

Article

Structure, Corrosion, and Tribological Properties of TiON Coatings Prepared by Reactive Magnetron Sputtering for Potential Biomedical Surface Applications

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Abstract

This study investigates titanium oxynitride (TiO_xN_y) coatings deposited by reactive magnetron sputtering on 316L stainless steel substrates in an Ar–N₂–O₂ gas mixture at a fixed N:O ratio of 1.6. The coatings were deposited under three reactive magnetron sputtering regimes with Ar flow rates of 33, 28, and 26 sccm and corresponding substrate biases of –50, –100, and –150 V, respectively, while the N₂ and O₂ flow rates were kept constant at 10 and 6 sccm. The coatings exhibited a dense microstructure, with thicknesses ranging from 2.13 to 5.51 μm. X-ray diffraction analysis revealed the formation of a multiphase structure comprising TiN, TiO_xN_y, and TiO. The deposition regime had a significant influence on the functional properties of the coatings. The lowest friction coefficients ($\mu \approx 0.26$ –0.28) and stable tribological behavior were characteristic of the Ar26 sample. The highest corrosion resistance was observed for the Ar28 sample, with a corrosion current density of $i_{\text{corr}} = 2.82 \times 10^{-7}$ A/cm² and a corrosion rate of $v_{\text{corr}} = 0.00573$ mm/year. All coatings exhibited hydrophilic surface behavior, with contact angles of 50–57°, which may be relevant for further evaluation in biomedical surface applications. Thus, the structure and functional properties of TiO_xN_y coatings can be regulated by selecting an appropriate deposition regime, including the Ar flow rate, relative reactive gas fraction, and substrate bias. However, additional biological tests, including cytotoxicity, hemocompatibility, endothelialization, and platelet adhesion studies, are required before conclusions about vascular implant applicability can be made.

Keywords: titanium oxynitride; thin protective coatings; biomedical surface modification; corrosion resistance; mechanical properties; tribological behavior



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1. Introduction

Cardiovascular diseases, particularly atherosclerotic arterial lesions, remain one of the leading causes of mortality worldwide. The use of metal coronary stents has significantly improved the effectiveness of restoring blood flow and reduced mortality in cases of myocardial infarction. Nevertheless, the clinical efficacy of stenting remains limited by the risk of restenosis and thrombosis caused by adverse interactions between the implant surface and the biological environment [1–3]. The use of drug-eluting coatings has reduced the incidence of restenosis, but is accompanied by a number of complications, including chronic inflammation, delayed endothelialization, and the risk of late thrombosis, which

necessitates the search for alternative non-pharmacological approaches to modifying the stent surface [4–6].

One of the most effective approaches to improving the biocompatibility of vascular implants is to modify their surface using functional coatings. Stainless steel 316L is widely used as a structural material for coronary stents due to its high mechanical strength and corrosion resistance. However, the presence of elements such as Cr, Ni, and Mo in its composition can lead to the release of ions under physiological conditions, causing inflammatory reactions and allergic effects, which reduces the material's long-term biocompatibility [7–9]. In this regard, the development of protective coatings capable of reducing the intensity of corrosion processes and preventing the migration of metal ions is of significant interest.

The surface characteristics of implants play a key role in regulating biological processes after implantation. The adsorption of blood plasma proteins, platelet activation, and the formation of the endothelial layer directly depend on the chemical composition, morphology, and surface energy of the material [10–12]. In particular, surface wettability is one of the determining factors influencing the nature of protein adsorption. Hydrophilic surfaces promote the formation of a more stable protein layer, reducing the likelihood of thrombus formation, whereas hydrophobic surfaces can initiate activation of the blood coagulation system [13]. Thus, controlling surface wettability is an important tool for improving the hemocompatibility of stents.

Ceramic coatings based on titanium compounds, such as TiO_2 and TiN , are widely used due to their high hardness, wear resistance, and corrosion resistance [14]. They are capable of effectively protecting the metal substrate from electrochemical degradation in physiological environments. However, single-phase TiO_2 and TiN coatings have limited potential for simultaneously optimizing mechanical, tribological, and biological properties.

In this regard, titanium oxynitride (TiO_xN_y) coatings are of particular interest, as they combine the advantages of both oxide and nitride phases. According to previous studies, Ti–O–N bonding states in titanium oxynitride coatings may influence their electronic structure, surface energy, and corrosion resistance [15–17]. Titanium oxynitride coatings have also been reported to improve barrier properties and reduce metallic ion release from the substrate [18–20]. Surface wettability and surface chemistry may influence protein adsorption and platelet interactions [21–24]; however, these biological effects require direct verification by cytotoxicity, hemocompatibility, endothelialization, and platelet adhesion tests. Therefore, in the present study, the biomedical relevance of TiO_xN_y coatings is discussed only in terms of structural, mechanical, tribological, electrochemical, and wettability-related properties, rather than as direct evidence of biocompatibility.

The phase composition and microstructure of TiO_xN_y coatings are largely determined by the conditions of their formation. Reaction magnetron sputtering parameters, such as gas mixture composition, pressure, and discharge power, have a significant influence on the crystallinity, defect density, and morphology of the coatings. Changing the ratio of reactive gas flows can affect the concentration of oxygen vacancies, the possible formation of mixed Ti–O–N bonding states, and the surface energy, which, in turn, may influence the mechanical, tribological, and electrochemical characteristics of the coatings.

The mechanical properties of coatings, including hardness, elastic modulus, and wear resistance, determine their ability to withstand mechanical stresses under stent operating conditions [25]. A mismatch between the mechanical properties of the coating and the substrate can lead to stress accumulation, crack formation, and subsequent coating failure. At the same time, corrosion resistance is a critically important parameter determining the implant's longevity in a physiological environment. In this regard, a comprehensive study of the relationship between the structure, mechanical, and electrochemical properties of coatings is of particular interest.

Although TiN-, TiO₂-, and TiO_xN_y-based coatings have been widely investigated as protective and functional layers for metallic biomaterials, most previous studies have focused mainly on individual aspects, such as phase composition, corrosion behavior, or biological response. Less attention has been paid to the simultaneous relationship between reactive magnetron sputtering regimes, coating structure, mechanical response, tribological behavior, corrosion resistance, and surface wettability of TiO_xN_y coatings deposited on 316L stainless steel. In particular, the combined influence of gas mixture composition and substrate bias on the multifunctional performance of TiO_xN_y coatings remains insufficiently clarified.

The novelty of this work lies in the comparative analysis of TiO_xN_y coatings deposited on 316L stainless steel under three reactive magnetron sputtering regimes with different Ar flow rates and substrate biases. Unlike studies focused on a single property or a single deposition parameter, this work evaluates the relationship between deposition regime, microstructure, phase composition, hardness, adhesion behavior, friction response, corrosion resistance, and wettability within one experimental framework. This approach makes it possible to identify deposition conditions that provide a balanced combination of functional properties for potential biomedical surface applications.

The aim of this work is to investigate the morphology, structural phase state, chemical composition, mechanical, tribological, electrochemical, and surface properties of TiO_xN_y coatings deposited on 316L stainless steel under three different reactive magnetron sputtering regimes and to clarify the relationship between deposition conditions and the functional behavior of the coatings.

