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Mechanical and tribological behavior of multilayer and monolayer TiN-based coatings

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Abstract. This study investigates the mechanical and tribological properties of monolayer TiN coatings and multilayer TiN/TiCN coatings deposited via direct current magnetron sputtering onto titanium substrates (VT1-0). The coatings were characterized by microstructure, nanohardness, elastic modulus, and tribological performance under lubricated friction conditions. Scanning electron microscopy (SEM) revealed that the coatings exhibit a uniform microstructure without visible defects and a typical columnar growth morphology. Nanoindentation tests demonstrated that the multilayer TiN/TiCN coatings possess enhanced hardness (up to 23.5 GPa) and elastic modulus (191 GPa) compared to the monolayer TiN, attributed to interlayer strengthening effects and redistribution of residual stresses. Tribological tests using a ball-on-disk configuration under lubricated conditions showed that the multilayer coatings exhibit a significantly lower coefficient of friction (0.10–0.13) and improved wear resistance compared to the TiN coating. This behavior is associated with TiCN layers, which reduce interfacial adhesion, promote uniform load distribution, and facilitates the formation of a protective tribofilm. The results confirm that the TiN/TiCN multilayer coatings offer superior mechanical and tribological properties, making them promising candidates for engineering components operating under friction and wear conditions.

Keywords: *multilayer coating, magnetron sputtering, coefficient of friction, nanohardness, wear resistance.*

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1. Introduction

Recent advances in materials science and surface engineering have increasingly focused on the development of high-performance protective coatings to reduce wear and friction in tribological components of machines and mechanisms [1-3]. Among such coatings, titanium nitride (TiN) and titanium carbonitride (TiCN) compounds are fascinating due to their high microhardness, chemical inertness, and thermal stability. Multilayer systems such as TiN/TiCN exhibit improved operational properties by combining the strength TiN with's enhanced plasticity of TiCN, effectively mitigating stress accumulation during service [4-6].

The key parameters determining the effectiveness of such coatings include tribological and nanomechanical properties, such as coefficient of friction (CoF), wear rate (WR), nanohardness, and elastic modulus. These properties are strongly influenced by the coating's structure, surface morphology, and deposition parameters. Direct current (DC) magnetron sputtering is widely used in scientific and industrial settings to produce coatings with high density, uniform microstructure, and strong adhesion to the substrate [7, 8]. However, optimizing multilayer systems requires comprehensive studies, including investigations of morphological features and tribological behavior under boundary lubrication conditions.

Most tribological studies of TiN/TiCN multilayer coatings have been conducted under dry sliding conditions in ambient air. While this approach allows for evaluating basic wear characteristics, it does not always reflect the actual operating conditions of tribological components in mechanical engineering and other industries. In recent years, growing attention has been paid to testing in liquid media that simulate realistic friction and wear processes in mechanical systems. Testing in lubricated environments–such as oils and functional fluids–provides deeper insight into the influence of lubrication on tribological behavior, including changes in CoF, WR, and the mechanism of protective film formation on the contact surface.

For instance, Su Y.L. [9] investigated multilayer TiN/TiCN/TiN coatings under both dry and lubricated conditions using HD-150 oil and water-based lubricants. Results showed that under HD-150 lubrication, the CoF decreased to 0.25, and wear was completely absent due to the formation of a protective tribofilm at the contact surface. In contrast, dry sliding conditions resulted in severe surface damage, iron oxide formation, and fluctuations in CoF.

Tribological tests of multilayer TiAlCN and TiTaCN coatings under dry and lubricated conditions [8] revealed that the CoF ranged from 0.13 to 0.85 in dry sliding. At the same time, under lubrication, it dropped significantly to 0.0015-0.081, with the minimum value (0.0015) recorded for $TiAlCN^{-1}$. The WR in lubricated environments was also considerably lower – for example, 7.4×10^{-9} mm³/N·m for $TiAlCN^{-2}$.

