ORIGINAL STUDY

OPTIMIZATION OF REACTIVE MAGNETRON SPUTTERING PARAMETERS FOR TION COATINGS DEPOSITED ON 316L STAINLESS STEEL

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Abstract. *TiON coatings were deposited on 316L stainless steel substrates by reactive magnetron sputtering under three different deposition regimes. The study aimed to determine the optimal sputtering conditions by comparing the mechanical properties, elemental composition, and corrosion behavior of the coatings. Instrumented indentation showed that the deposition regime strongly affected the mechanical response of the coatings. The highest hardness and elastic modulus were obtained for sample R3, reaching 639.3 HV and 152.6 GPa, respectively. SEM observations confirmed the formation of continuous coating layers, while EDS analysis revealed changes in elemental composition depending on the sputtering regime. The oxygen content decreased from R1 to R3, while nitrogen was detected only in the R3 coating. Electrochemical tests in 3.5 wt.% NaCl solution showed that R1 exhibited the lowest corrosion current density of* 1.13×10^{-7} *A/cm² and the lowest corrosion rate of 0.00231 mm/year. R3 demonstrated intermediate corrosion resistance but the best mechanical performance. Based on the combined mechanical and electrochemical results, the R3 regime was identified as the optimal sputtering condition for obtaining high-hardness TiON coatings with satisfactory corrosion resistance.*

Keywords: TiON coating, reactive magnetron sputtering, 316L stainless steel, microhardness, corrosion resistance, SEM, EDS.

1. Introduction

Titanium oxynitride (TiON or TiO_xN_y) coatings are widely considered promising surface layers for biomedical metallic materials because they combine the advantages of titanium oxide and titanium nitride phases. Titanium oxide contributes to chemical stability, corrosion resistance, and biocompatibility, while titanium nitride improves hardness, wear resistance, and mechanical durability. Therefore, TiON coatings are especially attractive for medical devices that require both biological safety and improved surface strength [1–4].

AISI 316L stainless steel is one of the commonly used metallic materials for biomedical applications due to its good mechanical properties and corrosion resistance. However, under long-term service conditions, its surface may still be exposed to wear, ion release, and degradation in physiological environments. Surface modification by protective coatings can improve the functional stability of 316L stainless steel and extend its potential use in biomedical devices [5, 6].

Reactive magnetron sputtering is an effective method for depositing TiON coatings because it allows the formation of thin, uniform, and dense films at relatively low substrate temperatures. This method also provides control over the coating composition and structure by varying technological parameters such as the $Ar/N_2/O_2$ gas flow rate, working pressure, discharge voltage, and substrate bias [7, 8].

The deposition parameters strongly influence the phase composition, surface morphology, density, mechanical properties, and corrosion behavior of TiON coatings. In particular, changes in the reactive gas atmosphere can affect the formation of titanium nitride, titanium oxide, and titanium oxynitride phases. These

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structural and compositional differences may lead to significant changes in microhardness, stiffness, and electrochemical stability [9].

Despite the active development of TiON coatings, the selection of optimal deposition conditions remains an important task. Many studies focus on the influence of individual parameters; however, the comparative evaluation of selected deposition regimes is still necessary to determine conditions that provide a favorable combination of mechanical properties and corrosion resistance. Therefore, the optimization of reactive magnetron sputtering parameters is important for obtaining TiON coatings with stable structure, high hardness, and improved protective performance.

The aim of this work is to optimize the technological parameters of reactive magnetron sputtering for TiON coatings deposited on 316L stainless steel. For this purpose, three deposition regimes were compared, and the relationship between sputtering parameters, surface morphology, elemental composition, mechanical properties, and corrosion behavior of the coatings was analyzed.

2. Materials and Methods

A 99.9% pure Ti target was used to deposit TiON coatings. The coatings were deposited on 316L stainless steel substrates. AISI 316L stainless steel is widely used in engineering and biomedical applications due to its favorable combination of mechanical properties, corrosion resistance, and manufacturability. However, its surface hardness and wear resistance are often insufficient for long-term service under demanding operating conditions. Therefore, surface modification by hard protective coatings is considered an effective approach to improve the performance and durability of 316L stainless steel.

