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Investigation of the Structure and Composition of TiN and CrN Coatings as a Function of Deposition Parameters

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ABSTRACT

Addressing corrosion and wear in assemblies, components, machine parts, and equipment operating in aggressive environments under severe wear conditions remains a pressing challenge and continues to draw focused scientific attention. This work aimed to investigate how the key magnetron sputtering parameters (working pressure, plasma current, and process-gas flow rates) affect the surface morphology, microstructure, and composition of TiN and CrN films deposited under different conditions. Microstructural analysis revealed that, across the investigated parameter window, the films exhibit a columnar cross-sectional architecture and a smooth surface morphology with no visible defects, showing no pronounced differences between the deposition regimes. After 30 min of deposition, the film thickness ranged from 0.17 to 0.46 µm for TiN and from 0.59 to 3.46 µm for CrN, depending on the sputtering conditions. The results demonstrate that plasma current and working pressure have a strong effect on film thickness and chemical composition, whereas variations in the working-gas flow rate exert a coupled influence on thickness, microstructure, and the stoichiometry of TiN and CrN layers. Elemental analysis further indicates that increasing the pressure to 0.65 Pa increases oxygen incorporation in the films. During chromium sputtering, raising the plasma current to 1.5 A leads to film delamination. For TiN, a balanced regime with a moderate N2 flow is preferable, providing a reasonable growth rate and a composition close to stoichiometric. For CrN, the range of stable operating conditions is substantially broader, and the process parameters have a more pronounced impact on its structure and composition. These findings can support the design of TiN/CrN wear-resistant multilayer coatings produced by magnetron sputtering for protecting machine parts and equipment against wear and corrosion.

Keywords: working chamber pressure, plasma discharge current, nitrogen flow rate, film elemental composition, film thickness.

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Introduction

The durability of parts and components operating in aggressive environments under intense friction and wear (mechanical engineering, transport, chemical and petrochemical industries, power generation, and related sectors) remains a critical challenge. Premature failure of such components under service conditions can lead to substantial economic losses [1]. Improving the tribological and corrosion performance of contacting surfaces largely determines their service life, operational safety, and reliability [[2], [3], [4], [5]]. For this reason, enhancing the tribo-corrosion behaviour of frictional surfaces continues to be a key focus of scientific research.

One of the most effective approaches to addressing this challenge is surface engineering through the deposition of coatings with high wear resistance, hardness, and corrosion resistance [[6],[7][8]]. Metal-nitride coatings are widely employed for these purposes because they combine features typical of covalent compounds with those of ionic crystals. The incorporation of nitrogen alters bond energy, expands the crystal lattice, increases interatomic distances, and raises the lattice parameter, imparting distinctive physical and chemical properties to these compounds [[9], [10]].

Metal–nitride coatings can be produced by chemical vapour deposition (CVD), physical vapour deposition (PVD), and related methods. Selecting an appropriate deposition route is crucial because it directly affects coating microstructure and performance. In practice, metal-nitride coatings are predominantly fabricated by PVD processes; among them, magnetron sputtering (MS) is one of the most frequently used techniques. MS enables control over coating structure and composition by adjusting process parameters such as working pressure, plasma discharge current, reactive-gas flow rate, substrate bias, and others [11].

Pressure governs the energy and transport of species, thereby affecting film densification and porosity; discharge current/power controls the growth rate and the degree of ionization; the nitrogen flow rate regulates nitride stoichiometry and the onset of the target "poisoning" regime; substrate bias intensifies ion bombardment, increasing density and adhesion while promoting the build-up of residual stresses; and temperature determines adatom mobility and crystallinity [[12], [13]]. For multilayer coatings (e.g., TiN/CrN), these parameters additionally control interfacial sharpness, bilayer periodicity, and stress partitioning, which collectively define the mechanical, tribological, and corrosion performance.

For nitride coatings (TiN, CrN, TiAlN/CrAlN), the key requirements are the optimization of working pressure and nitrogen flow together with strict composition control to ensure high density, hardness, corrosion resistance, and thermal stability. For carbonitrides and multicomponent/multilayer systems (TiCN, TiAlCrN, AlCrSiN, TiN/CrN), additional critical factors include regulating carbon-containing gases, distributing power between targets, and controlling architectural parameters (layer period, interface sharpness, and the balance of residual stresses).

