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ORIGINAL STUDY

EFFECT OF ARGON FLOW RATE DURING AIR PLASMA SPRAYING ON THE STRUCTURE AND PROPERTIES OF NiCrFeBSiC COATINGS

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Abstract. In this work, self-fluxing metal-ceramic coatings based on the NiCrFeBSiC system were obtained by air plasma spraying, and their structural, mechanical, and tribological properties were investigated. The effect of argon flow rate (1600 and 1800 L/h) during coating formation was considered. It was found that an increase in argon flow rate leads to an approximately twofold increase in coating thickness and a twofold decrease in porosity. According to the microhardness measurement results, the microhardness was 1015.8 ± 81.5 HV, while the elastic modulus was 203.9 ± 5.4 GPa. Tribological tests were carried out using the ball-on-disk configuration. At an argon flow rate of 1800 L/h, the friction coefficient showed a lower value (≈ 0.107), indicating improved wear resistance of the coatings. Scratch test results showed a high level of adhesion between the coatings and the substrate. Although the critical load values (L_c) were similar for both regimes, the coatings obtained at 1800 L/h exhibited a more gradual and stable failure behavior.

Keywords: air plasma spraying, self-fluxing metal-ceramic coatings, wear resistance, hardness, friction, adhesion.

1. Introduction

In modern industry, the performance and durability of components used in mechanical engineering and agriculture directly depend on the structure and properties of their surface layers [1]. During operation, components are exposed to friction, high temperature, and corrosion, which lead to rapid wear and failure [2]. This, in turn, leads to increased production costs [3]. Therefore, surface treatment of machine components with protective functional coatings to increase their service life is one of the important areas of modern scientific and technical research [4-6].

Among protective coatings, nickel-based self-fluxing alloys, particularly NiCrFeBSiC system alloys, are distinguished by their high efficiency. Due to the presence of boron and silicon in their composition, these alloys have a relatively low melting temperature and can form a dense, defect-free phase structure during remelting [7]. Nickel-based coatings are characterized by high adhesion to the metallic substrate, as well as high wear and corrosion resistance. These properties make them suitable for effective use under severe operating conditions [5].

Currently, one of the most widely used and effective methods for producing such coatings is air plasma spraying (APS). This method is based on using a high-temperature plasma jet to fully or partially melt the powder material and spray it onto the treated surface at high velocity. As a result, dense and multiphase coating layers are formed [8]. The microstructure of the obtained coatings is usually a complex system consisting of boron and silicon compounds dispersed in a nickel-based matrix. The main advantages of air plasma spraying include a wide range of processable materials, precise control of coating thickness and structure, and high production efficiency [9]. At the same time, the mechanical, tribological, and corrosion properties of the coatings depend on spraying parameters such as temperature, gas flow rate, and spraying distance, as well as powder particle size and subsequent heat treatment conditions [10].

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Recent studies show that plasma spraying of nickel-based self-fluxing coatings improves microstructural uniformity, reduces porosity, and enhances mechanical properties [11]. In addition, subsequent heat treatment or remelting processes increase the coating density and significantly improve its service performance. However, the effect of plasma gas flow rate, particularly argon flow rate, as one of the key technological parameters in the air plasma spraying process, on the structure and properties of coatings has not been sufficiently studied. In the literature, data on the effect of this parameter on coating thickness, porosity, and phase composition are limited and ambiguous, for example, in [12], the deposition of self-fluxing nickel-based coatings on cast iron components was investigated, and heat treatment was shown to increase their wear resistance by 1.5–2 times. However, the systematic effect of plasma spraying parameters, including gas flow rate, has not been sufficiently considered.

Therefore, the main objective of this work is to comprehensively investigate the effect of argon flow rate on the structure, mechanical properties, and tribological performance of NiCrFeBSiC coatings obtained by air plasma spraying, and to determine the optimal spraying parameters.

2. Materials and methods

In this study, Chinese-manufactured self-fluxing nickel powder based on the NiCrFeBSiC system was used as the coating material. The powder particles were mainly spherical, with an average size of approximately 150 μm , which ensured good flowability and uniform feeding during spraying.

Carbon structural steel grade 45, widely used in mechanical engineering, was used as the substrate material for coating deposition; its chemical composition is presented in Table 1, and the material is characterized by a combination of high strength and ductility. Samples were prepared from a steel rod with a diameter of 50 mm and fabricated in the form of discs with a thickness of 5 mm.

Table 1. Chemical composition of grade 45 steel.

C	Si	Mn	Ni	S	P	Cr	Cu
0.42 - 0.5	0.17 - 0.37	0.5 - 0.8	<0.3	<0.04	<0.035	<0.25	<0.3

Before coating deposition, the sample surfaces were specially prepared: first, they were ground using abrasive papers with different grit sizes (120–600), followed by sandblasting with electrocorundum particles of approximately 40 μm to improve adhesion. After treatment, the surfaces were cleaned with compressed air and then washed in an ethanol solution in an ultrasonic bath for 10 min at a frequency of 40 kHz and a temperature not exceeding 35 $^{\circ}\text{C}$, which ensured the complete removal of grease and other contaminants.

The coatings were deposited by air plasma spraying using an SX-60 system. The following technological parameters were used during spraying: arc current of 550 A, hydrogen flow rate of 150 L/h, argon flow rates of 1600 and 1800 L/h, spraying distance of 120 mm, and powder feed rate of 5 g/min. During the study, two different regimes with argon flow rates of 1600 and 1800 L/h were comparatively investigated.

The microstructural characterization of the as-deposited coatings was performed by scanning electron microscopy (SEM) in backscattered electron (BSE) mode, providing enhanced phase contrast. The elemental composition and spatial distribution of constituent elements were analyzed using energy-dispersive spectroscopy (EDS). These complementary techniques enabled a comprehensive evaluation of coating porosity, particle melting state, and microstructural homogeneity.

The mechanical properties of the coatings were evaluated by instrumented microindentation using a Fischerscope HM2000S system. During testing, a load of 1000 mN was progressively applied to the sample surface over 20 s using a diamond pyramidal indenter. The loading process was conducted under strictly controlled conditions, allowing continuous recording of the material's mechanical response throughout the loading cycle. The primary measured parameters were hardness (HV) and elastic modulus (E). Additionally, the load-indentation depth relationship was recorded and analyzed during the experiments.

The tribological properties were evaluated using a TRB³ tribometer under lubricated conditions in a ball-on-disk configuration (Fig. 1). A 100Cr6 steel ball with a diameter of 3 mm was used as the counterbody. The tests were conducted at a sliding radius of 2 mm, a sliding speed of 5 cm/s, a normal load of 10 N, and a total sliding distance of 200 m. As a result, the evolution of the coefficient of friction (CoF) was determined.

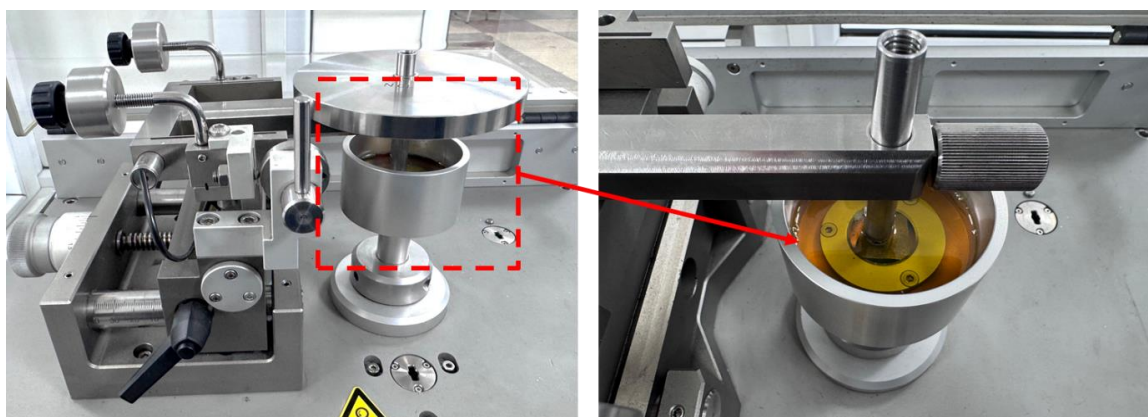


Fig.1. General view of the TRB3 tribometer for conducting tribological tests.

The adhesive strength of the coatings was evaluated by scratch testing using an RST300 instrument. During the test, a load of up to 100 N was applied to a diamond indenter, which was translated across the sample surface at a constant speed of 6 mm/min over a scratch length of 6 mm. Throughout the test, the failure behavior at the coating–substrate interface was continuously monitored. Critical loads corresponding to different stages of coating failure were determined, including the initiation of microcracks, crack propagation, and complete delamination of the coating layer. In addition, the friction force, indenter penetration depth, and acoustic emission signals were continuously recorded, enabling a comprehensive assessment of the mechanical stability of the coatings.

3. Results and discussion

Scanning electron microscopy (SEM) results demonstrated that the argon flow rate during air plasma spraying significantly affected the microstructure of the coatings (Fig. 2 and Fig. 3). At an argon flow rate of 1600 L/h, structural heterogeneity was observed in the cross-section of the obtained coating (Fig. 2). SEM images clearly reveal a considerable number of pores and incompletely melted particles. The average coating thickness was approximately 181 μm , while the porosity level was about 2%. The structure shows clearly visible interparticle boundaries, indicating relatively weak bonding between the particles. Most of the pores have a rounded shape and are mainly located between partially unmelted powder particles. In addition, microcracks and intergranular gaps were observed. These features indicate insufficient thermal input during spraying and a low degree of particle melting. According to the energy-dispersive spectroscopy (EDS) results, the main elements (Ni, Cr, Fe, B, Si, and C) were relatively uniformly distributed throughout the coating volume. Nickel predominates as the matrix phase, while chromium is uniformly distributed and contributes to the protective properties of the coating. Silicon and boron were observed to be concentrated in separate phases. The detection of oxygen indicates the occurrence of oxidation processes on the coating surface and in the pore regions.

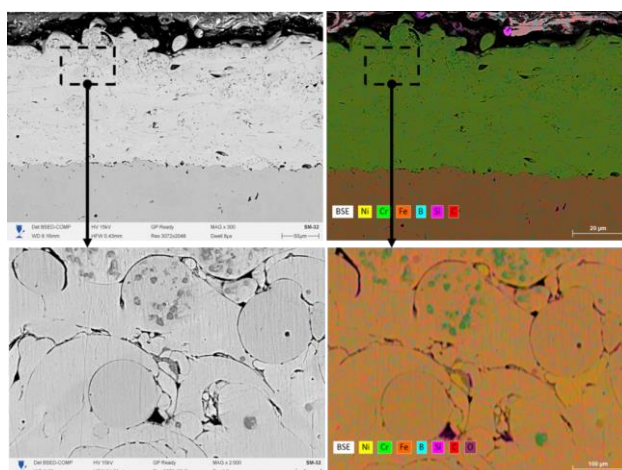


Fig. 2. Structure of the NiCrFeBSiC coating obtained by air plasma spraying at an argon flow rate of 1600 L/h.

At an argon flow rate of 1800 L/h, a significant improvement in the coating microstructure was observed (Fig. 3). The coating thickness was approximately 250 μm , which was about 40% higher compared with the coating obtained at 1600 L/h. SEM images show that the coating structure is dense, uniform, and composed of well-melted particles. A significant decrease in porosity was observed in the cross-section. Higher-magnification images reveal the formation of a fine-grained and compact microstructure. Although some pores remain, they are smaller in size and are uniformly distributed throughout the coating. Energy-dispersive spectroscopy (EDS) results showed a uniform distribution of elements (Ni, Cr, Fe, and Si) throughout the coating volume. The interface between the coating and the substrate was clearly observed, while no distinct transition layer was detected. A relatively high oxygen content was detected in the surface regions of the coating, which may be associated with high-temperature interaction with the atmospheric environment during plasma spraying. The obtained results indicate that increasing the argon flow rate enhances the degree of particle melting and improves the coating density and structural homogeneity.