2. Materials and Methods

A 99.99% pure Ti target was used to deposit the TiO_xN_y coating. The coatings were deposited on 316L stainless steel substrates. 316L stainless steel is widely used as a structural material for biomedical implants, including angioplasty devices and coronary stents, due to its favorable mechanical properties and corrosion resistance. However, under physiological conditions, localized corrosion and the release of alloying ions such as Ni, Cr, and Mo may occur, which can induce inflammatory or allergic reactions. Therefore, the development of protective surface coatings is an important approach to improving the long-term performance of 316L stainless steel implants. The substrate dimensions were 30 mm × 30 mm × 4 mm. Prior to sputtering, the substrate surface was progressively ground using abrasive paper of various grits (from P400 to P2500). The chemical composition of the 316L stainless steel substrate was taken from the nominal reference data for AISI 316L stainless steel and is presented in Table 1.

Table 1. Nominal chemical composition of the 316L stainless steel substrate.

C	Mn	P	S	Si	Cr	Ni	Mo	Ti	Fe
<0.03	<2.0	<0.045	<0.03	<1.0	16.0–18.0	10.0–14.0	2.0–3.0	<0.5	Remaining

Prior to coating deposition, ion cleaning of the substrate surface was performed to reduce surface contamination and improve adhesion between the coating and the base material [26]. During ion cleaning, the argon flow rate was 15 sccm, and the working pressure in the chamber was maintained at 0.2 Pa. The cleaning process was carried out at an applied voltage of 2.5 kV and a current of 50–80 mA for 20 min. In this study, the cleaning efficiency was not evaluated by a separate quantitative surface analysis method, such as XPS. Therefore, its effect was assessed indirectly based on the formation of continuous

coatings, the clearly defined coating/substrate interface observed in cross-sectional SEM images, and the coating behavior during scratch testing.

Titanium oxynitride (TiO_xN_y) was obtained by reactive magnetron sputtering of a titanium target (Ti 99.9%) in an Ar + N₂ + O₂ atmosphere at a constant N:O ratio of 1.6. The coatings were deposited under three different sputtering regimes. The Ar flow rate was 33, 28, or 26 sccm, while the N₂ and O₂ flow rates were kept constant at 10 and 6 sccm, respectively. At the same time, the substrate bias was varied from −50 to −150 V. Therefore, the samples are denoted as Ar33, Ar28, and Ar26 according to the Ar flow rate used in each deposition regime. The deposition time was 3 h for all coating regimes.

The ion energy at the substrate is mainly controlled by the applied substrate bias, whereas the Ar flow rate affects plasma density, collision processes, deposition rate, and the relative fraction of reactive gases in the discharge. Therefore, the observed changes in coating structure and properties should be interpreted as the combined effect of gas mixture composition and bias-controlled ion bombardment.

The deposition time for each coating was 3 h. The main deposition parameters used in this study are summarized in Table 2, including the Ar–N₂–O₂ gas flow rates, substrate bias, operating pressure, discharge voltage and current, target material, substrate temperature, target–substrate distance, and deposition time. The target power can be estimated from the discharge voltage and current as approximately 880–960 W. However, the base pressure, target diameter, and target power density were not available in the experimental log and therefore could not be included. This limitation should be taken into account when reproducing the deposition process.

Table 2. Magnetron sputtering parameters for coating deposition.

Parameter	$\text{TiO}_x\text{N}_y_{-33}$	$\text{TiO}_x\text{N}_y_{-28}$	$\text{TiO}_x\text{N}_y_{-26}$
Gas mixture flow rate (Ar:N ₂ :O ₂), sccm	Ar: 33 N ₂ : 10 O ₂ : 6	Ar: 28 N ₂ : 10 O ₂ : 6	Ar: 26 N ₂ : 10 O ₂ : 6
Substrate bias, V	−50	−100	−150
Operating pressure, Pa	0.8–1.0		
Voltage U, V:	440–480 V		
Current I, A:	2 A		
Target material	Ti, 99.9%		
Substrate temperature, °C	100		
Distance, mm	30		
Deposition time, h	3		

Note: Bold values indicate the varied Ar flow rates used to define the sample names.

It should be noted that in the present study, the Ar flow rate and substrate bias were varied simultaneously. Therefore, the individual contributions of these two parameters cannot be fully separated. For this reason, the obtained results are discussed as the effect of different deposition regimes rather than as the isolated effect of Ar flow rate alone.

The surface microstructure and morphology of the coatings were examined using a TESCAN VEGA4 LMH scanning electron microscope (SEM) (TESCAN, Brno, Czech Republic), and energy-dispersive X-ray analysis was performed on an Xplore 30 system (Oxford Instruments, Oxford, UK). An X'PertPRO X-ray diffractometer (Philips Corporation, Amsterdam, the Netherlands) with Cu K α radiation was used for X-ray phase analysis.

Coating hardness was measured using the FISCHERSCOPE HM 2000 system (Helmut Fischer GmbH, Sindelfingen, Germany), controlled by WIN-HCU software version 7.1. This device is designed to evaluate microhardness and other mechanical properties of materials

in accordance with ISO 14577 on cross-sections of samples. In all nine tests, the dwell time was 10 s at a load of 2000 mN. To investigate the adhesion characteristics and failure behavior of the coatings, scratch testing was performed using a Revetest Scratch Tester RST 300 (Anton Paar, Graz, Austria). Scratch testing was carried out at a maximum load of 27 N, with a normal loading rate of 26.99 N/min, an indenter travel speed of 9.63 mm/min, a scratch length of 10 mm, and a tip radius of 100 μm . This test was used to evaluate coating adhesion and damage evolution under progressively increasing normal load.

Tribological studies were conducted separately using an Anton Paar TRB3 tribometer (Anton Paar, Buchs, Switzerland) in a ball-on-disc configuration, in accordance with ASTM G133-95 and ASTM G99. These tests were used to evaluate the friction behavior of the coatings under dry sliding conditions. A 6 mm diameter 100Cr6 steel ball was used as the counterbody. The tests were performed at a normal load of 1 N, a linear sliding speed of 5 cm/s, and a wear track radius of 4 mm, which corresponds to an approximate rotational speed of 119 rpm.

Corrosion resistance was investigated using a CS300M potentiostat–galvanostat (CorrTest Instruments Corp., Wuhan, China). The coatings were tested with an exposed area of 1 cm^2 at room temperature (25 $^{\circ}\text{C}$) in a 3.5 wt.% NaCl solution. The experiment was performed using a three-electrode cell, where an Ag/AgCl electrode served as the reference electrode and a platinum electrode served as the counter electrode. Prior to each polarization experiment, the specimen was exposed to the electrolyte for 60 min of immersion until a stable open-circuit potential (OCP) was established. The corrosion potential and current density were obtained from the polarization curves using the Tafel extrapolation method for the four specimens. The potential was scanned in the range from -0.1 to 0.1 V relative to the OCP at a scan rate of 0.5 mV/s. The tests were repeated three times, and the results were analyzed using CS Studio6 software (version 6.3). The wettability of the TiO_xN_y surface was evaluated by the hanging drop method at 25 $^{\circ}\text{C}$ using deionized water.