Among PVD-derived metal-nitride coatings, titanium nitride (TiN) and chromium nitride (CrN) attract sustained interest because they offer a favourable combination of high hardness, strong adhesion, wear resistance, corrosion resistance, and thermal stability, making them versatile for a wide range of applications [[9], [14]]. However, quantitative "parameter → structure/composition" relationships for individual TiN and CrN coatings remain under active investigation. Several studies have examined how sputtering conditions (pressure, gas flow, current density, ionization) affect composition, microstructure, and residual stresses in TiN and CrN films. At low pressure and moderate plasma currents, TiN typically forms a NaCl-type cubic structure with a preferred (111) or (200) orientation. For example, study [15] showed that increasing the working pressure from 2 mTorr to 9 mTorr changes grain orientation and increases oxygen incorporation in TiN coatings. Increasing the nitrogen fraction during titanium sputtering (nitrogen and argon flow rates: 9.0 and 51 sccm) while reducing pressure to 2 Pa has been reported to decrease vacancy concentration and increase coating density, as demonstrated by Wei B. and co-workers [16].

For CrN, Bai H. and others [9] demonstrated that both the deposition method and sputtering parameters affect nitrogen content and defect population in CrN coatings and, consequently, their mechanical behaviour. The authors of [17] reported that residual stress and hardness depend strongly on the sputtering regime. The authors found that increasing the deposition pressure from 2 to 4, 5, and 7 mTorr reduced film deformation and increased the oxygen content. The pronounced effect of substrate bias was attributed to enhanced ionization of film-forming species. In [18], the influence of MS parameters on the tribological performance of CrN coatings was examined. Thin CrN films were deposited at pressures of 0.4 and 4 Pa; after testing, the coating synthesized at 0.4 Pa exhibited less surface damage due to its higher film density, and its hardness was higher by 5 GPa.

Overall, studies on TiN and CrN deposition indicate that controlling MS parameters enables tuning of composition (metal-to-nitrogen ratio and oxygen incorporation), microstructure (grain size and texture), and, consequently, key properties such as hardness and wear resistance. This knowledge underpins the design of multilayer coating systems and the optimization of deposition regimes for practical applications (e.g., tooling and mechanical engineering). In this context, the present work investigates the structure and composition of TiN and CrN films as a function of magnetron sputtering parameters (pressure, plasma current, and working-gas flow rate) to support the subsequent design of TiN/CrN multilayer coatings.

Experimental part

TiN and CrN films were deposited by direct-current magnetron sputtering (DCMS) in a high-vacuum system developed by the authors, employing two separate magnetrons (APEL-MRE100 and MKE-95/100; Applied Electronics, Tomsk, Russian Federation). A 99 mm diameter VT1-0 titanium target (VostokMetService, Ust-Kamenogorsk, Kazakhstan) and a 75 mm diameter ERKh-1 chromium target (Ural Metall Export–Kazakhstan, Astana, Kazakhstan) were used. Polished p-type single-crystal Si(100) substrates (SW GmbH, Schramberg, Germany) with dimensions of 10 × 10 mm were mounted inside the chamber on a 200 mm-diameter substrate holder. The chamber was evacuated to a base pressure of 5×10^{-3} Pa using diffusion and rotary pumps (2NVR-60D; Vakuummash, Kazan, Russian Federation). The chamber pressure was monitored with a Televac CC-10 vacuum gauge (The Fredericks Company, USA). The substrates were then ion-cleaned at 0.3 Pa with a plasma current of 40 mA and an accelerating voltage of 2.5 kV for 20 min using an APEL-IS-21CELL ion source (Applied Electronics, Tomsk, Russia). After ion etching, the cleaned substrates were positioned opposite the corresponding magnetrons for TiN and CrN deposition.