The substrate dimensions were 30 mm × 30 mm × 4 mm. Prior to sputtering, the substrate surface was progressively ground using abrasive paper with grit sizes from P400 to P2500.The chemical composition of the 316L stainless steel substrate was taken from 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, wt.%.

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	Balance

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. During ion cleaning, the argon flow rate was 15 sccm, and the working pressure in the chamber was maintained at 0.2–0.3 Pa. The cleaning process was carried out at an applied voltage of 2.5 kV and a current of 49–80 mA for 20 min.

TiON coatings were obtained by reactive magnetron sputtering of a titanium target in an Ar + N_2 + O_2 atmosphere. In this study, three deposition regimes were selected for comparative analysis in order to determine the optimal sputtering conditions for obtaining high-hardness TiON coatings. The selected samples differed in the composition of the reactive gas atmosphere, operating pressure, and discharge voltage, while the substrate bias was kept constant at −100 V. The deposition time was 3 h 45 min for all coating regimes. The main sputtering parameters used for the selected deposition regimes are summarized in Table 2.

Table 2. Magnetron sputtering parameters for TiON coating deposition.

Parameter	R1	R2	R3
Ar flow rate, sccm	25	25	33
N_2 flow rate, sccm	3.0	5.5	12.3
O_2 flow rate, sccm	1.5	10.9	4.1
N:O ratio	2	0.5	3
Substrate bias, V	−100		
Operating pressure, Pa	1.2–2.0	0.8–1.0	0.8–1.0
Discharge voltage, V	417–450	440–459	468–483
Current, A	2		
Target material	Ti, 99.9%		
Substrate temperature, °C	100		
Target–substrate distance, mm	30		

Table 2. (continued)

Parameter	**R1**	**R2**	**R3**
Deposition time	3 h 45 min		

The ion energy at the substrate is mainly controlled by the applied substrate bias, which was kept constant at −100 V in all selected regimes. Therefore, the observed changes in coating properties can mainly be associated with variations in the reactive gas atmosphere, operating pressure, and discharge voltage. However, the base pressure, target diameter, and target power density were not available in the experimental log and therefore were not included.

The surface microstructure and morphology of the coatings were examined using a TESCAN VEGA4 LMH scanning electron microscope (SEM) (TESCAN, Brno, Czech Republic). Energy-dispersive X-ray spectroscopy (EDS) was performed using an Xplore 30 system (Oxford Instruments, Oxford, UK).

Coating hardness was measured using a FISCHERSCOPE HM 2000 system (Helmut Fischer GmbH, Sindelfingen, Germany) controlled by WIN-HCU software version 7.1. The measurements were carried out in accordance with ISO 14577 [10]. The dwell time was 10 s at a load of 2000 mN.

Corrosion resistance was investigated using a CS300M potentiostat-galvanostat. The coatings were tested with an exposed area of 1 cm² at room temperature (25 °C) in a 3.5 wt.% NaCl solution. The experiment was performed using a three-electrode cell, where the TiON-coated sample was used as the working electrode, 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 immersed in the electrolyte for 60 min until a stable open-circuit potential (OCP) was established. The potential was scanned in the range from −0.1 to +0.1 V relative to OCP at a scan rate of 0.5 mV/s. The corrosion potential and corrosion current density were obtained from the polarization curves using the Tafel extrapolation method. The tests were repeated three times, and the results were analyzed using CS Studio6 software, version 6.3.

The obtained mechanical and electrochemical parameters were used to compare the selected deposition regimes and determine the optimal sputtering conditions for TiON coatings.

3. Results and Discussion

The mechanical response of the TiON coatings deposited under different sputtering regimes was evaluated by instrumented indentation. The corresponding load-depth curves and calculated mechanical parameters are presented in Fig. 1 and Table 3.