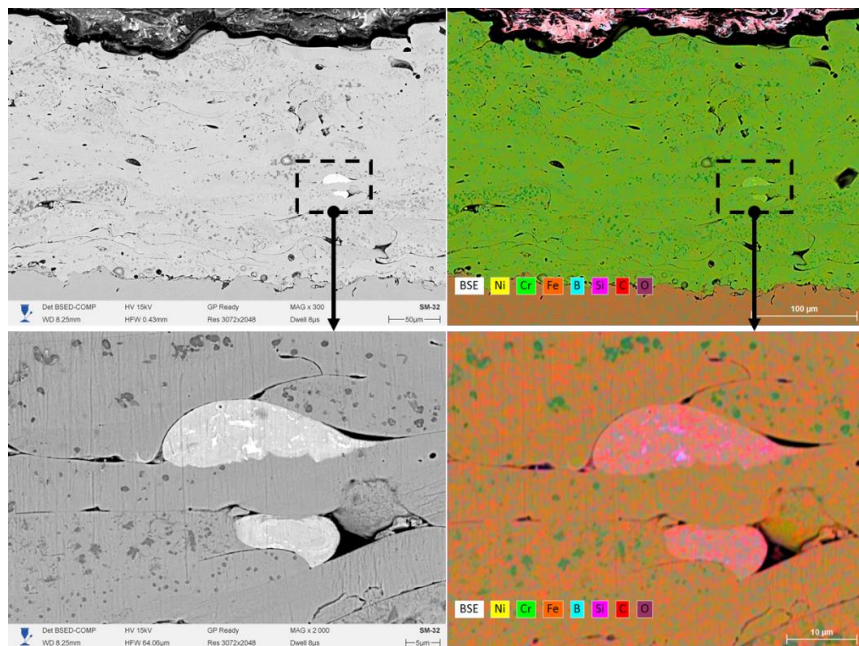


Fig. 3. Structure of the NiCrFeBSiC coating obtained by air plasma spraying at an argon flow rate of 1800 L/h.

Fig. 4 presents the microhardness and elastic modulus results of the coatings obtained by air plasma spraying. The graph illustrates the dependence of indenter penetration depth on the applied load. According to the curves in the graph, the red line corresponds to the 1800 L/h regime, while the black line represents the 1600 L/h regime. When the argon flow rate was increased to 1800 L/h, a decrease in the indenter penetration depth was observed under the same applied load. This indicates an increase in the hardness and elastic modulus of the coating. At an argon flow rate of 1600 L/h, the coating microhardness was 744.7 ± 111.7 HV, while the elastic modulus was 161.7 ± 14.3 GPa. At the 1800 L/h regime, these values increased to 1015.8 ± 81.5 HV and 203.9 ± 5.4 GPa, respectively. The obtained results indicate that increasing the argon flow rate significantly improves the mechanical properties of the coatings. This may be attributed to more complete and uniform particle melting, the formation of a denser structure, and reduced porosity.

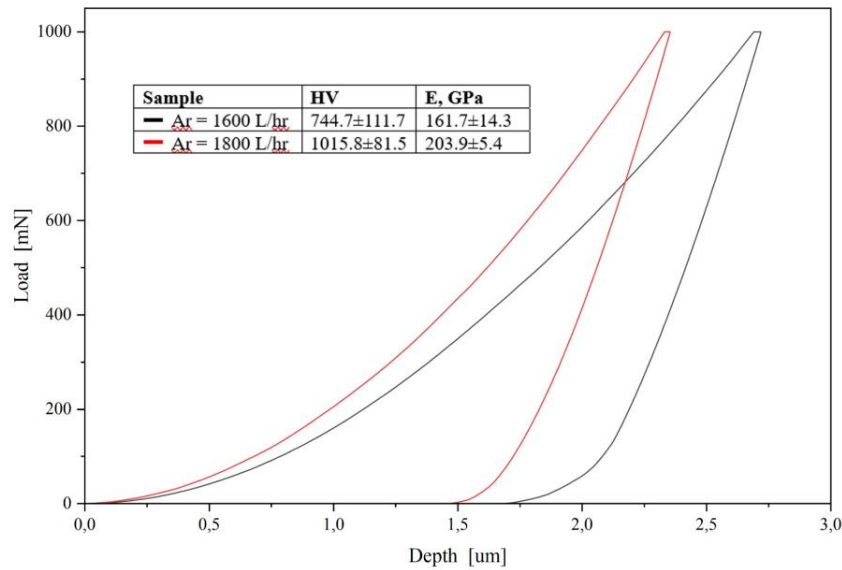


Fig. 4. Load-indentation penetration depth dependence curve.

[Fig. 5](#) shows the results of tribological testing of NiCrFeBSiC coatings obtained by air plasma spraying. The tests were carried out under lubricated conditions using a ball-on-disk configuration. The figure shows that the coefficient of friction for both coatings remained relatively stable throughout the test. At an argon flow rate of 1800 L/h, the coefficient of friction of the coating represented by the red line stabilized at approximately 0.107, showing a lower value compared with the coating obtained at 1600 L/h, represented by the black line. The decrease in the coefficient of friction with increasing argon flow rate may be attributed to the improved coating structure, namely reduced porosity, better particle melting, and increased microhardness.

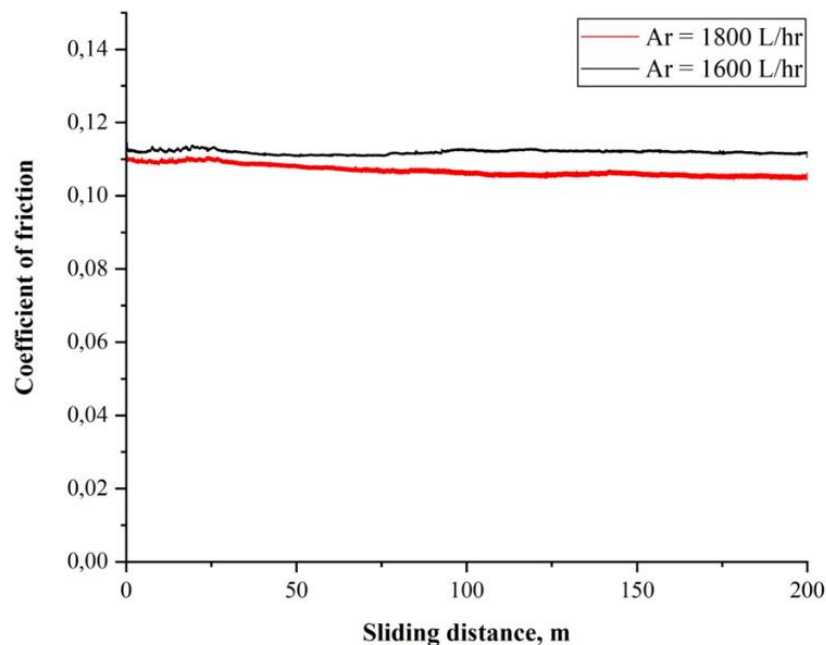


Fig. 5. Dependence of the coefficient of friction on sliding distance for NiCrFeBSiC coatings obtained at argon flow rates of 1600 and 1800 L/h.

Significant differences were observed in the adhesion strength test results of the NiCrFeBSiC coatings ([Fig. 6](#)). For the coating obtained at an argon flow rate of 1600 L/h, the critical loads were $L_{c1} = 32.44$ N, $L_{c2} = 68.43$ N, and $L_{c3} = 99.16$ N. These values characterize the coating failure stages: initiation of microcracks, crack propagation and partial delamination, followed by complete failure. Within the scratch length range of

2–5 mm, sharp changes in the coefficient of friction, friction force, penetration depth, and acoustic emission signal were observed, indicating the onset of coating degradation. The scratch track morphology confirms the presence of localized failure and delamination regions, with pronounced loss of coating integrity observed particularly at the final stage of the test. For the coating obtained at an argon flow rate of 1800 L/h, similar critical load values were recorded: $L_{c1} = 33.57$ N, $L_{c2} = 67.75$ N, and $L_{c3} = 99.61$ N. However, the failure behavior was more stable and uniform. The uniform distribution of defects along the scratch track and the absence of pronounced delamination zones indicate improved adhesion between the coating and the substrate. Scratch test results demonstrate that the argon flow rate directly affects the adhesion behavior of the coatings. Although the absolute values of the critical loads were similar for both regimes, the coating obtained at 1800 L/h exhibited a more uniform and stable failure behavior. This can be explained by the increase in the thermal energy of the plasma jet at a higher argon flow rate: the particles melt more completely, the splats bond more tightly with the substrate, and the cohesive strength increases. As a result, the mechanical load applied to the coating surface is distributed more uniformly, reducing the probability of local stress concentration.

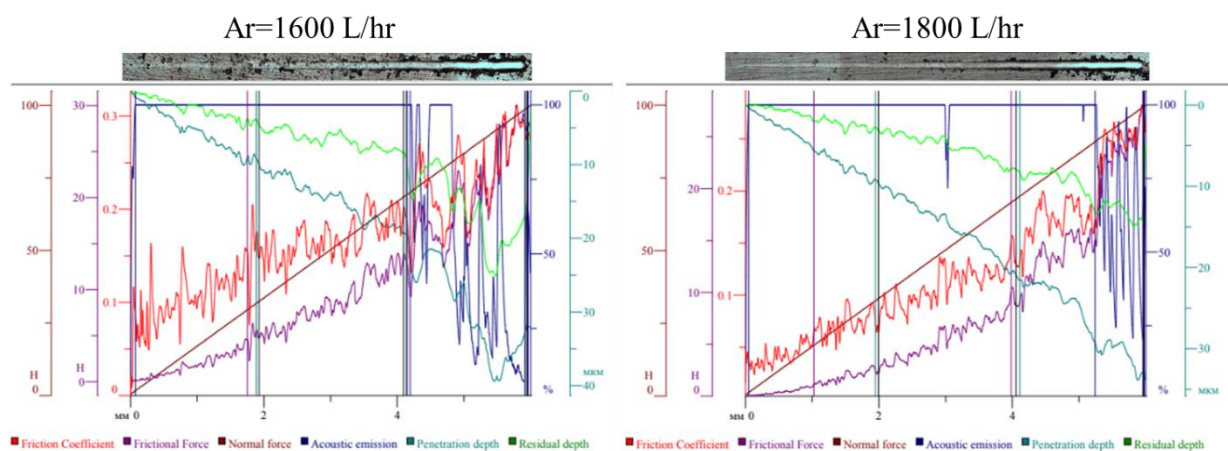


Fig. 6. Adhesion strength test results of NiCrFeBSiC coatings.

4. Conclusion

As a result of the study, it was established that the structure, mechanical properties, and tribological performance of self-fluxing NiCrFeBSiC-based coatings obtained by air plasma spraying directly depend on the argon flow rate. It was shown that the coating obtained at an argon flow rate of 1600 L/h exhibited structural heterogeneity, higher porosity, and incompletely melted particles, which led to reduced coating density and strength. These factors, in turn, resulted in poorer adhesion properties and lower service performance of the coating. When the argon flow rate was increased to 1800 L/h, a significant improvement in the coating microstructure was observed. The coating became denser, porosity decreased, and the degree of particle melting increased. As a result, the microhardness (1015.8 ± 81.5 HV) and elastic modulus (203.9 ± 5.4 GPa) increased, leading to enhanced mechanical properties. Tribological tests showed that increasing the argon flow rate led to a decrease in the coefficient of friction from 0.112 to 0.107, confirming improved wear resistance of the coating. In addition, although the critical load values obtained from scratch testing were comparable for both regimes, the coating deposited at 1800 L/h exhibited a more uniform and stable failure behavior. Thus, using an argon flow rate of 1800 L/h improves the structural integrity, mechanical properties, and tribological performance of NiCrFeBSiC coatings. The obtained results indicate that this regime is effective for producing wear-resistant and reliable coatings under industrial conditions.

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Conflict of interest

The authors declare that they have no competing interests.