The following deposition parameters were maintained throughout: substrate bias −100 V, deposition time 30 min, target-to-substrate distance 300 mm, and argon flow rate 1.3 L/h. Argon and nitrogen flow rates were regulated using RRG-12 flow controllers (Eltochpribor, Moscow, Russia). For TiN deposition, the plasma current was varied between 0.5 and 2 A, the working pressure between 0.3 and 0.65 Pa, and the nitrogen flow rate between 0.08 and 0.11 L/h. For CrN deposition, the plasma current ranged from 0.5 to 1.5 A, the working pressure from 0.3 to 0.65 Pa, and the nitrogen flow rate from 0.08 to 0.2 L/h.

Film thickness and morphology were evaluated by scanning electron microscopy (SEM) using a JXA-8230 microscope (JEOL, Tokyo, Japan) equipped with an energy-dispersive X-ray spectroscopy (EDS) system for elemental analysis.

Graphs and figures were prepared using OriginPro SR1 10.1.0.178 and CorelDRAW 25.0.0.230.

Results and Discussion

Microstructural analysis confirmed that, under the investigated conditions, the deposited films exhibit a columnar cross-sectional morphology and a smooth microstructure without visible defects. Depending on the sputtering parameters, after 30 min of deposition, the TiN and CrN thicknesses ranged from 0.17 to 0.46 µm and from 0.59 to 3.46 µm, respectively. Under identical magnetron sputtering conditions, the CrN deposition rate was approximately 3.5 times higher than that of TiN. The effects of the individual MS parameters on TiN and CrN films are discussed below.

Effect of Working Pressure in the MS Chamber

Based on the preceding results, the working pressure during titanium and chromium sputtering was set to 0.45 and 0.65 Pa while maintaining a nitrogen flow rate of 0.08 L/h. The thicknesses of TiN and CrN films synthesized at 0.65 Pa were lower than those obtained at 0.3 and 0.45 Pa. At elevated pressure (0.65 Pa), the TiN and CrN growth rates decrease due to an increased frequency of collisions in the plasma, which reduces the kinetic energy and mean free path of the sputtered species. This trend is consistent with previous reports [[15],[18]]. During titanium sputtering, increasing the pressure from 0.45 to 0.65 Pa decreased the TiN deposition rate from 0.16 to 0.13 µm/min, whereas during chromium sputtering, the CrN deposition rate decreased from 0.50 to 0.38 µm/min.

SEM examination of the surface morphology of TiN and CrN films deposited at 0.45 and 0.65 Pa revealed a smooth, uniform surface, with only occasional, randomly distributed nuclei. Figure 1 summarizes the surface morphology and EDS-derived composition. The left-hand images (×250) show a dense and homogeneous microstructure without pronounced defects. Both titanium nitride and chromium nitride surfaces exhibit a similar, uniform contrast, indicating good film continuity. The small number of micron-sized nuclei observed on the TiN and CrN surfaces may be associated with droplet-phase formation or microdroplets, a phenomenon occasionally reported for magnetron sputtering processes [[19], [20]]. Their low areal density suggests stable deposition conditions. Notably, the surface morphology did not change appreciably with variations in pressure, plasma current, or nitrogen flow rate; therefore, only cross-sectional images are discussed in the following sections.

The right-hand plot in Fig. 1 shows the chemical composition of the films as a function of working pressure. Elemental analysis indicates that increasing the pressure to 0.65 Pa increases the oxygen content to 26.45 at.% in CrN and to 10.32 at.% in TiN, which may adversely affect tribological performance. The increase in oxygen content with increasing pressure has been reported by many researchers [[15], [17], [18]]. This effect is commonly attributed to the incorporation of residual gases during film growth and to the pronounced affinity of Cr for oxygen.

On the basis of the pressure-dependent results, subsequent deposition experiments were conducted at 0.45 Pa. However, given the relatively low nitrogen content in CrN films, a higher nitrogen flow rate during chromium sputtering is required to promote nitride formation.

Effect of Magnetron Sputtering Plasma Discharge Current

To examine the influence of the MS plasma discharge current on the structure and composition of TiN and CrN films, films were deposited on Si substrates at a chamber pressure of 0.45 Pa with a nitrogen flow rate of 0.08 L/h and an argon flow rate of 1.3 L/h for 30 min.