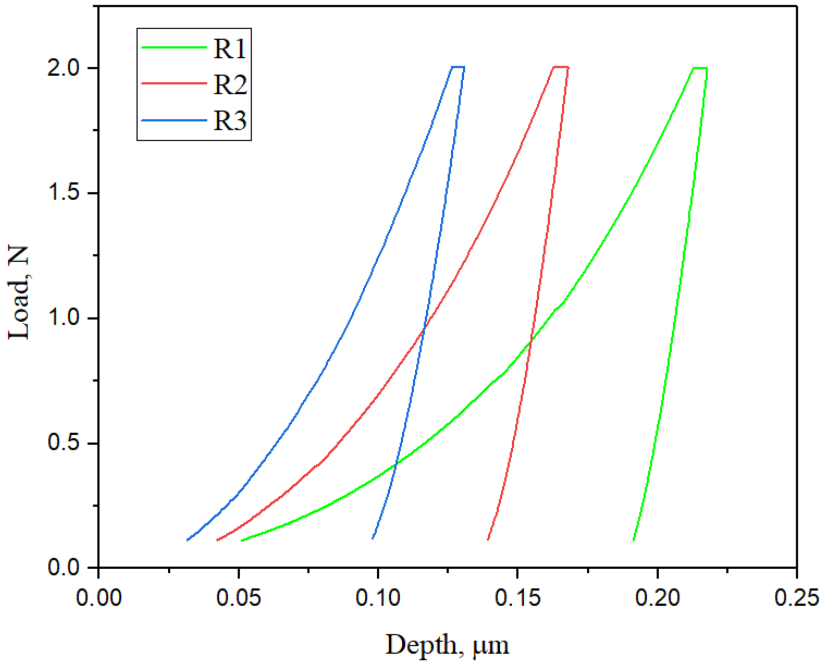


Fig. 1. Instrumented indentation load–depth curves of TiON coatings deposited under R1, R2, and R3 sputtering regimes.

Table 3. Mechanical properties of TiON coatings deposited under different sputtering regimes.

Sample	**HM, MPa**	**HV**	**E, GPa**
R1	1647.8 ± 86.2	197.7 ± 13.7	93.5 ± 4.9
R2	2829.2 ± 191.1	350.8 ± 34.2	126.0 ± 4.2
R3	4682.8 ± 303.1	639.3 ± 48.1	152.6 ± 6.3

The load–depth curves demonstrate a clear dependence of indentation behavior on the deposition regime. At the maximum applied load of 2 N, the penetration depth decreased in the following order: R1 > R2 > R3. The highest penetration depth was observed for R1, indicating the lowest resistance to indentation deformation. In contrast, R3 exhibited the smallest penetration depth, which confirms its higher resistance to plastic deformation.

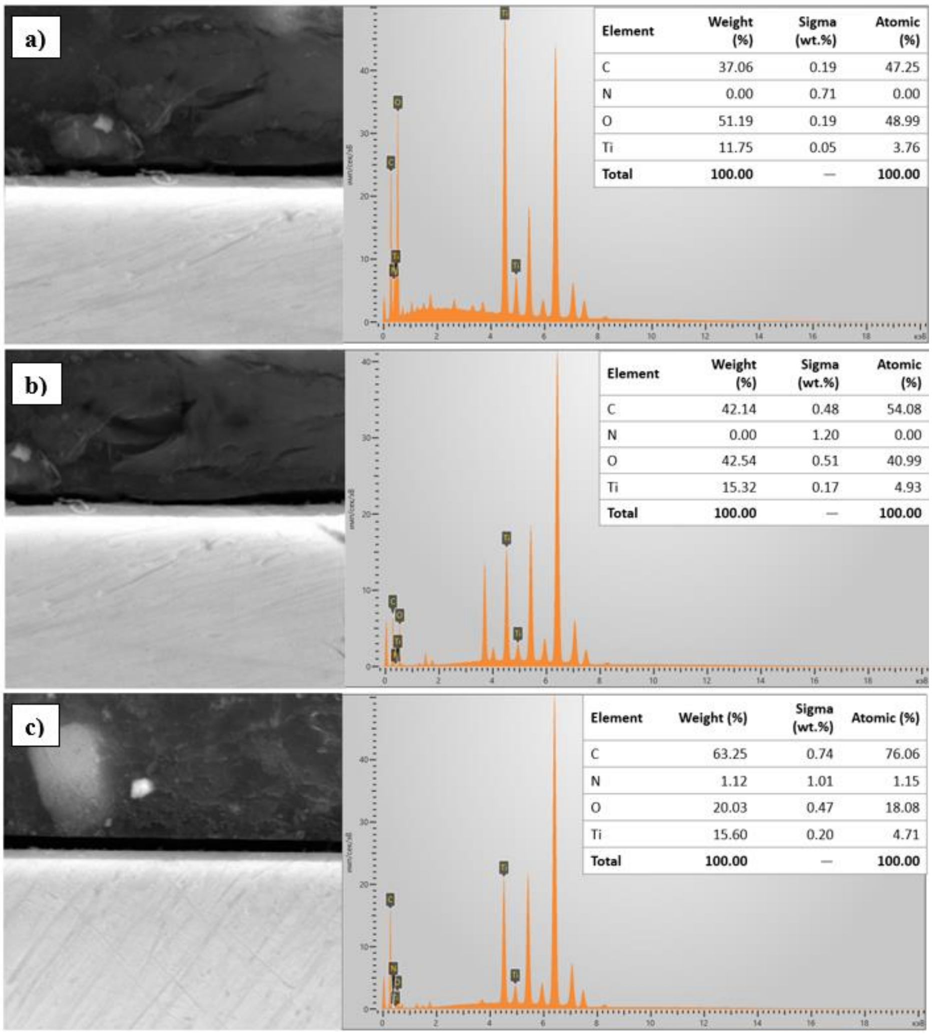
The calculated hardness values also showed a consistent increase from R1 to R3. The Martens hardness increased from 1647.8 ± 86.2 MPa for R1 to 2829.2 ± 191.1 MPa for R2 and reached 4682.8 ± 303.1 MPa for R3. A similar trend was observed for Vickers hardness, which increased from 197.7 ± 13.7 HV for R1 to 350.8 ± 34.2 HV for R2 and 639.3 ± 48.1 HV for R3. Thus, the hardness of the R3 coating was more than three times higher than that of R1.