In another study [10], the tribological performance of nanostructured coatings was evaluated under reciprocating sliding against 100Cr6 steel balls using paraffin oil as a lubricant, under varying loads (2 and 7 N) and temperatures (30 and 100°C). The CoF remained largely unaffected by testing parameters; however, a significant reduction in wear volume was observed at elevated temperatures.

Despite extensive research on TiN/TiCN multilayer coatings, tribological assessments under lubricated conditions remain limited in scientific literature. This study deposited monolayer TiN and multilayer TiN/TiCN coatings on VT1-0 titanium substrates via DC magnetron sputtering. The primary objective was to investigate the mechanical and tribological properties of these TiN/TiCN-based multilayer coatings under lubricated friction conditions.

2. Materials and methods

Multilayer TiN/TiCN and monolayer TiN coatings were deposited by direct current (DC) magnetron sputtering using a VT1-0 titanium target (equivalent to GRADE 2) with a diameter of 99 mm. As substrates, finely polished titanium plates (15 × 15 mm, VT1-0) and p-type Si(100) wafers (SW GmbH, Schramberg, Germany) were used. The silicon substrates were employed for morphological investigations, while 58 mm-diameter titanium discs served as substrates for tribological testing.

All substrates underwent surface preparation involving sanding with abrasive paper and polishing with diamond paste. The prepared substrates were mounted on a substrate holder inside the vacuum chamber. The chamber was evacuated to a base pressure of 10^{-3} Pa, after which ion cleaning was performed using argon at a pressure of 0.2 Pa, voltage of 2.5 kV, and current of 20-25 mA for 30 minutes, employing an APEL-IS-21CELL ion source (Applied Electronics, Tomsk, Russia).

Following ion cleaning, the substrates were positioned opposite the APEL-MRE100 magnetron source (Applied Electronics, Tomsk, Russia). A constant substrate bias voltage of –100 V was applied, and the target-to-substrate distance was maintained at 300 mm. The reactive gases used were propane and nitrogen, with argon as the inert carrier gas. The flow rates of both reactive and inert gases were regulated using PPG-12 mass flow controllers (Eltochpribor, Moscow, Russia). All deposition parameters for the TiN and TiN/TiCN coatings are summarized in Table 1.

Table 1. Deposition parameters of TiN and TiN/TiCN coatings by DC magnetron sputtering

Coating	Deposition parameters				
	Deposition time, min	Chamber pressure, Pa	Inert and reactive gas flow, L/h	Plasma current, A	Voltage, V
TiN	140	0.4-0.45	Ar = 1.3; N_2 = 0.08	2	530-550
TiN/TiCN-1	140	0.4-0.45	Ar = 1.3; C_3H_8 = 0.31; N_2 = 0.08	2	530-550
TiN/TiCN-2	140	0.4-0.45	Ar = 1.3; C_3H_8 = 0.31; N_2 = 0.08	2	530-550

Surface morphology of the coatings was examined using a JXA-8230 electron microscope (JEOL, Tokyo, Japan). All samples were analyzed in backscattered electron mode (COMPO) at various magnifications. Coatings were deposited on silicon substrates to facilitate cross-sectional thickness measurements.

Nanohardness measurements were conducted using a NanoScan-4D nanohardness tester (Nanoscan, Moscow, Russian Federation). Ten indents were made with a Berkovich indenter under a load of 100 mN. The penetration depth of the indenter into the coating ranged from 350 to 450 nm. The Young's modulus and hardness were determined using the Oliver and Pharr method [11]. Hardness and modulus profiles were plotted based on the obtained data, considering standard deviations.