References

- 1 Bukaeva, A. Z., Bulekbaeva, G. Zh., Madiyarova, A. S., Chazhabayeva, M. M., & Bilashova, G. S. (2024). *Study of modern methods for applying protective coatings to the metal surfaces of machine parts*. Yessenov University, 133(4), 47–54. DOI: <https://doi.org/10.52167/1609-1817-2024-133-4-47-54>. [In RUS]
- 2 Bulekbaeva, G. Zh., Tabylov, A. O., Kozhabekova, P. A., Bukaeva, A. Z., & Suyeuova, N. B. (2024). *Comprehensive assurance of surface layer quality parameters of parts using an innovative method of combined finishing-strengthening treatment*. L.N. Gumilyov Eurasian National University, 146(1), 8–20. DOI: <https://doi.org/10.32523/2616-7263-2024-146-1-8-20>. [ENG]
- 3 Smagulova, N. K., Kurmangaliyeva, L. A., & Iskakova, T. K. (2024). Review of studies of corrosion of metals and alloys. *Engineering Journal of Satbayev University*, 146(2), 1–8. DOI: <https://doi.org/10.51301/ejsu.2024.i2.01>. [ENG]
- 4 Khabay, A. (2024). Research of modern technologies for application of wear-resistant coatings on machine parts. *Yessenov Science Journal*, 47(2). DOI: <https://doi.org/10.56525/YJRK6370>. [ENG]
- 5 Li, C.-J., Luo, X.-T., Dong, X.-Y., Zhang, L., & Li, C.-X. (2022). Recent research advances in plasma spraying of bulk-like dense metal coatings with metallurgically bonded lamellae. *Journal of Thermal Spray Technology*, 31(1–2), 5–27. DOI: <https://doi.org/10.1007/s11666-022-01327-x> [ENG]
- 6 Buitkenov, D., Raisov, N., Alimbekuly, T., & Alibekova, B. (2025). Study of the tribological properties of self-fluxing nickel-based coatings obtained by gas-flame spraying. *Crystals*, 15, 862. DOI: <https://doi.org/10.3390/cryst15100862>. [ENG]
- 7 Skulev, H., Malinov, S., Sha, W., & Basheer, P. A. M. (2005). Microstructural and mechanical properties of nickel-base plasma sprayed coatings on steel and cast iron substrates. *Surface and Coatings Technology*. DOI: <https://doi.org/10.1016/j.surfcoat.2005.01.039>. [ENG]
- 8 Sytnykov, P. (2023). Structural-phase state and properties of sprayed plasma coatings based on self-fluxing alloy of NiCrBSi system with the addition of SHS composites. *Norwegian Journal of Development of the International Science*, 121, 151. DOI: <https://doi.org/10.5281/zenodo.10246>. [ENG]
- 9 Simunovic, K., Havrlisan, S., Saric, T., & Vukelic, D. (2020). Modeling and optimization in investigating thermally sprayed Ni-based self-fluxing alloy coatings: A review. *Materials (Basel)*, 13(20), 4584. DOI: <https://doi.org/10.3390/ma13204584>. [ENG]
- 10 Kazamer, N., Muntean, R., Vălean, P. C., Pascal, D. T., Mărginean, G., & Șerban, V.-A. (2021). Comparison of Ni-based self-fluxing remelted coatings for wear and corrosion applications. *Materials (Basel)*, 14(12), 3293. DOI: <https://doi.org/10.3390/ma14123293>. [ENG]
- 11 Rukhande, S. W., Rathod, W. S., & Bhosale, D. (2021). Sliding wear investigation of Ni-based coating for high-temperature application. *Tribology in Industry*, 43(2), 247–258. DOI: <https://doi.org/10.24874/ti.945.08.20.11>. [ENG]
- 12 Orlov, P. V., Korotaev, D. N., Grinberg, P. B., Fedosov, V. V. (2018) Methodical complex for investigation dependencies between microstructure and macro properties of materials and operating characteristics of products. [metodicheskiy-kompleks-dlya-issledovaniya-vzaimosvyazi-mikrostruktury-i-makrosoystv-materialov-s-ekspluatatsionnymi-harakteristikami-izdeliy.pdf](https://doi.org/10.24874/ti.945.08.20.11) [In RUS]

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ORIGINAL STUDY

INVESTIGATION OF THE EFFECT OF UNSM TREATMENT MODES ON THE STRUCTURAL AND MECHANICAL PROPERTIES OF THE SURFACE LAYER OF 45 STEEL

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Abstract. This study investigates the effect of Ultrasonic Nanocrystal Surface Modification (UNSM) treatment on the microstructure, hardness, roughness, and tribological characteristics of 45 steel. The treatment was carried out under static loads of 20, 40, and 60 N. Microstructural analysis was performed using scanning electron microscopy. Mechanical properties were evaluated by instrumental indentation and Vickers hardness methods. It was established that UNSM treatment promotes the formation of a hardened surface layer and grain refinement due to severe plastic deformation. The maximum instrumental hardness value, HIT, reached 5722.4 N/mm² at a load of 40 N. At the same time, a decrease in surface roughness and friction coefficient was observed. The obtained results demonstrate the high efficiency of UNSM treatment for improving the performance properties of 45 steel.

Keywords: Ultrasonic Nanocrystal Surface Modification (UNSM), 45 steel, surface hardening, grain refinement, microhardness, surface roughness, tribological properties, severe plastic deformation.

1. Introduction

Ensuring a high service life and reliability of machine friction units is one of the fundamental tasks of modern materials science. 45 steel, possessing an optimal combination of strength characteristics, machinability, and cost-effectiveness, is widely used in the manufacture of critical components such as shafts, axles, and gears [1,2]. However, under conditions of intensive cyclic loading and abrasive wear, conventional bulk heat treatment methods often fail to provide the required surface durability, leading to premature failure of the components [3].

One of the most promising methods for the targeted modification of surface layer properties is the technology of Ultrasonic Nanocrystal Surface Modification (UNSM). This method belongs to the processes of severe plastic deformation (SPD) and is based on the high-frequency impact action of a hard-alloy indenter on the material surface [4,5]. As a result of ultrasonic treatment, refinement of the initial ferrite-pearlite structure to a nanocrystalline state occurs in the surface layer, leading to a significant increase in microhardness [6,7]. Simultaneously, deep compressive residual stresses are formed, which inhibit the propagation of fatigue microcracks [8,9].

According to literature data, ultrasonic nanocrystal surface modification of medium-carbon steels is characterized by high efficiency in improving fatigue strength and microhardness. In particular, study [10] demonstrated that the application of UNSM treatment to S45C-type steel leads to significant grain refinement and an increase in fatigue life due to the formation of compressive residual stresses. It has been established that one of the determining factors affecting the efficiency of UNSM treatment is the magnitude of the static load. When varying the load within the range of 20–60 N, significant changes in the microstructure and mechanical characteristics of the surface layer are observed. The maximum strengthening effect is generally achieved at intermediate load values, whereas further load increase may result in defect accumulation and partial structural degradation [11].

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The efficiency of surface modification of 45 steel is determined by a combination of technological parameters, including static load, oscillation amplitude, and scanning step [12]. There exists an important scientific problem associated with identifying the optimal energy balance: insufficient treatment does not ensure the formation of a stable nanostructured layer, whereas excessive treatment leads to the “over-peening” effect-degradation of the microstructure and formation of microdefects that reduce the adhesion strength of the hardened layer [13].

In the present study, the static load in the range of 20 N, 40 N, and 60 N was selected as the variable parameter. The choice of these values was determined by the necessity to establish the transition threshold from moderate deformation impact to critical deformation conditions. A load of 20 N was considered as a regime of initial strengthening while maintaining high surface quality. Based on the analysis of literature data for medium-carbon steels [14,15], the value of 40 N appears to be the optimal regime for achieving maximum hardness. A load of 60 N was selected to investigate the risks of surface layer degradation and to determine the upper applicability limit of the method for 45 steel [16-19].

Despite the considerable number of studies devoted to UNSM treatment, the effect of static load on the complex interrelated characteristics of the surface layer of medium-carbon steels, including microstructure, microhardness, and surface roughness, remains insufficiently investigated. In particular, there is still no unambiguous understanding of the optimal load range that provides maximum strengthening without degradation of the surface layer.

In this regard, the aim of the present work is to investigate the effect of static load during UNSM treatment on the structural state and performance characteristics of the surface layer of 45 steel.

2. Materials and methods

To investigate the effect of UNSM treatment modes on the properties of 45 steel, an experimental methodology was implemented, including specimen preparation, treatment processing, and subsequent analysis of the structural and mechanical characteristics of the surface layer. 45 steel was used as the initial material. The chemical composition was determined by X-ray fluorescence analysis using a portable X-MET 8000 SMART analyzer (China). The results are presented in Table 1.

Table 1. Chemical Composition of 45 Steel.

Elements	Fe	Cr	Ni	Ti	Cu	Mn	C	W	Si
%	71.10	17.17	9.56	0.62	0.27	0.24	0.11	0.07	0.05

Surface treatment was carried out using the Ultrasonic Nanocrystal Surface Modification (UNSM) method [20] under static loads of 20 N, 40 N, and 60 N. The frequency of ultrasonic vibrations was 20 kHz, while the amplitude was 20 μm . The tool feed rate was 2000 mm/min, and the step between adjacent passes was 70 μm .

The microstructure of the surface and cross-section was investigated by scanning electron microscopy using a TESCAN VEGA 4 microscope (TESCAN, Brno, Czech Republic).

Microhardness was measured by the instrumental indentation method using a FISCHERSCOPE HM2000 hardness tester (Helmut Fischer GmbH, Sindelfingen, Germany) at a maximum load of 50 mN, loading time of 15 s, and holding time at maximum load of 5 s. In addition, measurements were carried out using the Vickers method with a semi-automatic HVS-1MDT-AXY microhardness tester (China) under a load of 0.01 kgf and an indentation time of 10 s.

The surface roughness parameters were determined using an SSR-300 profilometer (Shenzhen, Guangdong, China), and the values of R_a and R_z were calculated.

Tribological tests were carried out according to the ball-on-disk scheme using a TRB³ tribometer (Anton Paar, Austria) with a ball of 6 mm radius. The tests were performed under a load of 6 N, a sliding speed of 3 cm/s, a wear track radius of 3 mm, and a total friction path length of 60 m.

3. Results and discussion

As a result of the conducted experimental investigations, data characterizing the effect of UNSM treatment modes on the structural state and performance properties of the surface layer of 45 steel were

obtained. The evaluation included analysis of the microstructure, microhardness, surface roughness parameters, and tribological characteristics.

The microstructure of the surface and cross-sections of the samples was investigated by scanning electron microscopy. For this purpose, cross-sectional specimens were prepared, and their surfaces were subjected to electrolytic etching in a solution containing 10 g of oxalic acid and 90 g of distilled water in accordance with the procedures described in the literature [21].

Imaging was performed at an accelerating voltage of 20 kV in the secondary electron (SE) mode, which made it possible to analyze in detail the surface morphology, relief, and structural features of the material.

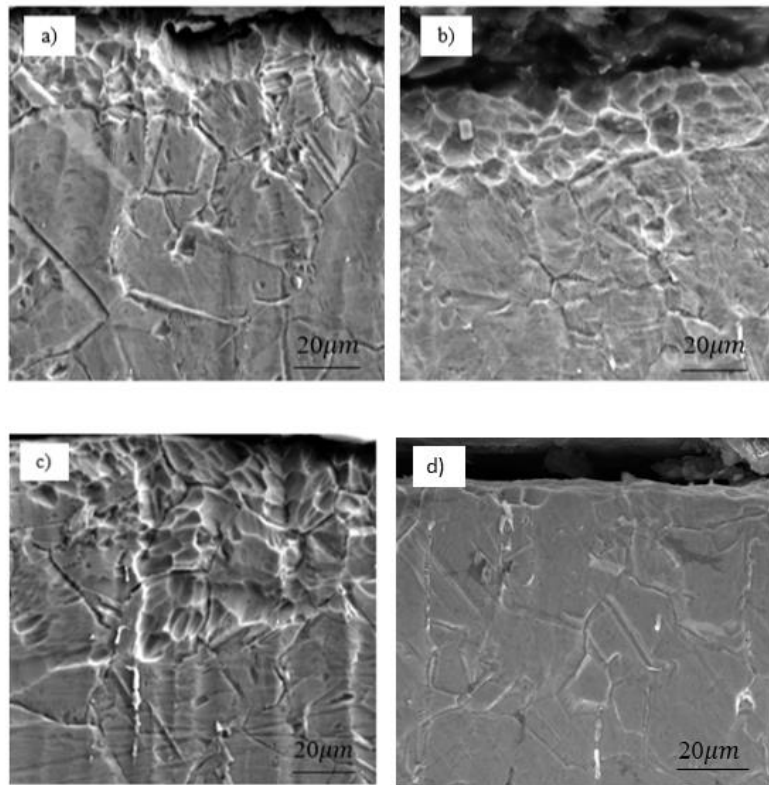


Fig. 1. Results of SEM analysis: (a) U 20N, (b) U 40N, (c) U 60N, (d) initial state.

Based on the SEM analysis results (Fig.1), a comparative investigation of the surface microstructure of the samples in the initial state (d) and after UNSM treatment under loads of 20 N, 40 N, and 60 N (a–c) was carried out.

The initial sample is characterized by a relatively homogeneous surface without pronounced signs of plastic deformation. After treatment under a load of 20 N (a), changes in the surface layer are observed: the grain boundaries become more pronounced, indicating the initial stage of severe plastic deformation. With an increase in load to 40 N (b), the degree of deformation impact increases. A more homogeneous fine-dispersed structure with a higher density of grain boundaries is formed, which indicates active refinement of the surface layer. The most pronounced changes are observed under a load of 60 N (c). The increase in static load during UNSM treatment leads to intensification of plastic deformation and structural refinement, which is the determining factor in improving the microhardness and wear resistance of the material.

To quantitatively evaluate the strengthening of the surface layer after UNSM treatment, microhardness measurements of the investigated samples were carried out using the instrumental indentation method. The values of the mechanical characteristics in the surface zone were determined. The microhardness measurement results are presented in Table 2.

Table 2. Surface Microhardness after UNSM Treatment.

Loads	HM [N/mm ²]	Elastic Modulus, GPa	HV	Hit [N/mm ²]
initial	3196.25	208.76	389.29	4120.67
U 20 N	4246.91	252.77	524.48	5552.55
U 40 N	4361.7	253.9	540.8	5722.4
U 60 N	4357.7	249.4	540.2	5716.1

Analysis of the data presented in [Table 2](#) shows that UNSM treatment leads to a significant increase in surface microhardness compared with the initial state. The obtained results are consistent with the microstructural analysis data, indicating grain refinement and the formation of a hardened surface layer. For the initial state, the hardness value was $hit=4120.67$ N/mm². After UNSM treatment, an increase in all investigated characteristics was observed. Under a load of 20 N, the hardness increased to 5552.55 N/mm², which is approximately 34.7% higher than the initial value. This indicates an increase in resistance to plastic deformation and contact strength of the surface.