3. Results

3.1. Morphology and Structural Phase Analysis

Figure 1 shows cross-sectional SEM images of TiO_xN_y coatings deposited under different deposition regimes, as well as the corresponding EDS spectra and quantitative elemental analysis results.

In all cases, for the Ar33, Ar28, and Ar26 samples, continuous coatings with a clearly defined interface between the coating and the substrate are formed, which indicates good adhesion. At the same time, their morphology and elemental composition depend significantly on the deposition regime, including the Ar flow rate, relative reactive gas fraction, and substrate bias.

The Ar33 sample is characterized by the greatest thickness (~ 5.5 μm), which indicates an increased deposition rate. According to EDS data, the coating is enriched with titanium (≈ 56.6 wt.%) and oxygen (≈ 33.8 wt.%) in the presence of carbon (≈ 9.65 wt.%). Its presence is likely due to residual gases and may lead to the formation of a less dense structure.

Sample Ar28 exhibits the minimum thickness (~ 2.1 μm), indicating a decrease in growth rate. The composition is characterized by a reduced titanium content (≈ 40.6 wt.%) with a comparable oxygen level (≈ 30.1 wt.%) and a significant carbon content (≈ 29.4 wt.%). The relatively high carbon content detected for the Ar28 sample should be interpreted with caution. It may be related to surface contamination, residual carbon-containing species in the vacuum chamber, sample preparation artifacts, or the contribution of carbon-based mounting materials during EDS analysis. Therefore, this value should not be considered as direct evidence of carbon incorporation into the TiO_xN_y lattice without additional chemical

state analysis. Further XPS analysis would be required to distinguish between surface contamination and chemically bonded carbon-containing species.

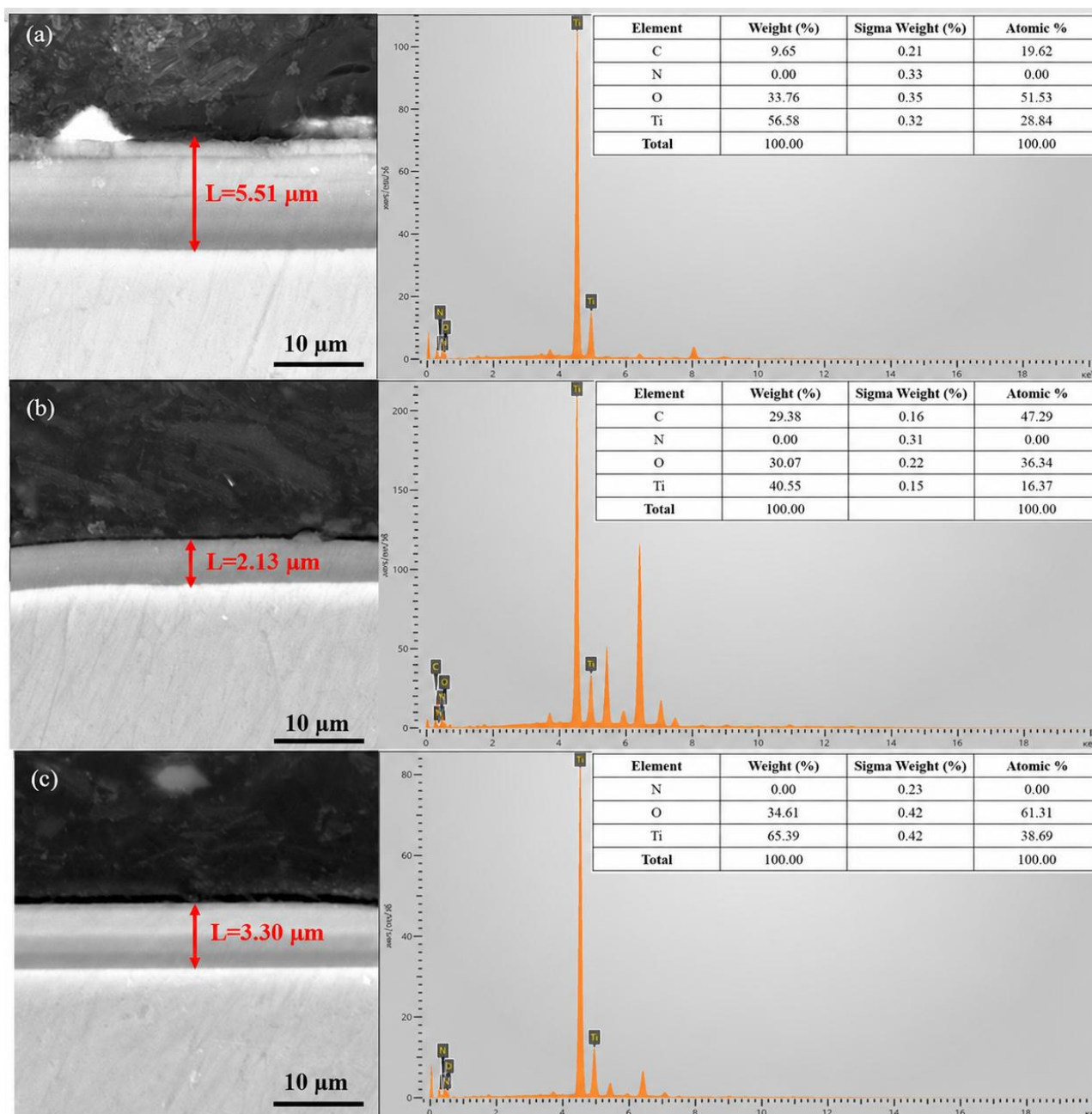


Figure 1. Cross-sectional SEM images, EDS spectra, and elemental analysis of TiO_xN_y coatings deposited under different deposition regimes: (a) Ar33, (b) Ar28, and (c) Ar26.

Nitrogen was not reliably detected by EDS in the analyzed coatings. This can be attributed to the limited sensitivity of EDS for light elements, the low X-ray emission energy of nitrogen, and possible background effects during analysis. Therefore, the EDS results should be used mainly for qualitative comparison of Ti, O, and C contents, whereas the formation of TiO_xN_y phases is supported primarily by the XRD results and the reactive $\text{Ar-N}_2\text{-O}_2$ deposition conditions. Direct confirmation of Ti–O–N bonding states, chemical environment, and valence states requires XPS analysis.

The Ar26 sample has an intermediate thickness ($\sim 3.3 \mu\text{m}$) and exhibits the highest concentrations of titanium ($\approx 65.4 \text{ wt.}\%$) and oxygen ($\approx 34.6 \text{ wt.}\%$) in the absence of carbon, indicating the formation of a more homogeneous and denser layer.

Thus, the Ar33 deposition regime is accompanied by an increase in coating thickness, but also by the appearance of impurities. Ar28 is characterized by the minimum thickness and the highest carbon content, whereas Ar26 demonstrates the most balanced characteristics. Among the coatings studied, the Ar26 deposition regime provides the most balanced combination of sufficient thickness, purity of composition, and dense structure.