During titanium sputtering, plasma currents below 0.5 A led to deviations of TiN from stoichiometry due to changes in the discharge zone, whereas currents above 2.0 A caused overheating of the magnetron housing. Similar behaviour has been reported in [[21],[22]]. Figure 2 presents a cross-sectional SEM image and the dependence of the TiN deposition rate and film thickness on the plasma current. The cross-section clearly reveals the “film-substrate” interface. The film exhibits a dense microstructure without cracks or visible defects. Thickness uniformity across the cross-section confirms stable deposition conditions and efficient mass transport of species to the substrate. As shown in the right panel of Fig. 2, increasing the plasma current results in an approximately linear rise in deposition rate from 5 to 15 nm/min, leading to a film thickness of ~0.5 µm. The chemical composition of TiN films as a function of plasma current is summarized in Table 1, where reducing the current is accompanied by changes in the titanium and nitrogen contents. Because the film deposited at 2 A exhibited a composition closest to stoichiometric, and given that titanium has a lower sputtering yield than chromium, the plasma current for titanium sputtering was fixed at 2 A in subsequent experiments.

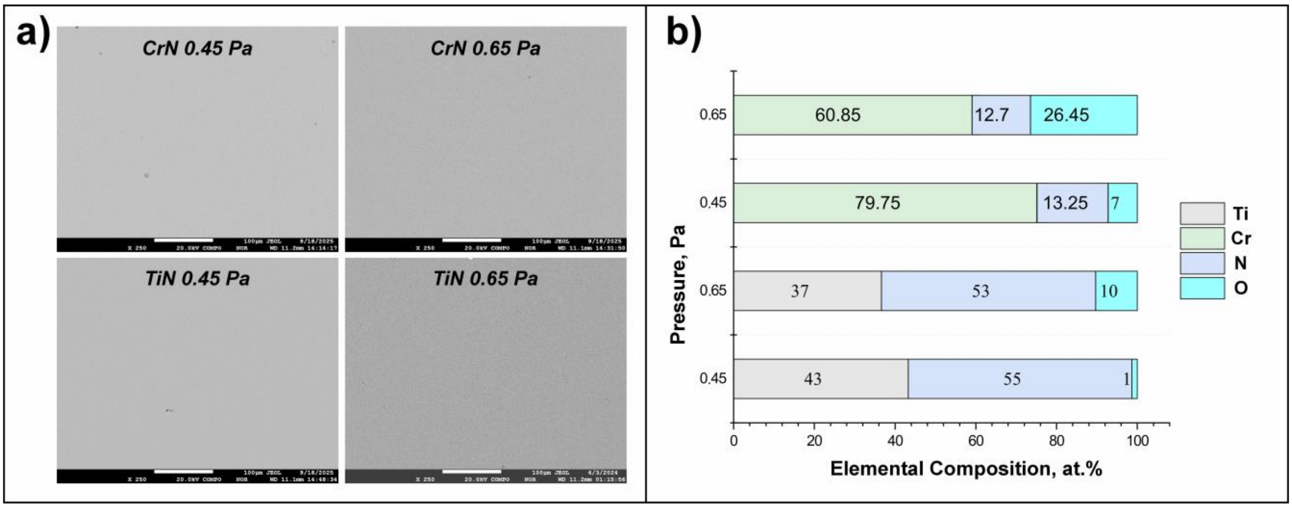

Figure 1 - Surface morphology (a) and chemical composition (b) of TiN and CrN films as a function of working pressure

