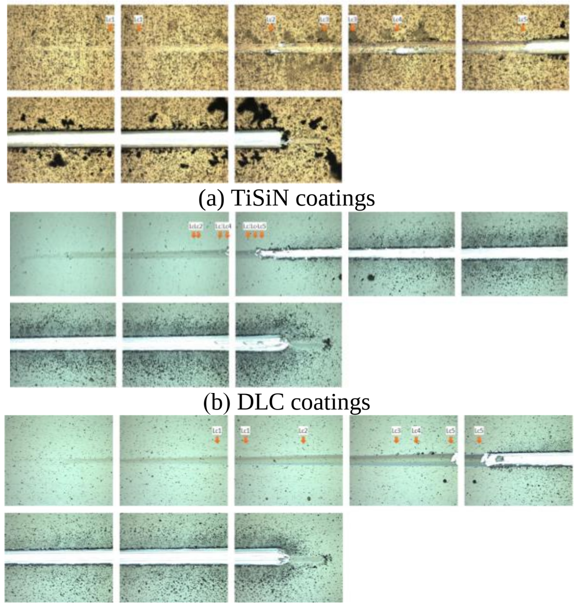


Figure 7. Hardness and modulus of the resultant coatings (The authors)

3) Microscratch Test

Optical images of the scratch tests are shown in Figure 8. The TiSiN coatings exhibited excellent scratch resistance, with a critical load (Lc) exceeding 80 mN. Furthermore, although the Lc values of the DLC and TiSiN/DLC hybrid coatings are lower than those of the TiSiN coatings, the TiSiN/DLC hybrid coatings demonstrated improved resistance, achieving a higher Lc than the DLC coatings. All Lc values are summarized in Table 2.



(a) TiSiN coatings

(b) DLC coatings

(c) TiSiN/DLC coatings

Figure 8. Optical images of the scratch test of the resultant coatings (The authors)

4) Tribometer Test

Tribometer tests were conducted with applied loads of 5 and 10 N and a sliding distance of 200 m. It was observed that higher loads resulted in higher wear rates. The TiSiN/DLC hybrid coatings confirmed the lowest wear rate compared to the other coatings, with wear rates of 2 x 10^{-7} mm³/N·m at 5 N and 3.5 x 10^{-7} mm³/N·m at 10 N. The TiSiN/DLC coatings presented excellent wear resistance, making them promising protective coatings for several tool and industrial applications.

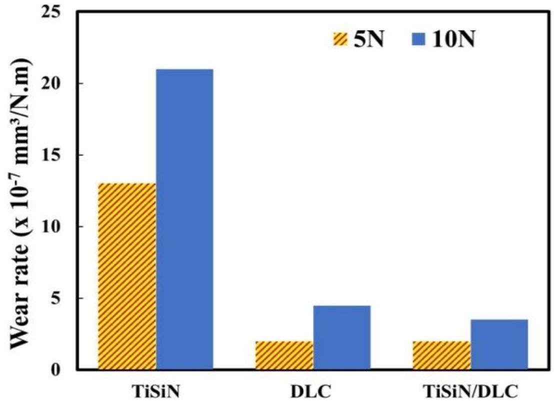


Figure 9. Wear rates of the resulting coatings (The authors)

According to the friction, the DLC and TiSiN/DLC hybrid coatings demonstrated a lower coefficient of friction (COF) than the TiSiN coatings. This lower COF is due to the smoother morphology, as confirmed by SEM and AFM. The TiSiN/DLC hybrid coatings demonstrated the lowest COF, with values of around 0.05 at 5 and 10 N, whereas the TiSiN coatings showed a COF of about 0.2. The average coefficients of friction of the resultant coatings are illustrated in Figure 10.

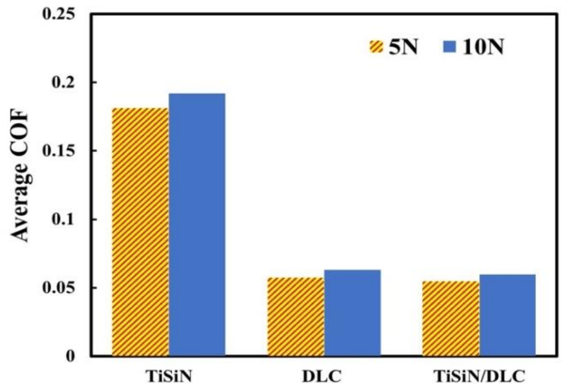


Figure 10. Average coefficients of friction of the resulting coatings (The authors)

IV. CONCLUSION

This study underscores the success achieved in fabricating and enhancing the mechanical and tribological properties of novel hybrid TiSiN/DLC coatings. Due to using DLC on the top surface and TiSiN as an interlayer, these coatings have demonstrated reduced surface roughness, increased hardness, and superior adhesion strength compared with individual TiSiN or DLC coatings. Notably, the TiSiN/DLC hybrid coatings exhibit remarkable wear resistance and a low coefficient of friction, indicating their potential for various industrial applications where superhard coatings and robust tribological performance are crucial.

The scientific novelty of this study lies in its comprehensive evaluation and characterization of TiSiN/DLC hybrid coatings tailored specifically for tungsten carbide cutting tools. Beyond highlighting their exceptional mechanical and tribological properties, this study establishes their superiority over conventional coatings. These hybrid coatings offer a unique combination of enhanced hardness, mechanical strength, wear resistance, and ultra-low friction coefficient, promising significant improvements in the durability and functionality of critical components across industries such as cutting tools, automotive applications, and medical implants.

However, despite these promising results, certain limitations must be acknowledged. The study primarily relies on laboratory-scale evaluations, necessitating further real-world testing under diverse operational conditions to validate the coatings' performance in practical industrial settings. In addition, assessing the long-term durability and stability of these coatings is imperative to ensure sustained effectiveness over extended usage periods. Future research endeavors could focus on optimizing coating thickness, exploring variations in composition, and evaluating the scalability of the fabrication process for industrial-scale implementation. Collaborative efforts with industry partners can provide valuable insights into specific application requirements, thus refining these coatings to meet customized industrial needs and further advancing the field of coating technology and material science.

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DECLARATIONS

Author Contributions

Conceptualization, P.S.; methodology, P.S.; validation, N.V.; formal analysis, N.P.; investigation, N.V.; resources, N.V.; data curation, N.P.; writing—original draft preparation, N.V.; writing—review and editing, N.P.; visualization, N.P.; supervision, P.S.; project administration, P.S. All authors have read and agreed to the published version of the manuscript.

Data Availability Statement

The data presented in this study are available in this article.

Conflicts of Interest

The authors declare no conflict of interest.

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