For a more in-depth analysis of the phase composition and crystal structure of the obtained coatings, X-ray diffraction analysis was additionally performed.

The X-ray diffraction (XRD) patterns of TiO_xN_y coatings obtained under different deposition regimes are shown in Figure 2. The diffractograms were processed using HighScore Plus software (PANalytical B.V., Almelo, The Netherlands, version 5.3.1).

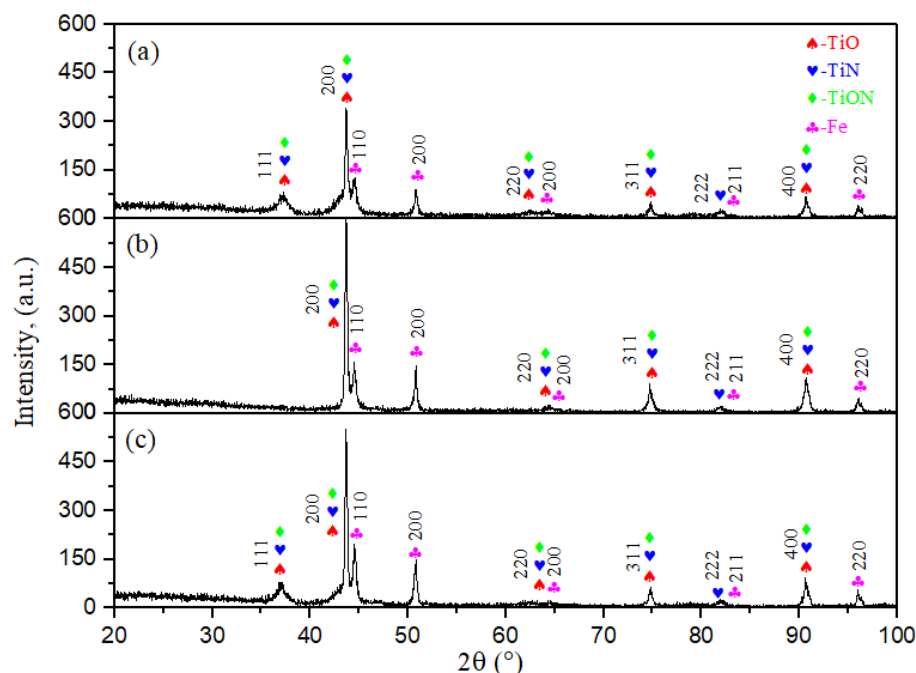


Figure 2. X-ray diffraction patterns of TiO_xN_y coatings deposited on a 316L stainless steel substrate under various deposition conditions: (a) Ar33, (b) Ar28, (c) Ar26.

In all samples (Ar33, Ar28, and Ar26), a crystalline multiphase structure is formed, comprising TiN, TiO_xN_y , and TiO_x . The main diffraction peaks in the $2\theta \approx 36\text{--}43^\circ$ region correspond to (111) and (200) reflections characteristic of the cubic TiN/ TiO_xN_y phase. A (200) plane texture is observed in all coatings, although its intensity varies.

Ar33. It is characterized by high-intensity (111) and (200) peaks, as well as clearly defined (220) and (311) reflections in the $2\theta \approx 61\text{--}63^\circ$ and $74\text{--}75^\circ$ regions. This indicates a well-developed crystalline structure and pronounced textured growth. At the same time, noticeable oxide peaks are observed in the region of $2\theta \approx 25^\circ$, indicating the presence of an oxide phase.

Ar28. Shows a decrease in the intensity of all diffraction peaks and their broadening. The (200) peak is weakened, the (220) and (311) reflections are weak, and the substrate ($\gamma\text{-Fe}$) contribution is maximal. This indicates a minimal coating thickness, reduced crystallinity, and increased structural defects.

Ar26. It is characterized by narrow and intense (111), (200), (220), and (311) peaks, indicating a high degree of crystallinity and structural order. The intensity of the oxide peaks in the $2\theta \approx 25^\circ$ region is reduced compared to Ar33 and Ar28, and the substrate contribution is less pronounced, indicating the formation of a denser coating.

As the structure transitions from Ar28 $\rightarrow$ Ar33 $\rightarrow$ Ar26, an increase in crystallinity and structural order is observed. The Ar28 sample is characterized by the most defective and thin structure. Ar33 exhibits high peak intensity, but with a noticeable contribution from the oxide phase. Ar26 combines high crystallinity, a smaller contribution from oxide phases, and a reduced substrate influence.

The highest-quality structure is formed under the Ar26 deposition regime, as evidenced by the high intensity and narrow width of the diffraction peaks, a lower amount of the oxide phase, and a denser coating.

3.2. Mechanical and Tribological Properties

The mechanical behavior of TiO_xN_y coatings obtained under different deposition regimes is presented in Figure 3, while the hardness, elastic modulus, and calculated mechanical performance indices are summarized in Table 3. All samples exhibit typical elastic–plastic behavior: elastic deformation predominates in the initial stage, and the contribution of the plastic component increases with increasing load.

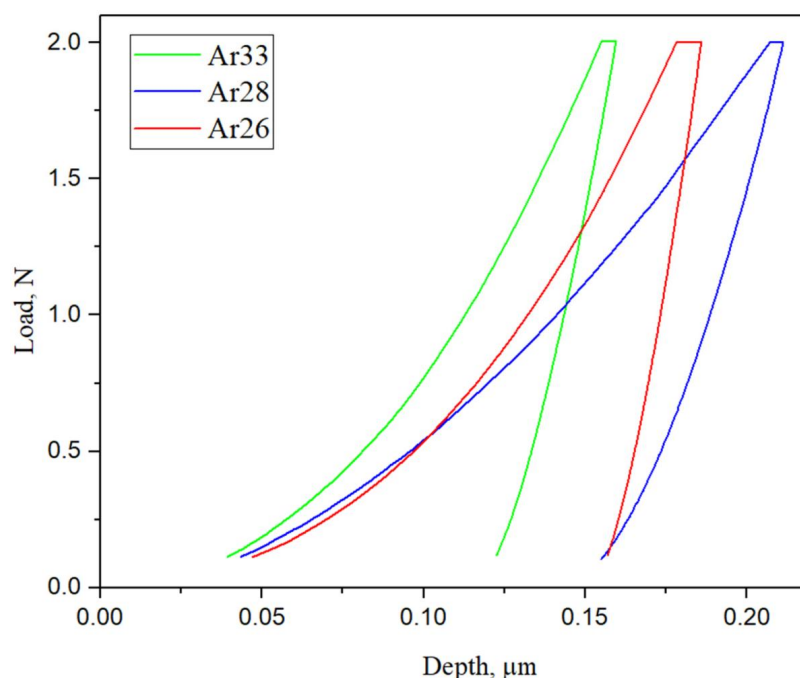


Figure 3. Load–displacement curves of TiO_xN_y coatings obtained from microindentation tests.

Table 3. Mechanical properties and calculated mechanical performance indices of TiO_xN_y coatings.