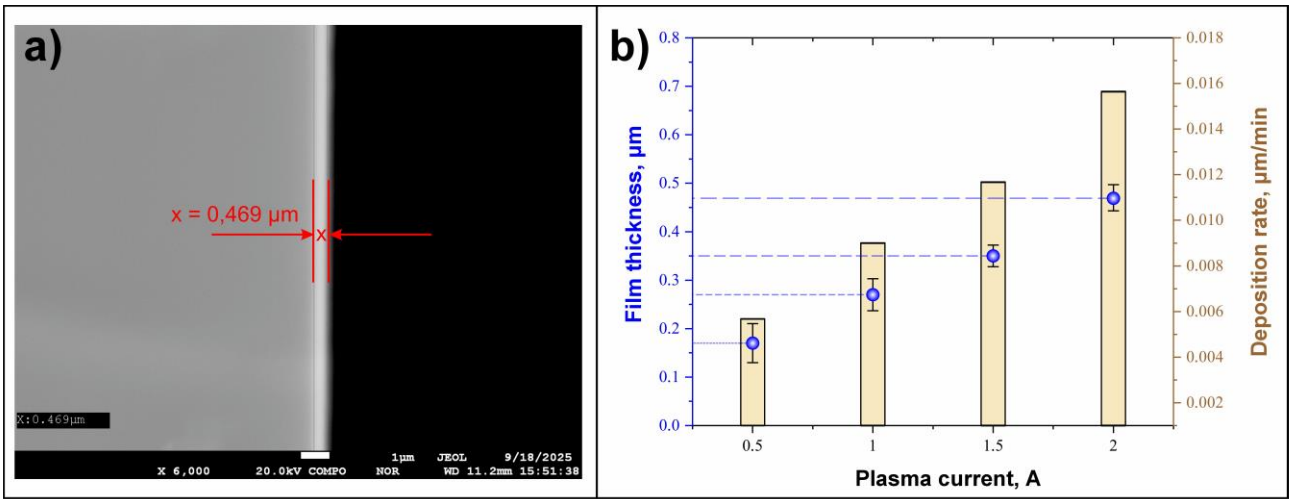

Figure 2 - Cross-sectional SEM image of the TiN film at 2 A (a) and the dependence of deposition rate and film thickness on plasma current (b)

Table 1 - Chemical composition of TiN films deposited at plasma currents from 0.5 to 2 A

Sample	Plasma current, A	Ti	N	O
7-1	2	43.25	55.4	1.35
7-2	1.5	43.1	53.8	3.1
7-3	1	39.5	59.3	1.2
7-4	0.5	32.2	62.5	5.3

During chromium sputtering, plasma currents above 1.5 A led to film delamination, which is attributed to an excessively high deposition rate and the build-up of residual stresses. This can degrade the adhesion durability of the CrN film, ultimately leading to delamination, as also reported in [[9], [17], [23]].

Figure 3 provides cross-sectional SEM images of the CrN film and shows the dependence of deposition rate and film thickness on plasma current. The deposition rate increased from 0.02 to 0.11 µm/min as the plasma current was raised, resulting in film thicknesses ranging from 0.5 to 3.46 µm. The higher growth rate can be explained by the intensification of ion-plasma processes, including a greater probability of ionization of sputtered species and an increased deposition efficiency [24]. The relatively small thickness scatter, shown as error bars, indicates good reproducibility of the deposition process.

Table 2 summarizes the composition of CrN films deposited at plasma currents from 0.5 to 1.5 A in 0.5 A increments. Elemental analysis shows that increasing the plasma current markedly reduces the oxygen content in the film, while the chromium fraction increases. At the lower current of 0.5 A, oxygen from residual chamber gases is incorporated into the growing film. Raising the current to 1.0-1.5 A increases the density of energetic ions in the plasma, thereby promoting nitridation [[25], [26]]. As a result, the chromium and nitrogen contents increase, whereas the oxygen level decreases.

Based on the current-dependent deposition results, a plasma current of 2 A was selected for titanium sputtering and 1 A for chromium sputtering as the working regimes for subsequent experiments.

Effect of Nitrogen Flow Rate during MS

The influence of nitrogen flow rate on the stoichiometry of individual TiN and CrN films was examined at a constant argon flow rate of 1.3 L/h.

For TiN films, the nitrogen flow rate was varied between 0.08, 0.18, 0.36, and 0.54 L/h. Under these conditions, a decrease in the TiN deposition rate was observed with increasing nitrogen flow (Fig. 4). This behaviour can be attributed to a reduction in the effective sputtering yield of the titanium target caused by the formation of a TiN compound film on the target surface; in addition, the density and kinetic energy of titanium species reaching the substrate decrease due to a higher collision frequency in the gas phase [[27], [28]]. In other words, the process transitions from a metallic mode to a reactive regime. Figure 4 shows the deposition rate as a function of nitrogen flow rate together with the corresponding TiN film compositions. As the nitrogen flow increases, the titanium fraction decreases, whereas the nitrogen content rises.