Tribological properties of the coatings were evaluated using a ball-on-disk configuration on a TRB3 tribometer (CSM Instruments, Peseux, Switzerland) at room temperature under lubricated sliding conditions with ambient humidity of 42%. A Wezzer 80W-90 transmission oil was used as a lubricant. The tribological testing parameters were as follows: sliding speed of 1 cm/s, normal load of 5 N, wear track radius of 5 mm, total sliding distance of 160 m (5000 cycles), and data acquisition frequency of 50 Hz. The test conditions met the international standard DIN 50324 [12]. A 3 mm diameter ball of 100Cr6 bearing steel was used as the counterbody.

3. Results and discussion

Figure 1 presents the surface morphology and coating thickness analysis results of TiN-based coatings obtained by scanning electron microscopy (SEM). All deposited coatings exhibit a uniform and smooth surface morphology, free of visible defects or microcracks. Minor inclusions (nucleation sites) are present, which may be attributed to the deposition process or the formation of microstructural phases. However, their occurrence is minimal and does not significantly impact on the overall morphology of the coatings.

No substantial variations in surface morphology were observed among the different coating types. The graph in the lower right corner of Figure 1 shows the measured thickness values of the coatings. The monolayer TiN coating exhibited a thickness of 4.4 µm, whereas the multilayer TiN/TiCN and TiN/TiCN/TiN/TiCN coatings showed thicknesses of approximately 4.0 µm. This difference is attributed to the higher deposition rate of TiN compared to TiCN under identical magnetron sputtering conditions. Cross-sectional SEM analysis of the monolayer TiN coating revealed a columnar microstructure characteristic of such coatings [13].

The results of nanohardness (H) and Young's modulus (E) measurements for all investigated coatings are presented in Figure 2a. The graph shows that the multilayer coatings exhibit the highest values, with H = 20–23.5 GPa and E = 161–191 GPa. It should be noted that these values reflect surface-level hardness, as the indenter penetration depth was confined to the uppermost layer. Nevertheless, the multilayer structure significantly contributes to surface hardness during nanoindentation due to several mechanisms: interfacial strengthening (Hall–Petch effect at the nanoscale), restriction of dislocation motion, and hardening induced by residual stress accumulation.

Studies [14-16] have demonstrated that alternating layers with differing mechanical properties enhance hardness compared to monolithic coatings. Among all tested samples, the four-layer TiN/TiCN/TiN/TiCN coating showed the highest hardness value of 23.5 GPa, which may be attributed to the optimal combination of TiN and TiCN layers. TiCN further reinforces the coating structure in this case, increasing stiffness and resistance to mechanical deformation [4, 17].