Sample	Martens Hardness, H_M (MPa)	Vickers Hardness, HV	Elastic Modulus, E (GPa)	H/E	H^3/E^2 , GPa
Ar33	3117.5 ± 266.0	428.0 ± 46.3	99.6 ± 4.2	0.0313	0.0031
Ar28	1738.0 ± 82.7	246.3 ± 22.5	52.8 ± 2.6	0.0329	0.0019
Ar26	2354.9 ± 190.0	285.1 ± 29.7	103.7 ± 9.3	0.0227	0.0012

Note: H/E and H^3/E^2 were calculated using H_M values converted from MPa to GPa.

The Ar33 coating is characterized by the smallest indenter penetration depth at the same load, which is consistent with its highest hardness values ($H_M = 3117.5 \pm 266.0$ MPa and $HV = 428.0 \pm 46.3$). This coating also exhibits the highest H^3/E^2 value (0.0031 GPa), indicating better resistance to plastic deformation. Such behavior may be related to its greater coating thickness and improved resistance to localized deformation during indentation.

The Ar28 sample exhibits the greatest indenter penetration depth, indicating lower resistance to deformation. This sample demonstrates the lowest hardness values ($H_M = 1738.0 \pm 82.7$ MPa and $HV = 246.3 \pm 22.5$) and the lowest elastic modulus ($E = 52.8 \pm 2.6$ GPa). Although Ar28 shows the highest H/E ratio (0.0329), its lower H^3/E^2 value (0.0019 GPa) indicates reduced resistance to plastic deformation. This behavior may be associated with its smaller coating thickness and a stronger influence of the stainless-steel substrate during indentation.

The Ar26 coating occupies an intermediate position in terms of penetration depth and hardness ($H_M = 2354.9 \pm 190.0$ MPa and $HV = 285.1 \pm 29.7$). Its elastic modulus reaches $E = 103.7 \pm 9.3$ GPa, while the H/E and H^3/E^2 values are 0.0227 and 0.0012 GPa, respectively. These results indicate a more balanced elastic–plastic response compared with Ar28, although its resistance to plastic deformation is lower than that of Ar33.

Overall, the mechanical response of the coatings is controlled not only by hardness but also by the combined effect of coating thickness, elastic modulus, and possible substrate contribution during indentation. Because the coatings are relatively thin, the measured hardness and elastic modulus values should be interpreted with caution, since the contribution of the 316L stainless-steel substrate cannot be completely excluded. Therefore, the calculated H/E and H^3/E^2 ratios provide a more detailed interpretation of the deformation behavior of the coatings than hardness values alone.

Figure 4 shows the dependence of the coefficient of friction μ on the sliding distance for TiO_xN_y coatings obtained under different deposition regimes.

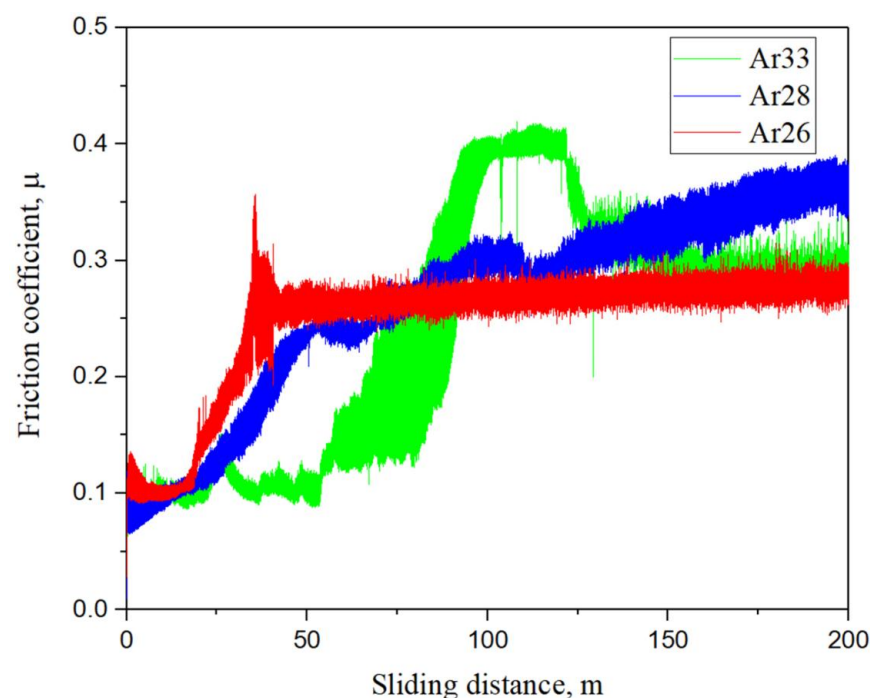


Figure 4. Results of tribological tests of TiO_xN_y coatings under various deposition conditions.

At the initial stage of testing, a running-in phase is observed for all samples, accompanied by an increase in the coefficient of friction, which is associated with an increase in the effective contact area between the counterbody and the coating surface.

The Ar33 coating is characterized by a sharp increase in the coefficient of friction after the running-in stage, reaching values of $\mu \approx 0.40$ – 0.42 in the range of sliding distances of ~ 80 – 120 m. This region is accompanied by pronounced fluctuations, indicating the instability of the tribological contact. With a further increase in sliding distance, a decrease

in μ to ~ 0.30 – 0.33 is observed; however, the fluctuations persist, indicating an unstable friction regime.

The Ar28 sample is characterized by a smoother change in the coefficient of friction. After the running-in stage is complete, μ gradually increases from ~ 0.25 to ~ 0.35 – 0.38 as the sliding length increases. The absence of sharp jumps indicates more stable contact compared to Ar33; however, the observed trend indicates a gradual deterioration of tribological characteristics.

The Ar26 coating demonstrates the most stable behavior. After the initial section, the coefficient of friction settles in the range of $\mu \approx 0.26$ – 0.28 and remains virtually unchanged throughout the entire test. The low values of μ and the absence of significant fluctuations indicate a steady-state sliding regime and a stable condition of the contacting surfaces.

A comparative analysis shows that the tribological characteristics of the coatings vary in the following order: Ar33 $\rightarrow$ Ar28 $\rightarrow$ Ar26, with a decrease in the coefficient of friction and an increase in process stability. The Ar33 sample is characterized by maximum values of μ and pronounced instability; Ar28 occupies an intermediate position with a tendency toward an increase in μ , whereas Ar26 demonstrates minimum and stable values of the coefficient of friction.

Thus, the most favorable tribological properties are realized for the coating obtained under the Ar26 deposition regime. This behavior may be associated with the combined effect of lower Ar flow, higher relative reactive gas fraction, and higher negative substrate bias, which promoted the formation of a denser and more homogeneous structure and ensured a stable friction regime.