The maximum values were achieved at a load of 40 N: 5722.4 N/mm², indicating the most effective surface strengthening under this treatment condition. With a further increase in load to 60 N, the values remained high at 5716.1 N/mm²; however, they were slightly lower than those obtained at 40 N. This indicates the effect of deformation hardening saturation, in which a further increase in load does not lead to a significant improvement in mechanical properties.

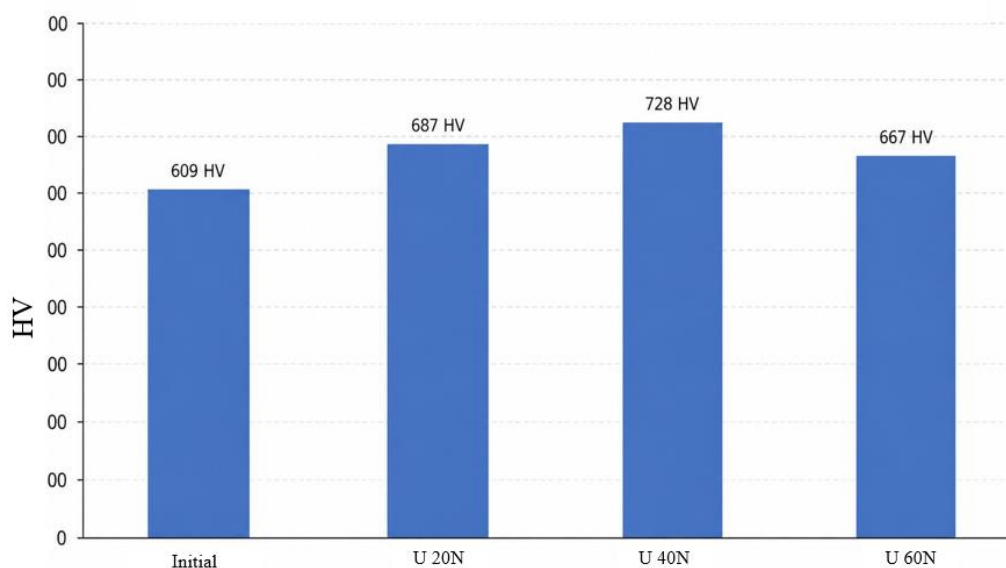


Fig. 2. Dependence of the Microhardness of 45 Steel on Static Load during UNSM Treatment (Vickers Method).

To additionally evaluate the mechanical properties of the surface layer after UNSM treatment, hardness measurements were carried out using the Vickers method with a semi-automatic microhardness tester. The obtained values were as follows: 687 HV at a load of 20 N, 728 HV at 40 N, and 667 HV at 60 N.

[Fig. 2](#) shows the dependence of microhardness on the magnitude of the static load. As can be seen, the hardness increases with increasing load up to 40 N, reaching its maximum value, after which a decrease in hardness is observed with a further increase in load to 60 N.

The obtained dependence indicates the presence of an optimal treatment regime corresponding to a load of 40 N. The decrease in hardness at a load of 60 N is associated with the effect of deformation hardening saturation and the possible development of local defects in the surface layer. Despite the difference in the absolute values obtained by the Vickers method and the instrumental indentation method, the trend in hardness

variation depending on the load remains the same. This confirms the reliability of the obtained results and indicates the formation of a hardened layer after UNSM treatment.

To evaluate the effect of UNSM treatment on the surface condition of the investigated samples, surface roughness parameters were determined.

The results of the surface roughness measurements are presented in [Table 3](#).

Table 3. Results of Surface Roughness Measurements of 45 Steel in the Initial State and after UNSM Treatment.

Loads	Ra (μm)	Rz (μm)
initial	0.031	0.538
U 20 N	0.022	0.364
U 40 N	0.027	0.418
U 60 N	0.024	0.343

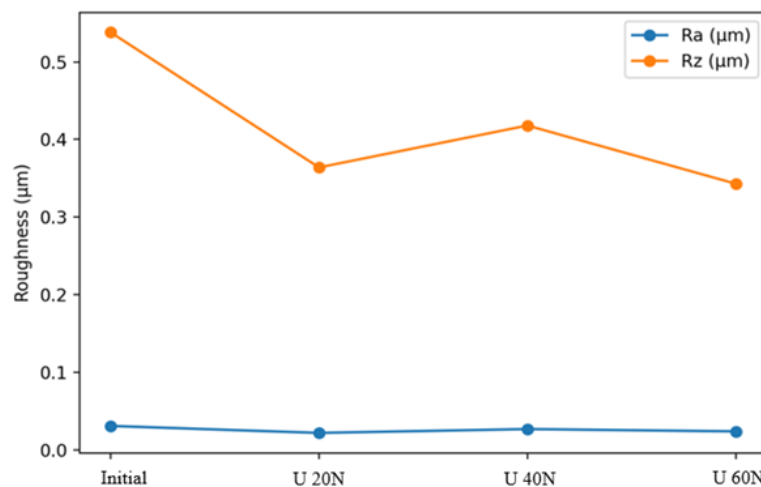


Fig. 3. Variation of Surface Roughness Parameters (Ra and Rz) of 45 Steel in the Initial State and after UNSM Treatment under Different Loads.

[Fig. 3](#) presents the results of surface roughness measurements for the initial sample and the samples after UNSM treatment under loads of 20 N, 40 N, and 60 N. The evaluation was carried out using the parameters of average arithmetic roughness Ra and profile height Rz. For the initial state, the roughness values were $Ra=0.031 \mu\text{m}$ and $Rz=0.538 \mu\text{m}$, corresponding to the most pronounced surface microrelief. Although UNSM treatment can lead to an increase in roughness due to the impact action of the indenter, in the present case a decrease in roughness parameters was observed. This is associated with the preliminary grinding and polishing of the surface, as well as with the effect of plastic smoothing of micro-irregularities during ultrasonic treatment.

After UNSM treatment under a load of 20 N, a noticeable decrease in roughness was observed: Ra decreased to $0.022 \mu\text{m}$ and Rz to $0.364 \mu\text{m}$, indicating smoothing of surface micro-irregularities due to plastic deformation of the surface layer. With an increase in load to 40 N, the value of Ra increased to $0.027 \mu\text{m}$ and Rz to $0.418 \mu\text{m}$ compared with the 20 N regime. This change may be associated with local material redistribution and the formation of additional micro-irregularities under higher impact intensity.

The lowest roughness values were obtained at a load of 60 N: $Ra=0.024 \mu\text{m}$ and $Rz=0.343 \mu\text{m}$, indicating the most effective surface leveling under this treatment condition. The decrease in roughness after UNSM treatment is confirmed by literature data, according to which severe plastic deformation of the surface layer leads to smoothing of micro-irregularities and reduction of the Ra and Rz parameters [22–24]. The reduction in roughness is caused by plastic flow of the material in the surface layer, resulting in smoothing of micro-irregularities and the formation of a more homogeneous surface relief.

To evaluate the performance characteristics of the surface layer after UNSM treatment, tribological tests were carried out using the ball-on-disk scheme.

The results of the tribological tests are presented in [Fig. 4](#).

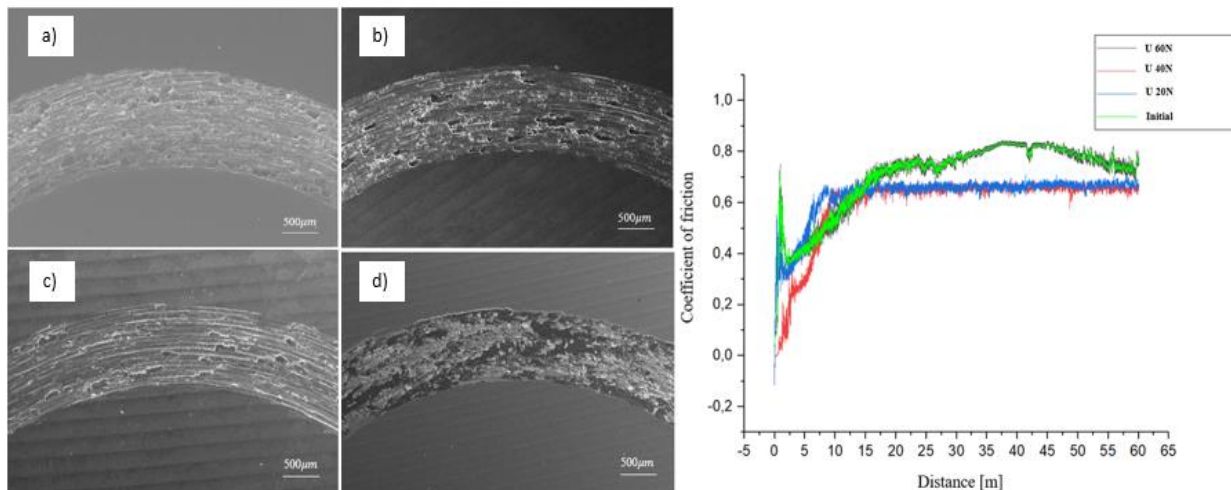


Fig. 4. Results of Tribological Tests of 45 Steel: (a) Initial State; (b) U 20 N; (c) U 40 N; (d) U 60 N.

Analysis of the tribological test results showed that UNSM treatment significantly affects the friction behavior of 45 steel. The initial state is characterized by a high coefficient of friction ($\mu \approx 0.697$) and pronounced surface wear. After treatment under a load of 20 N, a decrease in the coefficient of friction to $\mu \approx 0.630$ was observed, which is associated with partial surface leveling and strengthening of the surface layer. The minimum coefficient of friction ($\mu \approx 0.597$) was achieved at a load of 40 N, indicating the formation of an optimal hardened layer with high wear resistance. With an increase in load to 60 N, the coefficient of friction increased to $\mu \approx 0.703$, which may be associated with the development of microdefects. The tribological test results confirm that the optimal UNSM treatment regime corresponds to a load of 40 N.

4. Discussion

The obtained results demonstrate the complex nature of the effect of UNSM treatment on the properties of 45 steel. It was established that the increase in microhardness is accompanied by changes in the microstructure and a decrease in surface roughness, indicating the interrelation of the strengthening processes.

The increase in hardness is caused by plastic deformation of the surface layer under the action of repeated impact impulses, leading to grain refinement and the formation of a hardened layer. Simultaneously, smoothing of micro-irregularities occurs, resulting in a decrease in surface roughness.

After UNSM treatment, a decrease in the coefficient of friction and a reduction in surface wear are observed compared with the initial state. The most pronounced effect is achieved at a load of 40 N, at which the minimum coefficient of friction is recorded, corresponding to maximum strengthening and the formation of a homogeneous fine-dispersed structure. With an increase in load to 60 N, the tribological characteristics deteriorate, which is manifested by an increase in the coefficient of friction and greater wear.

The optimal treatment regime (40 N) provides a balance between the intensity of plastic deformation and preservation of the structural integrity of the surface, resulting in maximum improvement of the mechanical and tribological properties. Overall, UNSM treatment is an effective method for surface modification of 45 steel and can be recommended for improving the wear resistance and service life of engineering components.

5. Conclusion

In this work, a comprehensive investigation of the effect of Ultrasonic Nanocrystal Surface Modification (UNSM) treatment modes on the structural state and performance characteristics of the surface layer of 45 steel was carried out. The obtained results confirm the high efficiency of this surface modification method.

According to scanning electron microscopy data, it was established that UNSM treatment leads to intensive structural refinement and the formation of a hardened surface layer. The microstructural changes are caused by the development of severe plastic deformation under repeated impact loading.

The results of microhardness measurements by the instrumental indentation method showed that,

compared with the initial state (4120.67), a significant increase in hardness was observed after treatment: up to 5552.55 N/mm² at a load of 20 N, up to 5722.4 N/mm² at 40 N, and up to 5716.1 N/mm² at 60 N. According to the Vickers method, the values were 609 HV (initial state), 687 HV (20 N), 728 HV (40 N), and 667 HV (60 N). The maximum strengthening effect was achieved at a load of 40 N.

Surface roughness analysis showed a decrease in the surface parameters from Ra=0.031 µm and Rz=0.538 µm in the initial state to minimum values of Ra=0.022 µm and Rz=0.364 µm after treatment at 20 N, as well as Ra=0.024 µm and Rz=0.343 µm at 60 N, indicating smoothing of surface micro-irregularities.

The results of tribological tests showed that the coefficient of friction decreased from $\mu \approx 0.697$ for the initial state to $\mu \approx 0.630$ at a load of 20 N and reached a minimum value of $\mu \approx 0.597$ at a load of 40 N. With an increase in load to 60 N, the coefficient of friction increased to $\mu \approx 0.703$, which is associated with the possible formation of defects.