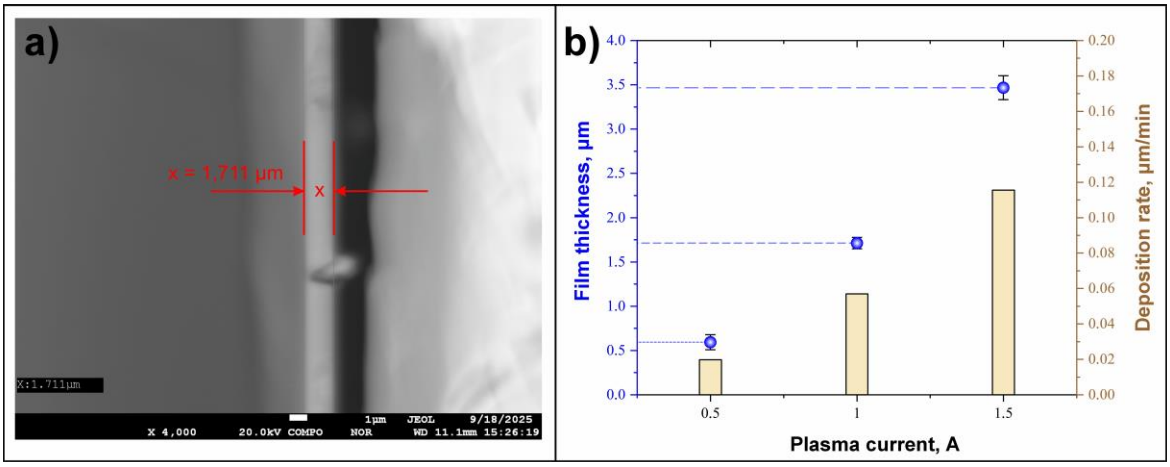

Figure 3 - Cross-sectional SEM image of the CrN film at 1 A (a) and the dependence of deposition rate and film thickness on plasma current (b)

Table 2 - Chemical composition of CrN films deposited at plasma currents from 0.5 to 2 A

Sample	Plasma current, A	Cr	N	O
1	0.5	60.5	12.69	26.81
3	1	79.75	13.03	7.22
2	1.5	81.3	12.66	6.04

Across all regimes, the nitrogen content was in the range of 44-56 at.% and titanium in the range of 36-47 at.%. Based on the composition data, 0.08 L/h is the optimal nitrogen flow rate for forming TiN.

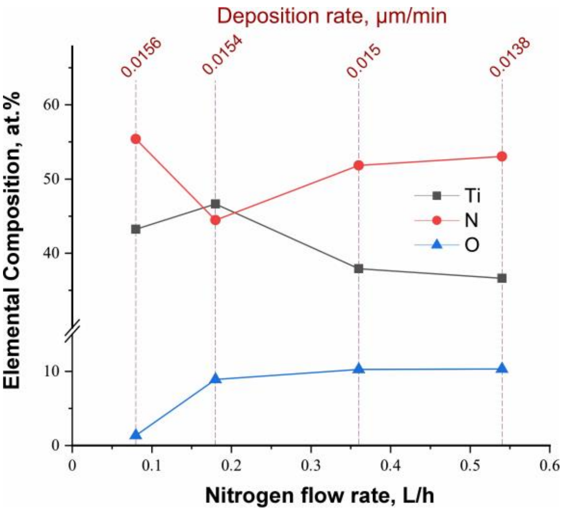

Figure 4 - Elemental composition and deposition rate of TiN films as a function of nitrogen flow rate

For CrN layers, the nitrogen flow rate was varied at 0.08, 0.12, 0.18, 0.22, and 0.26 L/h. As in the TiN case, increasing the nitrogen flow initially reduced the CrN deposition rate, after which the rate levelled off. However, unlike TiN, CrN films exhibited a substantially higher growth rate (0.055–0.057 µm/min), consistent with the higher sputtering yield of chromium [29]. Elemental analysis (Fig. 5) shows that increasing the nitrogen flow decreases the chromium content from ~80 to ~67 at.% while simultaneously increasing the nitrogen fraction to ~25 at.%. This behaviour indicates a transition from a metallic or nitrogen-deficient Cr(N) state toward a nitride phase approaching CrN; nevertheless, even at the maximum nitrogen flow, the composition remains shifted toward metal-rich stoichiometry. The elevated oxygen content (up to ~21 at.% at intermediate N2 flow) suggests a tendency of CrN films toward oxygen contamination, which may be associated with the high chemical reactivity of chromium [[30], [31], [32], [33]].