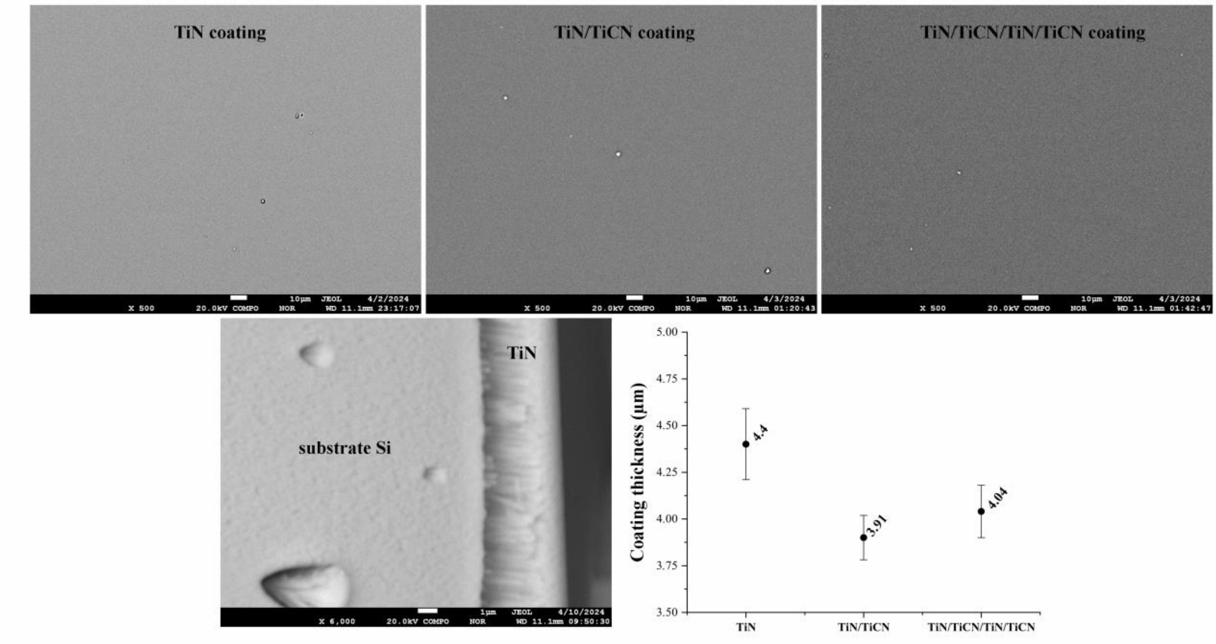
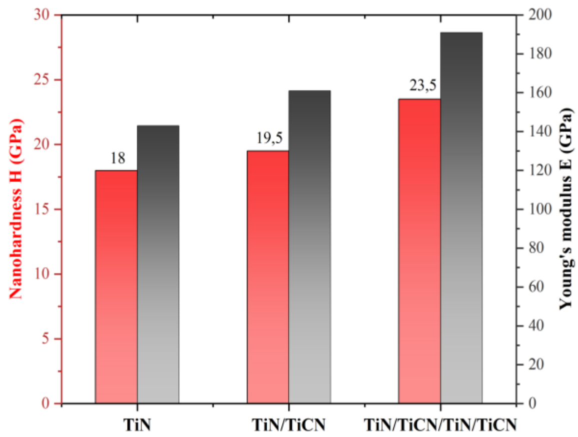
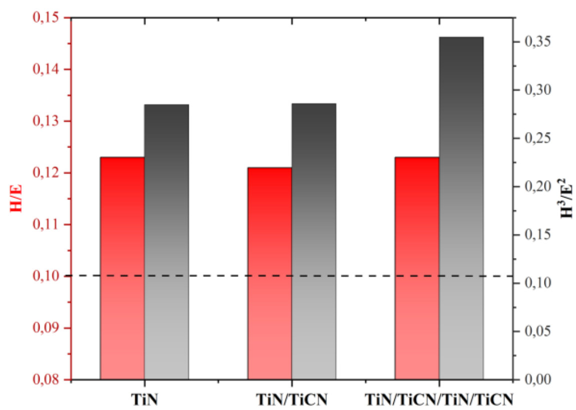


Figure 1. SEM images of surface morphology, cross-sectional view of TiN coating on a silicon substrate, and thickness measurements of the deposited coatings



(a)



(b)

Figure 2. Nanohardness and Young's modulus data of TiN-based deposited coatings

Figure 2b displays the calculated H/E and H^3/E^2 ratios, which are recognized indicators of resistance to mechanical degradation and failure [18]. According to the literature, higher values (H/E > 0.1) are associated with reduced wear rates and lower material loss [19]. From this perspective, all TiN/TiCN/TiN/TiCN multilayer coatings demonstrated the highest H/E and H^3/E^2 values, exceeding the threshold of H/E > 0.1 (indicated by dashed lines), confirming their superior resistance to mechanical failure.

The wear resistance of carbonitride coatings is determined by their microstructure, hardness, and adhesion. These characteristics significantly impact the CoF and WR, which are, in turn, governed by material mass loss during operation [6, 20]. Tribological tests of TiN-based coatings under lubricated sliding conditions revealed low CoF values. This effect can be attributed to a combination of factors: low interfacial adhesion, effective lubrication performance, the possible formation of oxide films, and the high stiffness of TiN all contribute to consistently low CoF under lubrication.

The CoF for all coatings was measured against 100Cr6 steel balls under lubricated conditions. The average CoF was calculated after completing 5000 sliding cycles.