Thus, UNSM treatment provides a comprehensive improvement in the properties of 45 steel, including increased microhardness, reduced surface roughness, and improved tribological characteristics. The optimal treatment regime corresponds to a load of 40 N, at which the best combination of structural, mechanical, and performance properties is achieved.

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Conflict of interest

The authors declare that they have no competing interests.

References

1. Suresh, S. *Fatigue of Materials*; 2. ed., repr. (with corr.), transferred to digital print.; Cambridge Univ. Pr: Cambridge, **2006**; ISBN 978-0-521-57847-9.
2. Bhushan, B. *Modern Tribology Handbook, Two Volume Set*; 0 ed.; CRC Press, 2000; ISBN 978-0-429-12672-7.
3. Cyganek, Z.; Rodak, K.; Grosman, F. Influence of Rolling Process with Induced Strain Path on Aluminum Structure and Mechanical Properties. *Arch. Civ. Mech. Eng.* 2013, 13, 7–13, [doi:10.1016/j.acme.2012.10.008](https://doi.org/10.1016/j.acme.2012.10.008).
4. Li, J.L.; Jia, B.; Wang, Z.Q.; Pang, B.J. Asymptotic Field near the Tip of a Mode I Quasi-Statically Propagating Crack in Rate-Sensitive Materials. *Key Eng. Mater.* 2010, 452–453, 141–144, [doi:10.4028/www.scientific.net/KEM.452-453.141](https://doi.org/10.4028/www.scientific.net/KEM.452-453.141).
5. Kishore, A.; John, M.; Ralls, A.M.; Jose, S.A.; Kuruveri, U.B.; Menezes, P.L. Ultrasonic Nanocrystal Surface Modification: Processes, Characterization, Properties, and Applications. *Nanomaterials* 2022, 12, 1415, [doi:10.3390/nano12091415](https://doi.org/10.3390/nano12091415).
6. Wu, K.; Park, H.-S.; Willert-Porada, M. Pyrolysis of Polyurethane by Microwave Hybrid Heating for the Processing of NiCr Foams. *J. Mater. Process. Technol.* 2012, 212, 1481–1487, [doi:10.1016/j.jmatprotec.2012.02.010](https://doi.org/10.1016/j.jmatprotec.2012.02.010).
7. Rahimi, F.; Eivani, A.R. A New Severe Plastic Deformation Technique Based on Pure Shear. *Mater. Sci. Eng. A* 2015, 626, 423–431, [doi:10.1016/j.msea.2014.12.024](https://doi.org/10.1016/j.msea.2014.12.024).
8. Holtzer, N.; Antonin, O.; Minea, T.; Marnieros, S.; Bouchier, D. Improving HiPIMS Deposition Rates by Hybrid RF/HiPIMS Co-Sputtering, and Its Relevance for NbSi Films. *Surf. Coat. Technol.* 2014, 250, 32–36, [doi:10.1016/j.surfcoat.2014.02.007](https://doi.org/10.1016/j.surfcoat.2014.02.007).
9. Lu, J.; Lu, K. Surface Nanocrystallization (SNC) of Materials and Its Effect on Mechanical Behavior. In *Comprehensive Structural Integrity*; Elsevier, **2003**; pp. 495–528 ISBN 978-0-08-043749-1.
10. Cao, X.J.; Pyoun, Y.S.; Murakami, R. Fatigue Properties of a S45C Steel Subjected to Ultrasonic Nanocrystal Surface Modification. *Appl. Surf. Sci.* 2010, 256, 6297–6303, [doi:10.1016/j.apsusc.2010.04.007](https://doi.org/10.1016/j.apsusc.2010.04.007).

11. Aringozhina, Z.; Magazov, N.; Rakhadilov, B.; Uazyrkhanova, G.; Amanov, A. Effect of Ultrasonic Nanocrystalline Surface Modification on Hardness and Elastic Modulus of Ti-6Al-4V Alloy. *AIMS Mater. Sci.* 2025, 12, 101–117, [doi:10.3934/matersci.2025008](https://doi.org/10.3934/matersci.2025008).
12. Morozov, Iu.G.; Belousova, O.V.; Belyakov, O.A.; Parkin, I.P.; Sathasivam, S.; Kuznetsov, M.V. Titanium Nitride Room-Temperature Ferromagnetic Nanoparticles. *J. Alloys Compd.* 2016, 675, 266–276, [doi:10.1016/j.jallcom.2016.03.111](https://doi.org/10.1016/j.jallcom.2016.03.111).
13. Chang, J.; Peng, X.; Li, J.; Ellis, T. Design and Fabrication of Ni/ZrO₂ Metal-Ceramic Functionally Graded Materials by a Moving-Magnetic-Field-Driving Method. *J. Mater. Res. Technol.* 2021, 13, 1000–1011, [doi:10.1016/j.jmrt.2021.05.044](https://doi.org/10.1016/j.jmrt.2021.05.044).
14. Hong, F.T.; Schneider, A.; Sarathy, S.M. Enhanced Lubrication by Core-Shell TiO₂ Nanoparticles Modified with Gallic Acid Ester. *Tribol. Int.* 2020, 146, 106263, [doi:10.1016/j.triboint.2020.106263](https://doi.org/10.1016/j.triboint.2020.106263).
15. Shankar, V.; Mariappan, K.; Sandhya, R.; Laha, K.; Bhaduri, A.K. Long Term Creep-Fatigue Interaction Studies on India-Specific Reduced Activation Ferritic-Martensitic (IN-RAFM) Steel. *Int. J. Fatigue* 2017, 98, 259–268, [doi:10.1016/j.ijfatigue.2017.01.033](https://doi.org/10.1016/j.ijfatigue.2017.01.033).
16. Maleki, E.; Unal, O.; Guagliano, M.; Bagherifard, S. The Effects of Shot Peening, Laser Shock Peening and Ultrasonic Nanocrystal Surface Modification on the Fatigue Strength of Inconel 718. *Mater. Sci. Eng. A* 2021, 810, 141029, [doi:10.1016/j.msea.2021.141029](https://doi.org/10.1016/j.msea.2021.141029).
17. Sicupira, F.L.; Sandim, M.J.R.; Sandim, H.R.Z.; Santos, D.B.; Renzetti, R.A. Quantification of Retained Austenite by X-Ray Diffraction and Saturation Magnetization in a Supermartensitic Stainless Steel. *Mater. Charact.* 2016, 115, 90–96, [doi:10.1016/j.matchar.2016.03.023](https://doi.org/10.1016/j.matchar.2016.03.023).
18. Lu, K. Making Strong Nanomaterials Ductile with Gradients. *Science* 2014, 345, 1455–1456, [doi:10.1126/science.1255940](https://doi.org/10.1126/science.1255940).
19. Sui, M.; Zhang, Q.; Kunwar, S.; Pandey, P.; Li, M.-Y.; Lee, J. Study on the Dimensional, Configurational and Optical Evolution of Palladium Nanostructures on c -Plane Sapphire by the Control of Annealing Temperature and Duration. *Appl. Surf. Sci.* 2017, 416, 1–13, [doi:10.1016/j.apsusc.2017.04.144](https://doi.org/10.1016/j.apsusc.2017.04.144).
20. Kishore, A.; John, M.; Ralls, A.M.; Jose, S.A.; Kuruveri, U.B.; Menezes, P.L. Ultrasonic Nanocrystal Surface Modification: Processes, Characterization, Properties, and Applications. *Nanomaterials* 2022, 12, 1415, [doi:10.3390/nano12091415](https://doi.org/10.3390/nano12091415).
21. Ligabo, I.A.; Braga, V.; Ferreira, C.C.A.; Siqueira, R.H.M.; Lourenço, J.C.; Abdalla, A.J.; Lima, M.S.F. Microstructure and Corrosion Behavior of AISI 316 Steel Layers Deposited on AISI 347 Steel Substrate by Laser Metal Deposition. *Metals* 2022, 12, 2161, [doi:10.3390/met12122161](https://doi.org/10.3390/met12122161).
22. Baek, S.-H.; He, S.; Jang, M.-S.; Back, D.-H.; Jeong, D.-W.; Park, S.-H. Ultrasonic Nanocrystal Surface Modification Effect on Reduction of Hydrogen Embrittlement in Inconel-625 Parts Fabricated via Additive Manufacturing Process. *J. Manuf. Process.* 2023, 108, 685–695, [doi:10.1016/j.jmapro.2023.11.024](https://doi.org/10.1016/j.jmapro.2023.11.024).
23. Kim, M.S.; Park, S.H.; Pyun, Y.S.; Shim, D.S. Optimization of Ultrasonic Nanocrystal Surface Modification for Surface Quality Improvement of Directed Energy Deposited Stainless Steel 316L. *J. Mater. Res. Technol.* 2020, 9, 15102–15122, [doi:10.1016/j.jmrt.2020.10.092](https://doi.org/10.1016/j.jmrt.2020.10.092).
24. Karademir, I.; Celik, M.B.; Husem, F.; Maleki, E.; Amanov, A.; Unal, O. Effects of Constrained Groove Pressing, Severe Shot Peening and Ultrasonic Nanocrystal Surface Modification on Microstructure and Mechanical Behavior of S500MC High Strength Low Alloy Automotive Steel. *Appl. Surf. Sci.* 2021, 538, 147935, [doi:10.1016/j.apsusc.2020.147935](https://doi.org/10.1016/j.apsusc.2020.147935).

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ORIGINAL STUDY

DEVELOPMENT AND CHARACTERIZATION OF WEAR-RESISTANT Al_2O_3 -BASED COATINGS PRODUCED BY DETONATION SPRAYINGDaryn Baizhan ¹, Elzhan Birzhan ^{1*}, Gulim Tleubergenova ¹¹Research Center "Surface Engineering and Tribology", Sarsen Amanzholov East Kazakhstan University, Ust-Kamenogorsk, Kazakhstan*Corresponding author: ebirzan0@gmail.com

Abstract. In this study, Al_2O_3 -based oxide coatings were deposited on 10Kh18N9 austenitic stainless steel using the detonation spraying method, and their structural, mechanical, corrosion, and tribological properties were investigated. The effect of barrel filling levels (53%, 58%, and 63%) on coating formation was examined. Phase analysis revealed that the α - Al_2O_3 phase was predominant under all spraying conditions, accounting for 77–80% of the coating composition. The highest microhardness was achieved at a barrel filling level of 58%, with a microhardness of 1362.3 HV and an elastic modulus of 190.0 GPa. Corrosion tests demonstrated that the coatings reduced the corrosion current density by up to two times compared with the untreated steel substrate, while the highest corrosion resistance was observed at a filling level of 63% ($I_{\text{corr}} = 0.00725 \text{ A/cm}^2$). Tribological tests showed that the wear rate against a ZrO_2 counterbody was significantly lower than that against a 100Cr6 steel counterbody.

Keywords: detonation spraying technology, aluminum oxide coating, wear-resistant coating, microhardness, corrosion resistance.

1. Introduction

At present, the intensive wear of pump equipment components operating in aggressive environments, such as abrasive and corrosive media, remains one of the most pressing engineering challenges. Such pumps are widely used for transporting hot and cold water, wastewater, slurries, and aggressive chemical solutions [1]. During operation, the most vulnerable components – including pump impellers, covers, and volute casings—are exposed to severe mechanical and chemical воздействия, resulting in significant material degradation and wear [2]. These components are commonly manufactured from 10Kh18N9 austenitic stainless steel. Although this material exhibits excellent corrosion resistance, its resistance to abrasive wear is relatively limited [3]. As a consequence, premature failure of pump components may occur, leading to reduced equipment reliability and shortened service life [4]. To address this issue, protective coatings with enhanced wear and corrosion resistance can be applied to the surfaces of pump components. Such coatings serve to mitigate the destructive effects of aggressive reagents and abrasive particles, thereby improving the durability and operational performance of the equipment [5–6].

Protective coatings based on aluminum oxide (Al_2O_3) are among the most widely used ceramic materials in industry due to their high hardness, chemical inertness, excellent wear resistance, and high melting point [7]. Owing to their outstanding chemical stability, Al_2O_3 coatings are extensively applied in acid-resistant pump impellers, pump casings, linings of acid-transport pipelines, and valves [8]. Al_2O_3 coatings produced by thermal spraying techniques are also widely utilized in various industrial sectors, including aerospace components, construction structures, and electrical and electronic applications [9].

At present, various thermal spraying technologies are widely employed for the production of wear- and corrosion-resistant coatings, including flame spraying using powder or wire feedstock, detonation spraying (DS), high-velocity oxy-fuel spraying (HVOF), and atmospheric plasma spraying (APS) [10–11]. Among these methods, APS is the most commonly used industrial technique for depositing Al_2O_3 ceramic coatings.