The elastic modulus also increased with the change in deposition regime, from 93.5 ± 4.9 GPa for R1 to 126.0 ± 4.2 GPa for R2 and 152.6 ± 6.3 GPa for R3. This increase indicates that the R3 coating has a stiffer structure and higher resistance to elastic deformation compared with the other samples.

The improvement in hardness and elastic modulus can be attributed to changes in the coating structure caused by the different sputtering conditions. Since the substrate bias was kept constant at −100 V for all samples, the observed differences are mainly related to variations in the $Ar/N_2/O_2$ reactive gas atmosphere, operating pressure, and discharge voltage. The highest mechanical properties of R3 suggest the formation of a denser TiON coating structure and a greater contribution of hard titanium nitride/oxynitride phases.

Overall, the indentation results indicate that the R3 deposition regime provides the most favorable mechanical performance among the investigated coatings. Therefore, R3 can be considered the optimal regime from the viewpoint of coating hardness and stiffness.

Fig. 2 presents the cross-sectional SEM micrographs, EDS spectra, and elemental compositions of the TiON coatings deposited under different sputtering regimes. The SEM images reveal the formation of continuous surface layers on the 316L stainless steel substrate. In all samples, the coating/substrate interface can be distinguished, indicating successful deposition of the TiON coatings.



Element	Weight (%)	Sigma (wt.%)	Atomic (%)
C	37.06	0.19	47.25
N	0.00	0.71	0.00
O	51.19	0.19	48.99
Ti	11.75	0.05	3.76
Total	**100.00**	—	**100.00**

Element	Weight (%)	Sigma (wt.%)	Atomic (%)
C	42.14	0.48	54.08
N	0.00	1.20	0.00
O	42.54	0.51	40.99
Ti	15.32	0.17	4.93
Total	**100.00**	—	**100.00**

Element	Weight (%)	Sigma (wt.%)	Atomic (%)
C	63.25	0.74	76.06
N	1.12	1.01	1.15
O	20.03	0.47	18.08
Ti	15.60	0.20	4.71
Total	**100.00**	—	**100.00**

Fig. 2. Cross-sectional SEM micrographs, EDS spectra, and elemental compositions of TiON coatings deposited under different sputtering regimes: (a) R1, (b) R2, and (c) R3.