From a mechanistic point of view, the differences in tribological behavior can be associated with coating density, defect formation, and resistance to plastic deformation. The Ar33 coating exhibits the highest hardness and the highest H^3/E^2 value, which indicates better resistance to plastic deformation; however, local brittle fracture and the formation of wear debris may occur during sliding. Such wear debris can act as third-body abrasive particles in the contact zone and lead to local fluctuations in the friction coefficient. The Ar28 coating demonstrates the lowest hardness and a lower H^3/E^2 value, suggesting reduced resistance to plastic deformation. Together with its smaller thickness, this may promote stronger substrate influence, easier defect initiation, and progressive damage accumulation during sliding. In contrast, the Ar26 coating shows a more stable tribological response, which may be related to a more homogeneous coating structure and a more balanced elastic–plastic behavior. Therefore, the tribological behavior is controlled not only by the friction coefficient itself, but also by the relationship between coating structure, deformation resistance, defect development, and coating integrity.

Figure 5 presents the results of scratch testing of TiO_xN_y coatings deposited under different deposition regimes, in the form of plots of normal load (Fn), coefficient of friction (CoF), indenter penetration depth (Pd), and acoustic emission (AE) signal. Additionally, optical images of the scratch tracks are provided, reflecting the nature of the coating failure.

For the Ar33 coating, during the initial loading stage (up to ~ 2 – 3 N), the coefficient of friction remains at a relatively stable level, which corresponds to elastic and elastic–plastic deformation. The appearance of the first acoustic emission peaks at loads of ~ 2 – 4 N indicates the initiation of local defects (Lc_1). In the load range of ~ 5 – 12 N, an increase in the AE signal and a gradual increase in penetration depth are observed, which corresponds to the development of cohesive damage (Lc_2 – Lc_3). With a further increase in load to ~ 15 – 16 N, a sharp increase in AE and the coefficient of friction is recorded, accompanied by an increase in penetration depth, which corresponds to adhesive failure of the coating and partial exposure of the substrate (Lc_4).

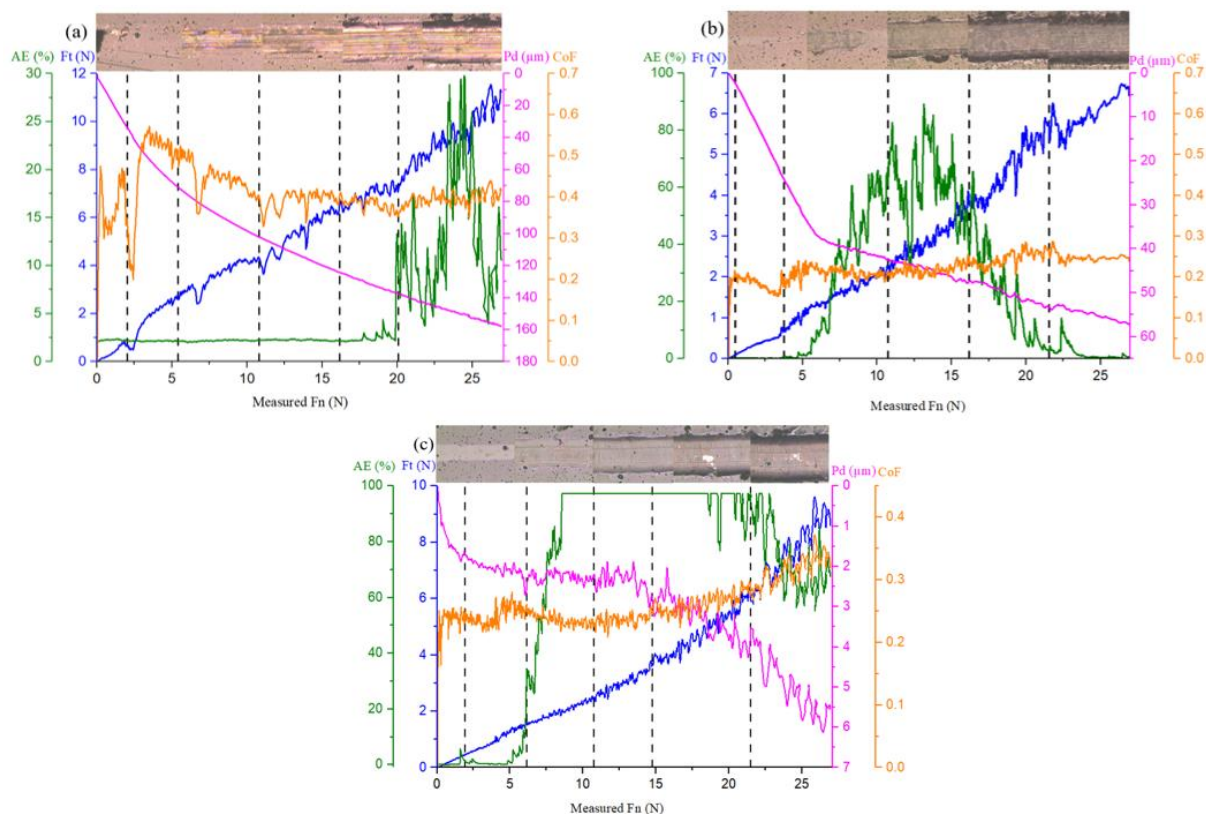


Figure 5. Scratch test results for TiO_xN_y coatings deposited under different deposition regimes: (a) Ar33, (b) Ar28, and (c) Ar26. The vertical dashed lines are used as visual guides to separate different regions of the scratch-test curves.

The Ar28 sample is characterized by an earlier appearance of acoustic emission signals, indicating reduced resistance of the coating to defect initiation. The transition to intensive damage development is observed in the range of ~6–11 N, where a significant increase in AE and penetration depth is recorded. Complete failure of the coating occurs at loads of approximately ~16 N and is accompanied by unstable behavior of the coefficient of friction. The amplification of signals from the substrate and the nature of the scratch track indicate a less stable coating structure.

The Ar26 coating demonstrates a more uniform change in all parameters during loading. The first AE signals appear at loads of ~2 N; however, further damage development occurs gradually, without sharp jumps. In the load range of ~6–12 N, a steady increase in penetration depth is observed at moderate friction coefficient values. Complete coating failure occurs at loads of ~14–15 N and is accompanied by an increase in AE activity and the friction coefficient; however, the nature of the damage remains less abrupt compared to other samples.

A comparative analysis shows that the deposition regime has a significant effect on the failure mechanism and adhesive properties of TiO_xN_y coatings. The Ar33 coating demonstrates higher resistance to failure during the intermediate stages of loading, whereas the Ar28 sample is characterized by earlier defect development. The Ar26 coating exhibits the most stable behavior during loading and a gradual failure pattern.

Thus, optimizing deposition conditions, particularly the Ar flow rate, relative reactive gas fraction, and substrate bias, can increase the resistance of TiO_xN_y coatings to mechanical failure and improve their adhesion properties.

The scratch test response can be interpreted in terms of coating cohesion, defect density, and coating–substrate interaction. Abrupt changes in acoustic emission, coefficient

of friction, or penetration depth are usually associated with the initiation and propagation of cracks, cohesive failure within the coating, or partial adhesive failure at the coating–substrate interface. The thinner Ar28 coating is more sensitive to substrate contribution during scratching, which can accelerate damage accumulation and reduce the apparent resistance of the coating to mechanical loading. The Ar33 coating, despite its higher hardness, may be more prone to localized brittle damage under concentrated contact stresses. The Ar26 coating demonstrates a more gradual damage evolution, which suggests better structural uniformity and improved resistance to localized defect propagation. Thus, the scratch test results support the conclusion that coating performance depends on the combined effect of hardness, thickness, cohesion, defect density, and substrate influence.