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However, due to the relatively low particle velocity achieved during atmospheric plasma spraying, the resulting coatings often exhibit high porosity. In addition, the elevated temperatures required to melt the particles promote the transformation of the initial α - Al_2O_3 powder into the thermodynamically metastable γ - Al_2O_3 phase. As a result, coating porosity typically remains within the range of 5–15%, leading to a deterioration of mechanical properties [12–13]. Among the thermal spraying technologies mentioned above, detonation spraying (DS) offers several advantages compared with alternative techniques such as high-velocity oxy-fuel spraying (HVOF) [14]. In the DS process, particles are accelerated by a detonation wave and impact the substrate surface at velocities of 800–1200 m/s. Upon collision with the metallic substrate, the particles form an exceptionally strong mechanical bond. Studies have confirmed that Al_2O_3 coatings produced by detonation spraying exhibit an adhesive strength exceeding 50 MPa and a porosity level of approximately 0.8% [15]. The high pressure generated during detonation, combined with rapid solidification, results in the formation of a highly dense coating layer. Consequently, detonation spraying enables the production of homogeneous coatings with porosity as low as 0.3% and strong adhesion to the substrate material [16].

Several studies on Al_2O_3 coatings produced by detonation spraying have previously been reported. In the work of Kantay et al., Al_2O_3 coatings were deposited on 12Kh18N10T stainless steel using detonation spraying with barrel filling levels ranging from 53% to 68%, and the influence of barrel filling on the phase composition and tribological properties of the coatings was investigated [17]. The authors found that the coating produced at a 53% filling level exhibited the highest wear resistance and concluded that an increase in the α - Al_2O_3 phase fraction led to enhanced hardness and wear resistance [18]. However, the corrosion resistance and adhesive strength of the coatings were not comprehensively evaluated in that study. In another study, Rakhadiev et al. produced gradient Al_2O_3 coatings by varying the barrel filling level with explosive gases to 53%, 56%, and 63%. It was established that coatings deposited at a 56% filling level were predominantly composed of the α - Al_2O_3 phase, whereas coatings produced at a 63% filling level were dominated by the γ - Al_2O_3 phase [19]. Nevertheless, corrosion testing and electrochemical characterization were not thoroughly investigated in this work either. Korobov et al. compared coatings with predominantly α - Al_2O_3 and γ - Al_2O_3 phase compositions. The researchers examined the possibility of increasing the α -phase content by adjusting the acetylene-to-oxygen ratio in the explosive gas mixture. However, they noted the limited availability of data on detonation-sprayed coatings in which α - Al_2O_3 is retained as the dominant phase [20]. Based on the analysis of the aforementioned studies, it can be concluded that the tribological performance and electrochemical corrosion resistance of Al_2O_3 coatings produced by detonation spraying under different barrel filling conditions have not yet been comprehensively evaluated.

Accordingly, the aim of this study was to deposit Al_2O_3 coatings on the surface of 10Kh18N9 austenitic stainless steel using the detonation spraying technique and to evaluate their wear and corrosion resistance.

2. Materials and methods

Austenitic stainless steel of grade 10Kh18N9 was selected as the substrate material for coating deposition. The chemical composition of the steel (wt.%) was as follows: chromium 17–18%; nickel 8–10%; carbon not more than 0.12%; manganese up to 2.0%; silicon up to 1.0%; and iron as the balance. The specimens were prepared in the form of discs with a diameter of 22 mm and a thickness of 5 mm. Prior to coating deposition, the 10Kh18N9 steel specimens underwent preliminary mechanical surface preparation. Specifically, the surfaces were subjected to grinding and polishing treatments. Subsequently, the specimens were grit-blasted to enhance surface roughness and improve coating adhesion. Al_2O_3 coatings were deposited using a CCDS2000 detonation spraying system. A general view of the installation is presented in [Fig. 1](#).



Fig. 1. CCDS2000 computerized detonation complex: (a) detonation barrel, (b) control unit, and (c) cooling system.

The experimental work was carried out under three different regimes with barrel filling levels of explosive gases set at 53%, 58%, and 63%. For each regime, 100 detonation shots were performed. The distance between the barrel exit and the substrate surface was 250 mm. A stoichiometric oxygen/acetylene (O_2/C_2H_2) mixture with a ratio of 1.856 was used as the combustible gas mixture. [Table 1](#) presents the deposition regimes for Al_2O_3 coatings produced by the detonation spraying method. The obtained samples were designated according to the barrel filling level as $Al_2O_3_{53\%}$, $Al_2O_3_{58\%}$, and $Al_2O_3_{63\%}$.

Table 1. Detonation spraying regimes for Al_2O_3 coatings.

Samples	Barrel filling with explosive gas (%)	Number of shots	Distance, mm	O_2/C_2H_2 ratio
$Al_2O_3_{53\%}$	53	100	250	1,856
$Al_2O_3_{58\%}$	58	100	250	1,856
$Al_2O_3_{63\%}$	63	100	250	1,856

To determine the phase composition of the obtained coatings, X-ray diffraction (XRD) analysis was performed. The investigations were carried out using an X'Pert Pro diffractometer (Philips Corporation, Amsterdam, The Netherlands). Measurements were conducted using $Cu-K\alpha$ radiation ($\lambda = 1.5406 \text{ \AA}$) at an accelerating voltage of 40 kV and a tube current of 40 mA. Diffraction patterns were recorded in the angular range of $20-90^\circ$ (2θ) with a step size of 0.02° . The surface morphology and cross-sectional microstructure of the coatings were examined using a SEM3200 scanning electron microscope. To determine the elemental composition and the distribution profile of elements across the coating surface, energy-dispersive X-ray spectroscopy (EDS) analysis was performed using an XFlash Detector 730M-300 system. The hardness of the samples was measured using the FISCHERSCOPE HM2000 instrumented indentation system in accordance with the international standard DIN EN ISO 14577-1. During testing, the load was gradually increased to 2000 mN over a period of 20 s. Tribological properties of the coatings were investigated using a TRB³ tribometer (Anton Paar, Graz, Austria) in a ball-on-disk configuration. ZrO_2 and 100Cr6 steel balls with a diameter of 3.00 mm were used as counter bodies. The applied load was set to 10.00 N, and the sliding speed was 3 cm/s, with a total sliding distance of 100 m. The corrosion properties of the samples were studied using electrochemical methods with an Autolab PGSTAT204 system in an HCl solution. Corrosion parameters were determined from the obtained polarization curves using the Tafel extrapolation method.

3. Results and discussion

The X-ray diffraction (XRD) analysis results of Al_2O_3 coatings obtained by detonation spraying at barrel filling levels of 53%, 58%, and 63% are presented in [Fig. 2](#). Analysis of the diffractograms showed that all

investigated samples contain two main polymorphic modifications of aluminum oxide: α -Al₂O₃ and γ -Al₂O₃ phases. The positions of the identified diffraction peaks are consistent with the reference data from the JCPDS database. The crystalline structure of the coatings is characterized by intense reflections corresponding to the crystallographic planes of the α -Al₂O₃ phase, namely (012), (104), (110), (113), (024), (116), (018), (300), (119), and (223). In addition, the presence of the γ -Al₂O₃ phase is confirmed by diffraction peaks corresponding to the (222), (400), and (440) planes. The clear presence of these reflections indicates that the obtained coatings possess a sufficiently well-crystallized structure.

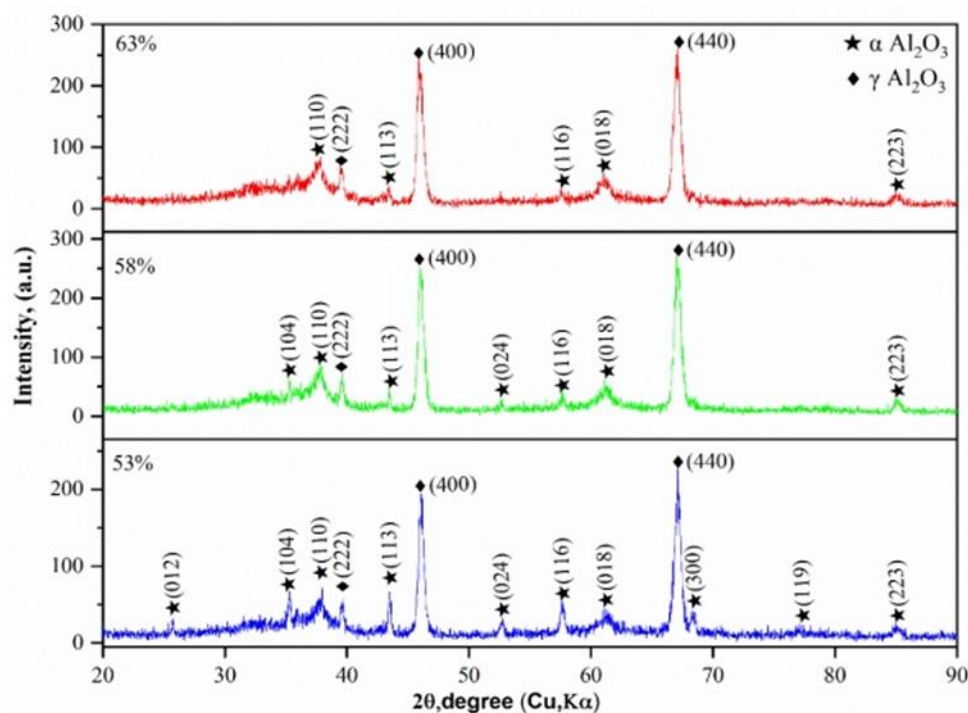


Fig. 2. X-ray diffraction patterns of Al₂O₃ coatings obtained at different detonation barrel filling levels.

According to the quantitative phase analysis results, the coatings obtained at barrel filling levels of 53%, 58%, and 63% contained 80.2%, 77.3%, and 79.1% of the α -Al₂O₃ phase, respectively. The corresponding fractions of the γ -Al₂O₃ phase were 19.8%, 22.7%, and 20.9%, respectively (Table 2). No significant differences in phase composition were observed between the investigated regimes, indicating that variations in barrel filling level do not substantially affect the phase state of aluminum oxide coatings. In all cases, α -Al₂O₃ remained the dominant phase. Such behavior can be explained by the high-temperature conditions generated during the detonation spraying process and the intensive heating of powder particles, which promote the stabilization of the α -Al₂O₃ phase.

Table 2. Phase composition of α -Al₂O₃ and γ -Al₂O₃ at different detonation barrel filling levels.

Samples	α -Al ₂ O ₃ , %	γ -Al ₂ O ₃ , %
Al ₂ O ₃ _53%	80,2	19,8
Al ₂ O ₃ _58%	77,3	22,7
Al ₂ O ₃ _63%	79,1	20,9

The analysis of the elemental line distribution of Al₂O₃ coatings (Fig. 3) revealed the formation of two distinct regions corresponding to the coating and the substrate in all investigated samples. For coatings obtained at barrel filling levels of 53%, 58%, and 63%, the concentrations of aluminum and oxygen were uniformly distributed across the coating thickness. At the coating – substrate interface, the intensities of Al and O sharply decreased, while the content of Fe, Cr, and Mn – main elements of the 10Kh18N9 steel substrate – increased significantly. The clearly defined transition zone indicates a high coating density and good

adhesion between the coating and the substrate. The elemental distribution profiles of samples produced at 58% and 63% barrel filling levels appeared more homogeneous compared with those obtained at 53%. This suggests that an increase in barrel filling level leads to a higher degree of particle melting during spraying and improves coating homogeneity. In addition, the coating thickness was found to depend on the barrel filling level: $188.1 \pm 15.1 \mu\text{m}$ for the 53% regime, $502.7 \pm 25.1 \mu\text{m}$ for the 58% regime, and $370.5 \pm 11.1 \mu\text{m}$ for the 63% regime. The maximum coating thickness was observed at 58% filling. The reduction in thickness at the 63% filling level, despite the increased powder feed, may be explained by partial particle loss due to splashing during spraying and incomplete participation in coating formation. As a result, the coating growth rate decreases, leading to a reduced final thickness.

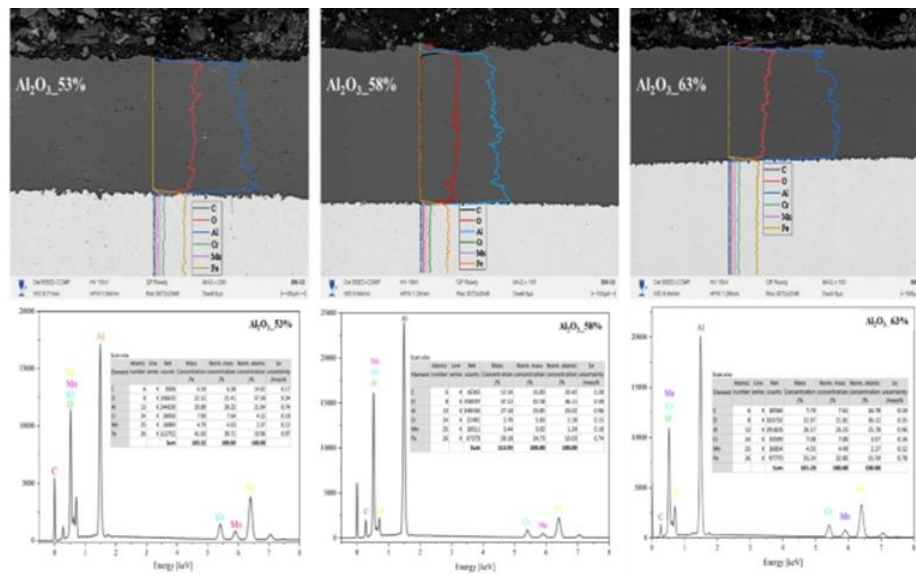


Fig. 3. Microstructure of Al_2O_3 coatings obtained at different detonation barrel filling levels.