Figure 3 presents the graphs of the CoF with averaged values of all TiN-based coatings deposited on titanium substrates after tribological testing.

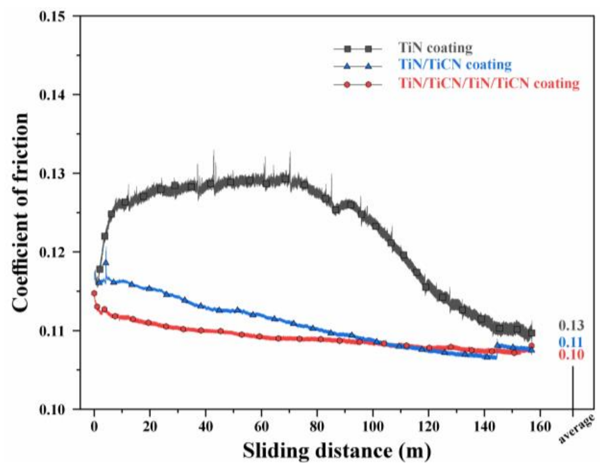


Figure 3. Averaged CoF of the deposited coatings on titanium substrates

According to the results, the average CoF values range from 0.10 to 0.13. Among the tested coatings, monolayer TiN exhibited the highest CoF of 0.13. In contrast, the multilayer coatings demonstrated smoother and lower CoF values than the monolayer TiN coating.

These improved tribological properties are attributed to introducing the TiCN layer, which reduces adhesive friction and enhances load distribution across the coating surface [21, 22]. Incorporating two TiCN layers with differing composition and structure further reduced friction, possibly due to improved crystallinity. These layers may enhance the lubricating behavior of the surface and lower contact resistance, ultimately resulting in minimal friction.

No visible wear tracks were observed on the surfaces of the coatings after tribological tests, which may indicate either minimal material loss or complete wear resistance, as previously reported in studies [23-26].

Thus, the findings of this study confirm that the TiN/TiCN multilayer coatings (both two- and four-layer variants) exhibit significantly higher wear resistance and durability than pure TiN coatings.

4. Conclusions

A comprehensive study was conducted on the mechanical and tribological properties of monolayer TiN and multilayer TiN/TiCN coatings deposited via direct current magnetron sputtering. The main findings are summarized as follows.

SEM analysis revealed that all coatings possess a homogeneous, defect-free microstructure with a characteristic columnar morphology.

Due to interlayer strengthening and dislocation blocking effects, the TiN/TiCN multilayer coatings demonstrated enhanced hardness (up to 23.5 GPa) and elastic modulus (191 GPa) compared to TiN.

Under lubricated sliding conditions, the multilayer TiN/TiCN coatings exhibited significantly lower friction coefficients (0.10–0.13) and superior wear resistance than monolayer TiN. These improved tribological properties are attributed to the optimized layer architecture, reduced adhesion, and formation of protective tribofilms.

No significant wear tracks were observed on the multilayer coatings, confirming their high durability under lubricated friction conditions.

The results confirm that TiN/TiCN multilayer coatings offer superior operational performance to monolayer TiN, making them promising candidates for engineering components subjected to friction and wear. Future work will further optimize layer thickness, interfacial architecture, and deposition parameters to enhance coating performance under real-world operating conditions.

Author contributions

Conceptualization: AKK., AAM., AVP.; Data curation: AKK., BBK; Formal analysis: AKK., AVP.; Funding acquisition: BKK.; Investigation: AKK., AAM., AVP.; Methodology: AKK., BBK; Project administration: BKK.; Resources: BBK; Software: AKK., AVP; Supervision: AAM.; Validation: AAM., AVP.; Visualization: AKK., AAM., AVP.; Writing – original draft: AKK.; Writing – review & editing: AKK. All authors have read and agreed to the published version of the manuscript.