3.3. Corrosion and Surface Properties

Figure 6 shows the potentiodynamic polarization curves of TiO_xN_y coatings deposited under different deposition regimes, and Table 4 lists the corresponding electrochemical parameters determined by the Tafel extrapolation method.

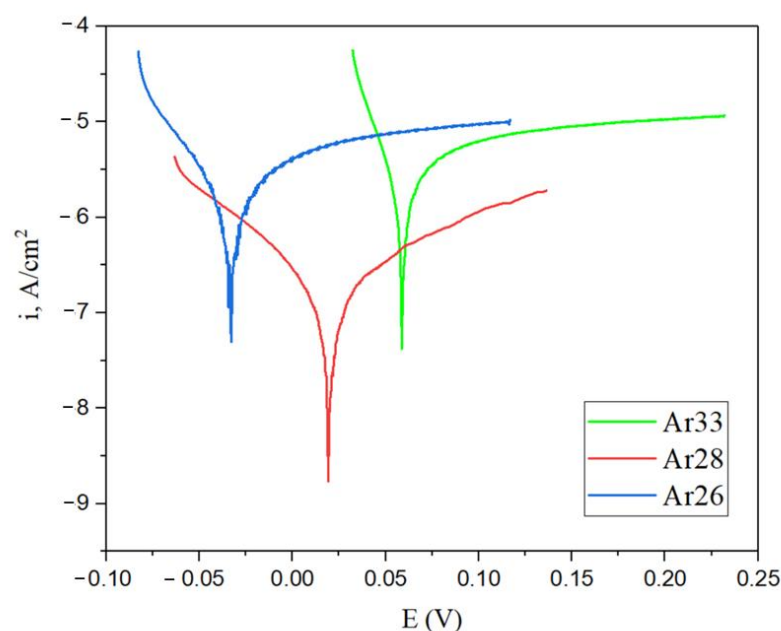


Figure 6. Potentiodynamic polarization curves of TiO_xN_y coatings obtained under different deposition regimes: Ar33, Ar28, and Ar26.

Table 4. Corrosion test results.

Parameter	Ar33	Ar28	Ar26
E_{corr} (mV)	58.83	19.38	−32.99
i_{corr} (A/cm^2)	5.26×10^{-6}	2.82×10^{-7}	4.57×10^{-6}
β_a (mV)	454.08	135.27	399.07
β_c (mV)	35.18	79.12	72.36
v_{corr} (mm/year)	0.1069	0.00573	0.0930

Analysis of the polarization curves shows that all coatings exhibit characteristic passive behavior in the test solution; however, their electrochemical characteristics vary significantly depending on the deposition conditions. The lowest current density values in the corrosion potential region are observed for the Ar28 coating, indicating its increased corrosion resistance. This observation is confirmed by quantitative data: for the Ar28 sample, the corrosion current density is $i_{\text{corr}} = 2.82 \times 10^{-7} \text{ A}/\text{cm}^2$, which is an order of

magnitude lower than the values obtained for the Ar33 ($5.26 \times 10^{-6} \text{ A/cm}^2$) and Ar26 ($4.57 \times 10^{-6} \text{ A/cm}^2$) coatings.

The corrosion rate shows a similar trend. For the Ar28 coating, the value of v_{corr} is 0.00573 mm/year, whereas for the Ar33 and Ar26 samples, this parameter reaches 0.1069 mm/year and 0.0930 mm/year, respectively. A significant reduction in v_{corr} indicates the coating's effective barrier properties, which prevent the aggressive medium from penetrating to the substrate.

Corrosion potential values range from -32.99 mV (Ar26) to 58.83 mV (Ar33); however, changes in E_{corr} are not a determining factor for corrosion resistance, as corrosion current density remains the key parameter.

Analysis of the Tafel coefficients shows that for the Ar28 coating, a decrease in the anodic coefficient β_a to 135.27 mV is observed compared to Ar33 (454.08 mV) and Ar26 (399.07 mV), as well as an increase in the cathodic coefficient β_c to 79.12 mV. This indicates a slowing of anodic dissolution and suppression of cathodic reactions, leading to a reduction in the overall corrosion rate.

Thus, a comprehensive analysis of the polarization curves and electrochemical parameters shows that the optimal corrosion characteristics of TiO_xN_y coatings are achieved under the Ar28 deposition regime. The increased corrosion resistance of this coating is due to the formation of a more stable passive state and a reduction in the intensity of anodic and cathodic processes.

Although the potentiodynamic polarization results provide important information about the corrosion resistance of the TiO_xN_y coatings, it should be noted that electrochemical impedance spectroscopy (EIS) measurements were not performed in this study. Therefore, the barrier properties, charge transfer resistance, and long-term stability of the coating/electrolyte interface were not evaluated in detail. In addition, post-corrosion surface observations were not carried out, which limits the direct analysis of local corrosion damage and surface degradation after electrochemical testing. Future studies should include EIS measurements and post-corrosion SEM analysis to obtain a more complete understanding of the corrosion protection mechanism of TiO_xN_y coatings.

The results of measuring the contact angle of TiO_xN_y coatings obtained under different deposition regimes are presented in Figure 7. The contact angle was determined using the sessile drop method with deionized water.

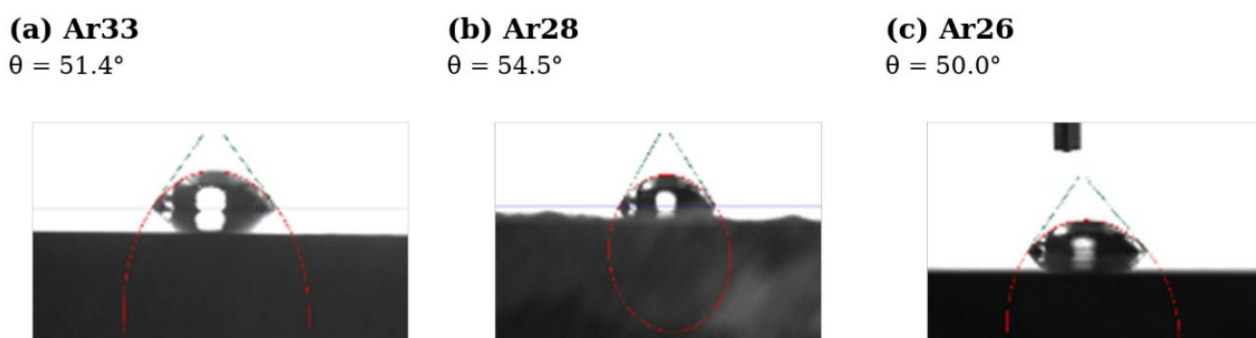


Figure 7. Water contact angle images of TiO_xN_y coatings deposited under different deposition regimes: (a) Ar33, $\theta = 51.4^\circ$; (b) Ar28, $\theta = 54.5^\circ$; and (c) Ar26, $\theta = 50.0^\circ$. The colored dashed lines indicate the contact-angle fitting lines generated by the measurement software.