Mechanical testing results demonstrated that the coating properties strongly depend on the spraying regime (Fig. 4). At a barrel filling level of 53%, the coatings exhibited a Young's modulus of 121.0 GPa and a hardness of 860.1 HV. The highest values were recorded for the 58% barrel filling regime, where the Young's modulus reached 190.0 GPa and the hardness increased to 1362.3 HV. This improvement is associated with the formation of a denser and more homogeneous coating structure. In contrast, at a 63% barrel filling level, both the Young's modulus and hardness decreased to 167.7 GPa and 1174.5 HV, respectively. This reduction can be explained by a decrease in coating density caused by particle splashing and partial loss of material during deposition.

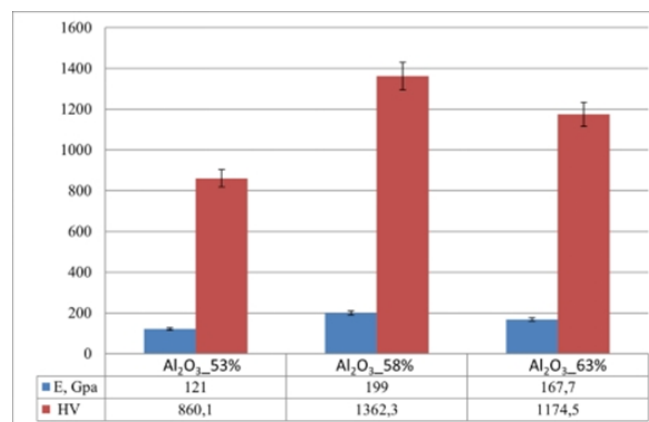


Fig. 4. Microhardness and Young's modulus of Al_2O_3 coatings obtained at different detonation barrel filling levels.

The corrosion test results (Fig. 5) demonstrated that the obtained coatings significantly improve corrosion resistance compared with the initial 10Kh18N9 stainless steel substrate. The corrosion current density (I_{corr}) for the uncoated steel was 0.01343 A/cm^2 , whereas for the coatings produced at barrel filling levels of 53%, 58%, and 63%, this value decreased to 0.01210 A/cm^2 , 0.00873 A/cm^2 , and 0.00725 A/cm^2 , respectively. The reduction in I_{corr} indicates a slowdown of the electrochemical corrosion process and an enhancement of the protective properties of the coatings. A similar trend was observed for the calculated corrosion current density values, which decreased by approximately two times when transitioning from the uncoated substrate to the coating obtained at 63% barrel filling. In addition, the corrosion rate (CR) also decreased gradually: for the uncoated steel, the value was $2.08 \times 10^{-4} \text{ mm/year}$, while for coatings produced at 53%, 58%, and 63% barrel filling levels, it decreased to $1.88 \times 10^{-4} \text{ mm/year}$, $1.35 \times 10^{-4} \text{ mm/year}$, and $1.12 \times 10^{-4} \text{ mm/year}$, respectively. These results confirm that the coatings provide effective protection against aggressive environments and significantly reduce the intensity of corrosion damage.

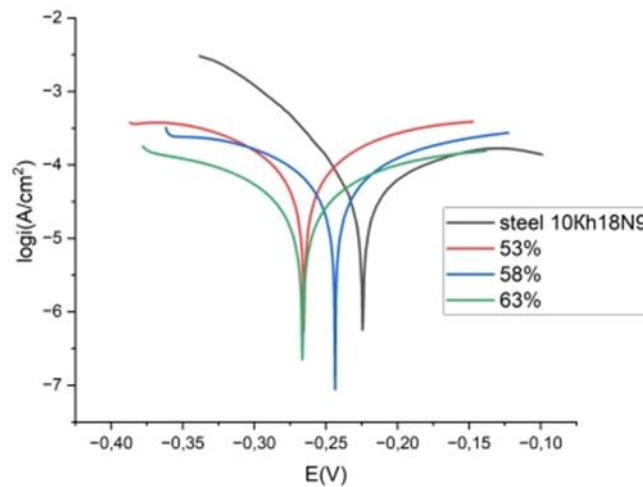


Fig. 5. Potentiodynamic polarization test results of Al_2O_3 coatings obtained at different detonation barrel filling levels.

Tribological test results demonstrated that the wear behavior of the coatings strongly depends on the counter-body material used. During interaction with the ZrO_2 counter-body, all investigated samples exhibited relatively low wear intensity (Fig. 6), characterized by small cross-sectional areas of the wear tracks. For coatings obtained at barrel filling levels of 53%, 58%, and 63%, the wear track areas were $3421.1 \mu\text{m}^2$, $4407.1 \mu\text{m}^2$, and $6377.0 \mu\text{m}^2$, respectively. The coefficient of friction remained stable within the range of 0.55–0.60, with no significant fluctuations observed during the tests. Analysis of the wear track microrelief revealed a relatively smooth profile with a limited number of abrasive grooves. This indicates a low degree of surface damage and confirms that the coatings maintain a high level of wear resistance during contact with the ZrO_2 counter-body.

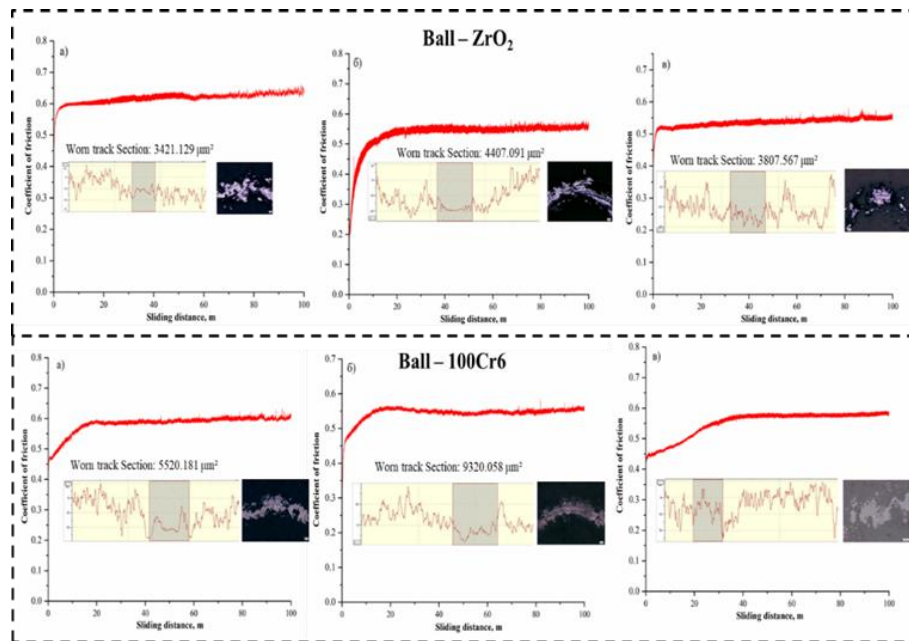


Fig. 6. Friction coefficient curves and wear track profiles of Al_2O_3 coatings tested with ZrO_2 and 100Cr6 counterbodies: (a) Al_2O_3 _53%, (b) Al_2O_3 _58%, (c) Al_2O_3 _63%.

It was found that, compared with the ZrO_2 counterbody, the use of a 100Cr6 steel counterbody leads to a significant increase in the wear intensity of the coatings and higher material loss (Fig. 7). The cross-sectional areas of the wear tracks for the samples obtained at detonation barrel filling levels of 53%, 58%, and 63% with explosive gas were $5520.2 \mu\text{m}^2$, $9320.1 \mu\text{m}^2$, and $6377.0 \mu\text{m}^2$, respectively, which are considerably higher than the values obtained when using the zirconium oxide counterbody. The coefficient of friction stabilized in the range of 0.55-0.65 and was characterized by pronounced fluctuations during the test. Analysis of the microprofiles of the wear tracks revealed features typical of microplastic deformation, abrasive microcutting, and local spalling mechanisms. These phenomena can be explained by the intensified mechanical action on the coating surface caused by the high hardness and abrasive activity of 100Cr6 steel. As a result, the use of the 100Cr6 counterbody causes an increase in friction-induced loading, intensive degradation of the coating, and a higher wear rate. The obtained results indicate that the properties of the counterbody material are among the key factors determining the tribological behavior of the coatings.

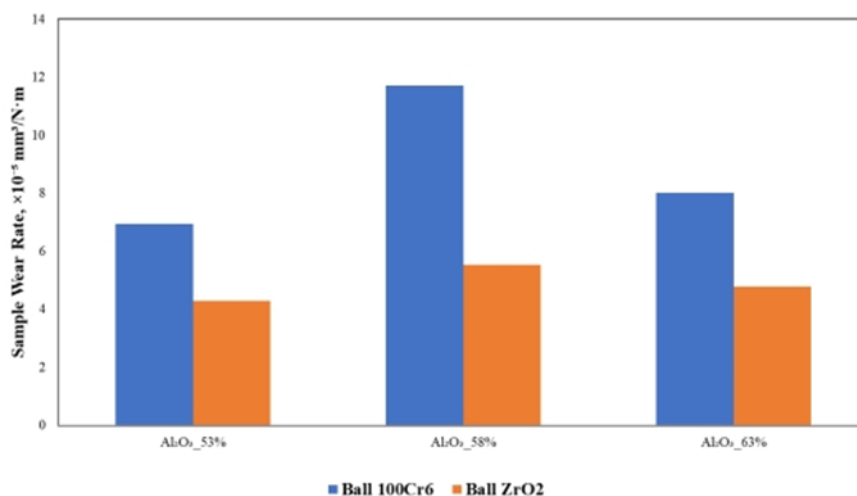


Fig. 7. Specific wear of Al_2O_3 coatings obtained at different filling volumes of the detonation barrel with explosive gas under testing conditions against 100Cr6 and ZrO_2 counterbodies.

4. Conclusion

As a result of the study, it was established that the structure, mechanical properties, and tribological characteristics of Al₂O₃ coatings obtained by detonation spraying directly depend on the filling level of the detonation barrel with explosive gas. When the filling level of the detonation barrel with explosive gas was increased to 58%, the microstructure of the coating improved significantly: the coating became denser, and the degree of particle melting increased. As a result, the microhardness (1362.3 HV) and elastic modulus (190.0 GPa) reached their maximum values, indicating an improvement in mechanical properties. In addition, under the 58% filling condition, the corrosion current density decreased significantly compared with the initial steel. Although the highest corrosion resistance ($I_{\text{corr}} = 0.00725 \text{ A/cm}^2$) was observed at the 63% filling level, the mechanical properties decreased compared with those obtained at 58%. The reduction in coating thickness can be explained by the scattering and loss of part of the powder during the spraying process. Tribological tests showed that the wear rate against the ZrO₂ counterbody was significantly lower than that against the 100Cr6 steel counterbody, confirming that the counterbody material has a direct influence on the wear behavior of the coating. Thus, a barrel filling level of 58% is the optimal regime for obtaining Al₂O₃ coatings.

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Conflict of interest

The authors declare that they have no competing interests.