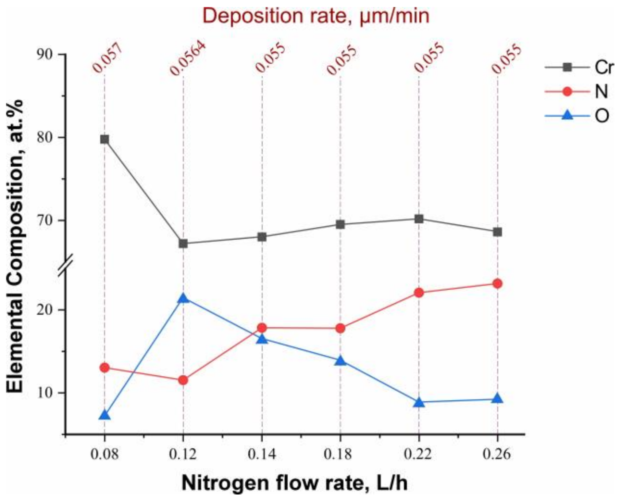

Figure 5 - Elemental composition and deposition rate of CrN films as a function of nitrogen flow rate

Thus, optimizing the nitrogen flow rate is a key lever for controlling both the deposition rate and the stoichiometry of TiN and CrN coatings. For TiN, a balanced regime with a moderate N_2 flow is preferable, providing a reasonable growth rate together with a composition close to stoichiometric. For CrN, the range of stable operating conditions is substantially broader, indicating higher process robustness during reactive magnetron deposition.

Conclusions

The present work investigated the structure and composition of TiN and CrN films as a function of key magnetron sputtering parameters (pressure, plasma current, and process-gas flow rates) to support the design of TiN/CrN multilayer coatings. The main conclusions are as follows:

- The experiments showed that plasma current and working pressure strongly affect film thickness and chemical composition, whereas variations in the working-gas flow rate exert a coupled influence on thickness, microstructure, and layer stoichiometry.
- For TiN deposition, a near-optimal regime was identified at a working pressure of 0.45 Pa, with titanium sputtered at an N2 flow rate of 0.08 L/h and a plasma current of 2 A.
- For CrN deposition, a working pressure of 0.45 Pa, a plasma current of 1 A, and an N2 flow rate of 0.22 L/h were found to be optimal; however, the N2 flow rate requires further refinement.

Further studies will focus on a more detailed assessment of film properties under the identified operating regimes and on evaluating the behaviour of these layers within a TiN/CrN multilayer architecture.

Conflicts of interest. On behalf of all authors, the corresponding author states that there is no conflict of interest.

CRediT author statement: **A. Kenzhegulov**: Conceptualization, Methodology, Software; **K. Smailov**: Data curation, Writing draft preparation; **A. Mamaeva and N. Bakhytuly**: Visualization, Investigation; **A. Kenzhegulov**: Supervision; **A. Uskenbayeva**: Software, Validation; **Zh.Zh. Alibekov**: Reviewing and Editing.

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TiN және CrN жабындарының құрылымы мен құрамын тұндыру параметрлеріне байланысты зерттеу