No significant coating delamination or large-scale defects were observed at the coating/substrate interface. The cross-sectional images suggest that the deposition regime influences the morphology of the deposited layers; however, due to the limited contrast between the coating and substrate, a detailed assessment of coating density and microstructural features requires additional high-magnification observations.

The EDS spectra confirm the presence of titanium and oxygen in all investigated coatings. Quantitative analysis revealed a gradual decrease in oxygen content from 51.19 wt.% for R1 to 42.54 wt.% for R2 and 20.03 wt.% for R3. Simultaneously, the titanium concentration increased from 11.75 wt.% to 15.60 wt.%. Nitrogen was detected only in the R3 coating, where its concentration reached 1.12 wt.%.

The observed compositional changes indicate that the deposition regime significantly affects the chemical composition of the TiON coatings. The reduced oxygen content and the appearance of nitrogen in sample R3 suggest enhanced nitride or oxynitride formation, which may contribute to the superior mechanical properties observed during indentation testing.

The corrosion behavior of the TiON coatings was evaluated by potentiodynamic polarization measurements in a 3.5 wt.% NaCl solution. The corresponding polarization curves are presented in Fig. 3, while the electrochemical parameters obtained by Tafel extrapolation are summarized in Table 4.

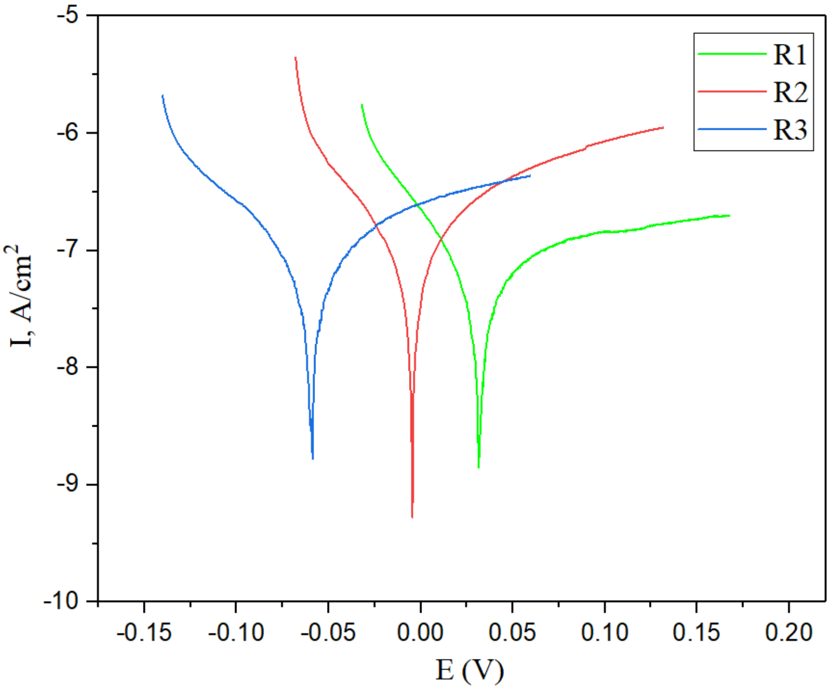


Fig. 3. Potentiodynamic polarization curves of TiON coatings deposited under different sputtering regimes in 3.5 wt.% NaCl solution.

Table 4. Electrochemical corrosion parameters of TiON coatings obtained from Tafel extrapolation.

Parameter	R1	R2	R3
E_{corr} (mV)	31.71	−4.55	−59.34
i_{corr} (A/cm^2)	1.13×10^{-7}	2.22×10^{-7}	1.79×10^{-7}
β_a (mV)	544.63	178.36	279.81
β_c (mV)	66.57	93.51	111.69
v_{corr} (mm/year)	0.00231	0.00451	0.00365

The polarization curves demonstrate that the deposition regime significantly affects the electrochemical behavior of the coatings. The corrosion potential (Ecorr) shifted from 31.71 mV for R1 to −4.55 mV for R2 and −59.34 mV for R3. Although the corrosion potential became more negative for R2 and R3, the corrosion resistance is more reliably evaluated using the corrosion current density (i_{corr}) and corrosion rate (V_{corr}).