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Conflict of interest

The authors declare no conflict of interest.

Data availability statement

The original contributions presented in this study are included in the article. Further inquiries can be directed to the corresponding author.

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TiN негізіндегі көпқабатты және бірқабатты жабындардың механикалық және трибологиялық қасиеттері

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Аңдатпа. Берілген жұмыста тұрақты токты магнетронды бүрку әдісімен титан негізіндегі (ВТ1-0) үлгілерге тұндырылған моноқабатты TiN және көпқабатты TiN/TiCN жабындарының механикалық және трибологиялық қасиеттері зерттелді. Жабындардың микроқұрылымы, наноқаттылығы, серпімділік модулі және майланған үйкеліс жағдайындағы трибологиялық сипаттамалары талданды. Сканерлейтін электрондық микроскопия (СЭМ) нәтижелері барлық жабындардың біртекті және ақаусыз, бағана құрылымды микроструктураға ие екенін көрсетті. Наноиндентация әдісі бойынша алынған мәліметтерге сәйкес, TiN/TiCN көпқабатты жабындарының қаттылығы (23.5 ГПа дейін) мен серпімділік модулі (191 ГПа) моноқабатты TiN-мен салыстырғанда жоғары, бұл қабатаралық беріктену және қалдық кернеулердің қайта бөлінуімен түсіндіріледі. «Шар-диск» схемасы бойынша майланған үйкеліс жағдайында жүргізілген трибологиялық сынақтар көпқабатты жабындардың үйкеліс коэффициентінің (0.1–0.13) төмен екенін және тозуға төзімділігінің жоғары екенін көрсетті. Бұл нәтиже TiCN қабаттарының енгізілуімен байланысты, олар жабын бетінің адгезиясын төмендетіп, жүктеменің біркелкі таралуына және қорғаныштық трибоқабат түзілуіне ықпал етеді. Алынған нәтижелер TiN/TiCN көпқабатты жабындарының механикалық және трибологиялық қасиеттерінің жақсарғанын дәлелдейді және оларды үйкеліс пен тозуға ұшырайтын инженерлік компоненттерде қолдануға болашағы зор екенін көрсетеді.

Негізгі сөздер: *көпқабатты жабын, магнетронды бүрку, үйкеліс коэффициенті, наноқаттылық, тозуға төзімділік.*

Механическое и трибологическое поведение многослойных и однослойных покрытий на основе TiN

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Аннотация. В данной работе исследованы механические и трибологические свойства монослойных покрытий TiN и многослойных покрытий TiN/TiCN, осажденных методом магнетронного распыления постоянного тока на титановые подложки (ВТ1-0). Проведена их характеристика с точки зрения микроструктуры, нанотвердости, модуля упругости и трибологических характеристик в условиях трения со смазкой. Сканирующая электронная микроскопия (СЭМ) показала, что покрытия имеют однородную микроструктуру без видимых дефектов и характерный столбчатый рост. Испытания методом наноиндентирования продемонстрировали, что многослойные покрытия TiN/TiCN обладают повышенной твердостью (до 23.5 ГПа) и модулем упругости (191 ГПа) по сравнению с монослойным TiN, что объясняется межслойным упрочнением и перераспределением остаточных напряжений. Трибологические испытания по схеме «шар-диск» в условиях смазанного трения показали, что многослойные покрытия демонстрируют значительно меньший коэффициент трения (0.1-0.13) и повышенную износостойкость по сравнению с TiN. Это обусловлено введением слоев TiCN, которые снижают адгезию, способствуют равномерному распределению нагрузки и формированию защитной трибопленки. Полученные результаты подтверждают, что многослойные покрытия TiN/TiCN обладают улучшенными механическими и трибологическими характеристиками, что делает их перспективными для применения в инженерных компонентах, работающих в условиях трения и износа.

Ключевые слова: *многослойное покрытие, магнетронное распыление, коэффициент трения, нанотвердость, износостойкость.*

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