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Қабылданды: *29 мамыр 2026*

Агрессивті ортада және қарқынды тозу жағдайында жұмыс істейтін тораптар, құрамдас бөлшектер, машиналар мен жабдық элементтерінің коррозиясы мен тозуы мәселесін шешу өзекті және ерекше ғылыми назар аударатын нысан болып табылады. Бұл жұмыстың мақсаты әртүрлі шарттарда тұндырылған TiN және CrN қабықшаларының морфологиясына, микроқұрылымына және құрамына магнетрондық тозаңдатудың негізгі параметрлерінің (қысым, плазма тогы, жұмыс газдарының ағыны) әсерін зерттеу. Нәтижесінде микроқұрылымдық талдау зерттелген жағдайларда көлденең қимасында бағаналы құрылымы және тегіс беті морфологиялы, көрінетін ақаулары жоқ қабықшалардың қалыптасатынын, олардың микроқұрылымы көрінетін ақаулардан бос және шашырау жағдайларынан айтарлықтай айырмашылық көрсетпейтінін анықтады. Тозаңдату параметрлеріне байланысты, 30 минуттық тұндыру кезінде TiN және CrN қабықшаларының қалыңдығы сәйкесінше 0,17–0,46 мкм-ге дейін және 0,59–3,46 мкм -ге дейін аралығында болды. Жүргізілген жұмыс нәтижелеріне сүйене отырып, плазмалық тоқ пен қысым қабықша қалыңдығына мен химиялық құрамына елеулі ықпал ететінін, ал жұмыс газы ағынының өзгеруі TiN және CrN қабаттарының қалыңдығына, құрылымына және құрамының стехиометриясына кешенді әсер ететініні анықталды. Элементтік талдау нәтижелері қысымды 0,65 Па-ға дейін арттыру қабықшалардағы оттегі мөлшерін өсетінін көрсетті.

Хромды тозаңдату кезінде плазма тогын 1,5 А-ға дейін ұлғайту қабықшаның қабыршақтануына әкеледі. TiN қабықшалары үшін өсу жылдамдығын қабылдауға болатын деңгейді сақтай отырып, стехиометрияға жақын құрамды қамтамасыз ететін N_2 ағынының орташа мәндеріндегі компромистік режим қолайлы. CrN үшін тұрақты режимдер диапазоны едәуір кең және құрылым мен құрамға үлкен әсер етеді. Алынған нәтижелер машиналар мен жабдық бөлшектерін тозу мен коррозиядан қорғауға қолданылатын магнетрондық тозаңдату әдісімен көпқабатты тозуға төзімді TiN/CrN жабын жүйелерін жобалау кезінде пайдалы болуы мүмкін.

Түйін сөздер: камераның жұмыс қысымы, плазмалық разряд тогы, азот ағынының жылдамдығы, қабықшаның элементтік құрамы, қабықшаның қалыңдығы.

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Исследование структуры и состава покрытий TiN и CrN в зависимости от параметров осаждения

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АННОТАЦИЯ

Решение проблемы коррозии и износа узлов, компонентов, деталей машин и оборудовании, работающих в условиях агресиной среди и интенсивного износа, является актульным и является объектом особого научного внимания. Целью настоящей работы являлась исследование влияния основных параметров магнетронного распыления (давление, ток плазмы, поток рабочих газов) на морфологию, микроструктуру и состав пленок TiN и CrN, осажденных в разных условиях. В результате, микроструктурный анализ показал, что при исследованных режимах формируются пленки с колонной структурой по поперечному сечению и гладкой поверхностной морфологией микроструктура котрого без видимых дефектов и существенно не отличающегося от режимов напыления. В зависимости от параметров распыления при 30 минутной осаждении толщина пленок было в пределах от 0,17 до 0,46 мкм и от 0,59 до 3,46 мкм для TiN и CrN, соответственно. По результатам проведённых работ установлено, что ток плазмы и давление существенно влияют на толщину и химический состав пленок, тогда как изменение потока рабочего газа оказывает комплексное влияние на толщину, структуру и стехиометрию состава слоёв TiN и CrN. Результаты элементного анализа показали, что при увеличении давления до 0,65 Па возрастает содержание кислорода в пленках. В случае распылении хрома повышения тока плазмы до 1,5 А приводит к отслоению пленки. Для плеок TiN предпочтителен компромиссный режим с умеренным потоком N2, обеспечивающий приемлемую скорость роста и близкую к стехиометрической композицию. Для CrN диапазон стабильных режимов существенно шире и проявялет большего влияние на структуру и состав. Полученные результаты могут быть полезны при проектировании многослойной системы износостойкого покрытия TiN/CrN методом магнетронного распыления, которые используется для защиты от износа и коррозии деталей машин или оборудовании.

	Ключевые слова: рабочее давление камеры, ток разряда плазмы, скорость потока азота, элементный состав пленки, толщина пленки.
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