Among the investigated coatings, R1 exhibited the lowest corrosion current density of 1.13×10^{-7} A/cm^2 and the lowest corrosion rate of 0.00231 mm/year. In contrast, R2 showed the highest corrosion current density (2.22×10^{-7} A/cm^2) and the highest corrosion rate (0.00451 mm/year), indicating the lowest corrosion resistance. Sample R3 demonstrated intermediate behavior with an icorr value of 1.79×10^{-7} A/cm^2 and a corrosion rate of 0.00365 mm/year.

The improved corrosion performance of R1 may be associated with the higher oxygen content detected by EDS analysis. The increased oxygen concentration can promote the formation of a more protective oxide-rich surface layer, which acts as a barrier against electrolyte penetration. In contrast, the lower oxygen content observed in R3 and especially the compositional changes in R2 may reduce the protective effect of the coating, resulting in higher corrosion activity.

The obtained results indicate that the deposition regime has a significant influence on the electrochemical performance of TiON coatings. While R3 exhibited the best mechanical properties, R1 provided the highest corrosion resistance. Therefore, the selection of the optimal deposition regime requires consideration of both mechanical and electrochemical characteristics depending on the intended application.

A comparative analysis of the mechanical and electrochemical properties of the TiON coatings indicates that the deposition regime has a significant influence on coating performance. The investigated coatings exhibited different combinations of hardness and corrosion resistance depending on the sputtering conditions.

Sample R1 demonstrated the highest corrosion resistance, exhibiting the lowest corrosion current density (1.13×10^{-7} A/cm²) and the lowest corrosion rate (0.00231 mm/year). However, its mechanical properties were the lowest among the investigated coatings, with a hardness of 197.7 HV and an elastic modulus of 93.5 GPa.

Sample R2 showed intermediate mechanical properties but the highest corrosion activity, as indicated by the maximum corrosion current density (2.22×10^{-7} A/cm²) and corrosion rate (0.00451 mm/year). Therefore, this deposition regime cannot be considered optimal from either a mechanical or electrochemical perspective.

The best mechanical performance was obtained for sample R3. This coating exhibited the highest hardness (639.3 HV), the highest Martens hardness (4682.8 MPa), and the highest elastic modulus (152.6 GPa). Although its corrosion resistance was slightly lower than that of R1, the corrosion rate remained low (0.00365 mm/year), indicating acceptable electrochemical stability.

EDS analysis revealed a decrease in oxygen content and the appearance of nitrogen in sample R3, suggesting enhanced formation of titanium oxynitride and nitride-containing phases. These compositional changes are consistent with the significant improvement in hardness and stiffness observed during indentation testing.

Considering the overall balance between mechanical and electrochemical properties, the R3 deposition regime can be regarded as the optimal sputtering condition among the investigated samples. This regime provides a substantial improvement in hardness while maintaining satisfactory corrosion resistance, making it the most promising candidate for protective TiON coatings on 316L stainless steel.

4. Conclusions

TiON coatings were successfully deposited on 316L stainless steel substrates by reactive magnetron sputtering under three different deposition regimes (R1–R3). The influence of sputtering parameters on the mechanical properties, elemental composition, and corrosion behavior of the coatings was investigated.

Instrumented indentation measurements revealed a significant dependence of the mechanical properties on the deposition regime. The highest hardness and elastic modulus were obtained for sample R3, reaching 639.3 HV and 152.6 GPa, respectively.

SEM observations confirmed the formation of continuous coating layers on the substrate surface. EDS analysis demonstrated changes in elemental composition with increasing nitrogen incorporation, accompanied by a decrease in oxygen content from R1 to R3.

Electrochemical measurements in 3.5 wt.% NaCl solution showed that sample R1 exhibited the highest corrosion resistance with the lowest corrosion current density (1.13×10^{-7} A/cm²) and corrosion rate (0.00231 mm/year). Sample R2 showed the lowest corrosion performance, whereas R3 demonstrated intermediate corrosion behavior.

Considering the combined mechanical and electrochemical characteristics, the R3 deposition regime was identified as the optimal sputtering condition. This regime provided the highest hardness while maintaining satisfactory corrosion resistance, making it a promising candidate for protective TiON coatings on 316L stainless steel.

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Conflict of interest

The authors declare that they have no competing interests.

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