It was found that the contact angle values of all TiO_xN_y -coated samples are in the range of $47\text{--}57^\circ$, indicating hydrophilic surface behavior. The average contact angle is 51.37° for the Ar33 sample, 54.46° for Ar28, and 50.01° for Ar26. The highest average value is observed for Ar28, whereas the lowest value is observed for Ar26.

The obtained results indicate that the deposition regime affects the wettability of TiO_xN_y -coated surfaces. These differences may be associated with changes in surface morphology, coating density, and surface chemical state formed under different combinations of Ar flow rate, relative reactive gas fraction, and substrate bias. However, since quantitative roughness measurements were not performed, the relationship between wettability and surface morphology should be considered qualitative.

The combined variation in Ar flow rate and substrate bias affects plasma characteristics, deposition rate, and ion bombardment conditions. The Ar33 regime is associated with a higher coating thickness, whereas the Ar26 regime, with a higher negative substrate bias and higher relative fraction of reactive gases, promotes the formation of a more homogeneous and dense TiO_xN_y coating structure. This structural uniformity may contribute to the more stable tribological behavior of the Ar26 coating.

The improved corrosion resistance of the Ar28 sample may be related to the formation of a coating structure that provides a more effective barrier against electrolyte penetration and aggressive ion transport toward the substrate. At the same time, the observed differences in the structural, mechanical, tribological, electrochemical, and surface properties of the TiO_xN_y coatings cannot be attributed solely to the Ar flow rate. In the present study, the Ar flow rate and substrate bias were varied simultaneously, while the relative fraction of reactive gases also changed with decreasing Ar flow. Therefore, the results should be interpreted as the combined effect of the deposition regime, including Ar flow rate, relative reactive gas fraction, and bias-controlled ion bombardment.

Although the individual contribution of each deposition parameter cannot be quantitatively separated in the present experimental design, their possible roles can be discussed qualitatively. The Ar flow rate mainly affects plasma density, sputtering yield, collision frequency in the plasma, and the flux of sputtered Ti species arriving at the substrate. Therefore, the higher Ar flow used for the Ar33 regime may have promoted a higher deposition rate, which is consistent with the greatest coating thickness observed for this sample. However, at higher Ar flow, the relative fraction of reactive gases in the Ar- N_2 - O_2 mixture is lower, which may limit the formation of a more homogeneous oxynitride structure and contribute to less stable tribological behavior.

The relative fraction of reactive gases increases when the Ar flow rate decreases, because the N_2 and O_2 flow rates were kept constant at 10 and 6 sccm, respectively. As a result, the Ar26 regime corresponds to a higher relative amount of reactive N_2 and O_2 species in the discharge. This can promote the formation of Ti-O-N-containing phases and influence the defect structure, surface chemical state, and coating density. At the same time, changes in the reactive gas fraction may also affect the deposition rate and phase formation kinetics. Therefore, the thinner Ar28 coating and the denser Ar26 coating can be interpreted as a result of the combined influence of reactive gas fraction, deposition rate, and ion bombardment conditions.

The substrate bias controls the energy of ions impinging on the growing coating surface. A higher negative bias increases ion bombardment energy, which can enhance adatom mobility, promote densification, reduce loosely bonded regions, and improve coating compactness. This effect may explain the more homogeneous structure and stable friction behavior of the Ar26 coating deposited at the highest negative bias. However, excessive ion bombardment can also increase residual stresses, promote re-sputtering, and affect defect formation. Therefore, the improved corrosion resistance of Ar28 may be associated with an intermediate balance between coating thickness, reactive gas fraction, and ion bombardment energy, whereas the stable tribological behavior of Ar26 may be mainly related to stronger bias-assisted densification. Thus, the observed structural, mechanical, tribological, electrochemical, and wettability changes are governed by the coupled action of

Ar flow rate, relative reactive gas fraction, and substrate bias-controlled ion bombardment rather than by any single parameter alone.

It should be noted that this study has certain limitations. Surface morphology was evaluated mainly by SEM, while quantitative surface roughness measurements by AFM or profilometry were not performed. Therefore, the relationship between surface morphology, wettability, tribological behavior, and corrosion resistance should be interpreted qualitatively, since it was not directly supported by quantitative roughness parameters. In addition, XPS analysis was not carried out in this work; therefore, Ti–O–N bonding states, chemical environment, and valence states were not directly determined. The formation of TiO_xN_y phases was discussed based on XRD results and the reactive Ar– N_2 – O_2 deposition conditions. Future studies should include AFM or profilometry and XPS analysis to clarify the role of surface roughness and chemical bonding states in the functional properties of the coatings.

4. Conclusions

The properties of TiO_xN_y coatings deposited on 316L stainless steel are determined by the combined influence of deposition conditions, including the Ar flow rate, relative reactive gas fraction, and substrate bias. These factors affect plasma characteristics, bias-controlled ion bombardment, coating growth kinetics, and structure formation.

In this coupled deposition system, the Ar flow rate mainly affects plasma density, sputtering yield, and deposition rate, whereas the relative fraction of N_2 and O_2 influences Ti–O–N phase formation and surface chemical state. The substrate bias determines the energy of ion bombardment and therefore affects adatom mobility, coating densification, residual stress development, and defect formation.

Among the studied coatings, the Ar26 deposition regime provided the most balanced combination of structural and tribological properties. This coating exhibited a dense and homogeneous structure, high crystallinity, and stable friction behavior with a low coefficient of friction of $\mu \approx 0.26$ – 0.28 . These characteristics may be associated with the combined effect of lower Ar flow, higher relative reactive gas fraction, and higher negative substrate bias.

The Ar33 coating exhibited the greatest thickness and the highest hardness values, which may be related to the higher deposition rate and reduced substrate influence during mechanical testing. However, the presence of impurities and less stable tribological behavior indicate that this regime is not optimal for achieving a balanced combination of functional properties.

The Ar28 coating demonstrated the highest corrosion resistance, with a corrosion current density of $i_{\text{corr}} = 2.82 \times 10^{-7} \text{ A/cm}^2$ and a corrosion rate of $v_{\text{corr}} = 0.00573 \text{ mm/year}$. This may indicate the formation of a more effective barrier layer that reduces electrolyte penetration and aggressive ion transport toward the substrate.

All coatings exhibited hydrophilic surface behavior, with contact angles in the range of 50 – 57° . These results, together with the improved corrosion resistance and tribological stability observed for selected deposition regimes, indicate that TiO_xN_y coatings may be considered promising protective surface layers for 316L stainless steel. However, their suitability for vascular implants or other biomedical applications cannot be confirmed based on the present data alone. Further cytotoxicity, hemocompatibility, endothelialization, and platelet adhesion studies are required to evaluate their biological performance.

It should be noted that in this study, the Ar flow rate and substrate bias were varied simultaneously. Therefore, their individual contributions cannot be fully separated. Future work should include independent variation of these parameters to clarify their separate effects on the structure and functional properties of TiO_xN_y coatings.

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