References

1. Kakar A.K. et al. Abrasive, Erosive and Corrosive Wear in Slurry Pumps - A Review // International Journal of Engineering Research and Technology. - 2020. (academia.edu) https://www.academia.edu/128761333/Abrasive_Erosive_and_Corrosive_Wear_in_Slurry_Pumps_A_Review
2. Nagentrau M. et al. Experimental investigation of premature failure of cyclone feed pump impeller used in diamond mining applications // Engineering Failure Analysis. - 2026. - Art. 109414. https://www.researchgate.net/publication/310233610_Failure_Analysis_of_Slurry_Pump_Impeller_Fractured_at_Coahuasi_Mine
3. Tyshchenko O. et al. Electrochemical Behavior of Plasma-Nitrided Austenitic Stainless Steel in Chloride Solutions // Materials. - 2024. - Vol. 17. <https://doi.org/10.3390/ma17174338>
4. Li G. et al. Failure analysis of the impeller of slurry pump used in zinc hydrometallurgy process // Engineering Failure Analysis. - 2006. - Vol. 13. - P. 1101-1112. <https://doi.org/10.1016/j.engfailana.2005.07.021>
5. Meta Corrosion: In-depth Analysis, Economic Impacts and Inhibition Strategies for Enhanced Infrastructure Durability. https://www.researchgate.net/publication/380710897_Meta_Corrosion_In-depth_Analysis_Economic_Impacts_and_Inhibition_Strategies_for_Enhanced_Infrastructure_Durability
6. Szala M., Waczak M. Metallic and Ceramic Materials Integrity - Surface Engineering for Wear, Corrosion and Erosion Prevention // Materials. - 2024. - Vol. 17, No. 7. - Art. 1541. <https://doi.org/10.3390/ma17071541>
7. Suresh Babu P. et al. Thermal Spray Coatings for Erosion-Corrosion Resistant Applications // Transactions of the Indian Institute of Metals. - 2020. - Vol. 73. - P. 2141-2159. <https://doi.org/10.1007/s12666-020-02053-0>
8. Mazur M. et al. Study of microstructure and corrosion behavior of nano-Al₂O₃ coating layers on TiO₂ substrate // Scientific Reports. - 2024. - Vol. 14. - Art. 18449. <https://doi.org/10.1038/s41598-024-68566-6>
9. Bejinariu C. et al. Microstructure, Corrosion Resistance, and Tribological Properties of Al₂O₃ Coatings Prepared by Atmospheric Plasma Spraying // Materials. - 2022. - Vol. 15, No. 24. - Art. 9013. <https://doi.org/10.3390/ma15249013>
10. Durga Prasad C. et al. Thermal spray coatings on high-temperature oxidation and corrosion applications - A comprehensive review // Surface Interfaces. - 2024. <https://doi.org/10.1016/j.surfin.2024.104840>
11. Sharma A. et al. Thermal Spray High-Entropy Alloy Coatings: A Review // Journal of Thermal Spray Technology. - 2020. - Vol. 29. - P. 857-893. <https://doi.org/10.1007/s11666-020-01047-0>
12. Pawowski. The Science and Engineering of Thermal Spray Coatings. - 2nd ed. - Chichester: John Wiley & Sons, 2008. - 626 p. <https://doi.org/10.1002/9780470754085>

13. Wang Z. et al. Particle-in-flight behavior and its influence on the microstructure and mechanical properties of plasma-sprayed Al_2O_3 coatings // Journal of the European Ceramic Society. - 2008. - Vol. 28, No. 6. - P. 1143–1152. <https://doi.org/10.1016/j.jeurceramsoc.2007.09.039>
14. Raza A. et al. An Influence of Oxygen Flow Rate and Spray Distance on the Porosity of HVOF Coating and Its Effects on Corrosion // Materials. - 2022. - Vol. 15, No. 18. - Art. 6329. <https://doi.org/10.3390/ma15186329>
15. Yi Y. et al. Al_2O_3 coating prepared on stainless steel by D-gun spraying and its tribological properties in FNaK molten salt // Surface and Coatings Technology. - 2018. - Vol. 349. - P. 667–675. <https://doi.org/10.1016/j.surfcoat.2018.06.043>
16. Sundararajan G. et al. A Review on Detonation Gun Sprayed Coatings // Journal of Minerals and Materials Characterization and Engineering. - 2012. - Vol. 11. - P. 285. <http://dx.doi.org/10.4236/jmmce.2012.113022>
17. Kantay N., Rakhadilov B., Kurbanbekov S., Yeskermessov D., Yerbolatova G., Apsezhanova A. Influence of Detonation-Spraying Parameters on the Phase Composition and Tribological Properties of Al_2O_3 Coatings // Coatings. - 2021. - Vol. 11, No. 7. - Art. 793. <https://doi.org/10.3390/coatings11070793>
18. Kantay N., Rakhadilov B. et al. Influence of Detonation-Spraying Parameters on the Phase Composition and Tribological Properties of Al_2O_3 Coatings // Coatings. - 2021. - Vol. 11, No. 7. - Art. 793. <https://doi.org/10.3390/coatings11070793>
19. Rakhadilov B. et al. Tribological and Mechanical Properties of Gradient Coating on Al_2O_3 -Based Coating Produced by Detonation Spraying Methods // Advances in Tribology. - 2023. - Art. 1520135. <https://doi.org/10.1155/2023/1520135>
20. Korobov Y. et al. Comparative Study of Structures and Properties of Detonation Coatings with α - Al_2O_3 and γ - Al_2O_3 Main Phases // Coatings. - 2021. - Vol. 11, No. 12. - Art. 1566. <https://doi.org/10.3390/coatings11121566>

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ORIGINAL STUDY

**STUDY OF THE STRUCTURE AND PROPERTIES OF ALLOYS
OBTAINED BY ELECTROSLAG REMELTING OF STEEL SCRAP
ALLOYED WITH CARBIDE-FORMING ELEMENTS**Kenesbekov A¹, Rahimberdinov A², Kusainov T^{1,*} Karibayeva L¹¹ Institute of Composite Materials, Ust-Kamenogorsk 070000, Kazakhstan² Asia Quartz LLP, Ust-Kamenogorsk, Kazakhstan*Corresponding author: kusainov.temirlan00@mail.ru

Abstract. This study presents a comprehensive investigation of the effect of plastic deformation on the microstructure, mechanical, tribological, and corrosion properties of an iron-containing alloy produced by the electroslag remelting (ESR) process. The study focused on two states of the material: as-cast after ESR and deformed after subsequent rolling (ESR-R). Using X-ray fluorescence and X-ray diffraction analysis, as well as electron microscopy, the characteristics of the alloy's phase composition and microstructure were determined. It was shown that plastic deformation leads to grain refinement, an increase in defect density, and the formation of secondary phases. A significant increase in the microhardness and wear resistance of the material after rolling was observed, accompanied by a decrease in the coefficient of friction. At the same time, an increase in corrosion current density and corrosion rate was observed, despite a shift of the corrosion potential toward a more positive region. The results obtained indicate the complex nature of the effect of plastic deformation on the alloy's service properties and the need to account for the material's structural state during its application. Translated with DeepL.com (free version).

Keywords: electroslag remelting, plastic deformation, alloy microstructure, microhardness, tribological properties, corrosion resistance, iron-based alloys.

1. Introduction

One of the most productive and effective methods currently available is the electroslag remelting process, which produces high-quality metallic materials with low levels of non-metallic impurities and enhanced structural uniformity.

The essence of this process lies in remelting the consumable electrode in a bath of molten electrically conductive slag placed in a water-cooled mold, where heat is generated by Joule heating [1]. This mechanism allows for control over the melting of the electrode and the direction of ingot crystallization, which in turn enables the production of metal with a specified macro- and microstructure [2].

ESHP offers a number of advantages, the main one being the presence of a slag bath, which acts as a filtering and refining medium. The metal passes drop by drop through a layer of molten slag, where non-metallic inclusions are removed, alloying elements are redistributed, and the content of harmful impurities is reduced [2, 3]. This process helps to increase the purity of the metal and improve its performance properties [4]. The production of a high-quality ingot essentially depends on factors such as heating rate, the technological parameters of the electric arc melting process, and the thermal conditions during solidification [3, 5]. Deviations from optimal operating conditions can lead to the formation of defects, such as liquidation inhomogeneities and gas contamination, which negatively affect the material's properties [5].

The ESR method has become widely used in the production of structural, tool, stainless, and heat-resistant steels, as well as iron- and nickel-based alloys, for which high structural uniformity and minimal defect content are critical [4, 6]. Historically, the technology was developed in the mid-20th century and was further advanced

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thanks to the work of the scientific school associated with E.O. Paton and B.E. Paton, which facilitated its industrial implementation [7,8].

Despite the high quality of the metal after electroslog remelting, the as-cast state of the alloy is characterized by a dendritic structure and microsegregation of alloying elements [3,9]. The presence of such structural features can have a significant impact on the mechanical, tribological, and corrosion properties of the material, especially when operated in aggressive environments containing chloride ions [10].

The corrosion resistance of metallic materials is a critical factor determining their durability and reliability. Even minor structural inhomogeneities can initiate the localization of electrochemical processes, leading to the development of pitting and crevice corrosion [10,11]. Consequently, there is a need for additional processing of the material to improve its microstructure.

One effective method for modifying the structure after ESR is plastic deformation, specifically rolling. As a result of the deformation process, grain refinement, phase redistribution, a reduction in chemical heterogeneity, and an increase in the density of crystal lattice defects occur [9,12]. These changes lead to an increase in the material's hardness and strength properties, and also affect its tribological behavior [12].

At the same time, an increase in dislocation density and residual stresses can have an ambiguous effect on corrosion properties. On the one hand, improved structural homogeneity contributes to the formation of a more stable passive layer; on the other hand, increased defect density can accelerate electrochemical metal dissolution processes [11,13].

Thus, the effect of plastic deformation on the performance properties of alloys produced by the electroslog remelting process is complex in nature and requires detailed investigation. Establishing the relationship between structural changes and alterations in mechanical, tribological, and corrosion characteristics is a pressing scientific challenge.

The aim of this work is to investigate the effect of plastic deformation on the structure, mechanical, tribological, and corrosion properties of an iron-containing alloy produced by electroslog remelting, as well as to establish the patterns of change in its characteristics during the transition from the as-cast state to the deformed state.

2. Materials and Methods

2.1 Materials and sample preparation

The investigated material was a Fe-based alloy produced by electroslog remelting (ESR). Two structural conditions of the same alloy were studied:

1. the alloy after electroslog remelting in the as-cast condition (denoted as ESR);
2. the same alloy after ESR followed by plastic deformation by rolling (denoted as ESR-R).

The alloy contained Fe as the main component (>90 wt.%) with alloying additions including Cr, Ni, Ti, and Mo. These alloying elements were selected due to their potential influence on mechanical, tribological, and corrosion properties.

The rolled samples were obtained by plastic deformation of the ESR alloy under controlled rolling conditions. Prior to characterization, all specimens were mechanically ground and polished according to standard metallographic preparation procedures.

2.2 Chemical composition analysis

The chemical composition of the investigated alloy was determined by X-ray fluorescence (XRF) analysis using a portable Hitachi analyzer operating in PQA mode. The measurement time was 10 s for each acquisition. Measurements were performed at two different locations on the sample surface, and the obtained values were averaged. The elemental concentrations were expressed in weight percent (wt.%).

2.3 Phase composition analysis

Phase and structural characterization of the alloy was carried out by X-ray diffraction (XRD) using an X'Pert PRO diffractometer (PANalytical, Netherlands) with Cu K α radiation.

The diffraction measurements were performed under the following conditions:

- tube voltage: 40 kV;
- tube current: 30 mA;
- scanning step: 0.02°;
- counting time: 1 s per step;
- scanning range: $2\theta = 20\text{--}90^\circ$.

The obtained diffraction patterns were analyzed using HighScore software combined with the PDF-4 crystallographic database.

2.4 Microstructural characterization

The microstructure of the investigated alloys was studied by scanning electron microscopy (SEM) in both secondary electron (SE) and backscattered electron (BSE) imaging modes.

Elemental distribution and local chemical composition were analyzed using energy-dispersive X-ray spectroscopy (EDS) combined with elemental mapping. The analysis was performed for both ESR and ESR-R conditions in order to evaluate the influence of rolling deformation on structural homogeneity and elemental redistribution.

2.5 Mechanical characterization

2.5.1 Vickers microhardness

Vickers microhardness measurements were performed using a load corresponding to the HV0.1 scale. The indentation imprints were analyzed and averaged values were calculated for each structural condition.

2.5.2 Instrumented indentation

Instrumented indentation tests were carried out using a FISCHERSCOPE HM2000 system based on the dynamic indentation method according to DIN EN ISO 14577-1.

The tests were performed using a Vickers diamond indenter with an angle of 136° between opposite faces. The loading range was 0.1–2000 mN, while the indenter approach speed was 2 $\mu\text{m/s}$. The displacement resolution of the instrument was 0.1 nm.

The following parameters were determined:

- Martens hardness (HM);
- nanohardness;
- elastic modulus.

Experimental data processing was performed using WIN-HCU software.

2.5.3 Rockwell hardness

Additional hardness measurements were carried out using the Rockwell B scale (HRB) to evaluate the macroscopic hardness behavior of the investigated alloy states.

2.6 Tribological testing

Tribological behavior was evaluated using a TRB3 tribometer (Anton Paar Srl, Switzerland) under dry sliding conditions according to the ball-on-disk method described in ASTM G99.

A 3 mm diameter 100Cr6 steel ball was used as the counterbody. The testing conditions were as follows:

- normal load: 15 N;
- sliding speed: 3 cm/s;
- wear track radius: 4 mm;
- sliding distance: 200 m.

The tribological performance was evaluated based on the friction coefficient, wear track morphology, wear track cross-sectional area, and wear rate.

2.7 Scratch testing

Scratch tests were performed to evaluate the deformation and damage behavior of the investigated alloy under progressive loading conditions. During the tests, the friction force, penetration depth, residual depth, and acoustic emission signals were continuously recorded.

The obtained data were used to analyze the resistance of the material against local mechanical damage and crack initiation.

2.8 Electrochemical corrosion testing

Electrochemical corrosion behavior was investigated using a CorrTest CS300M potentiostat/galvanostat.

The tests were carried out in a 3.5 wt.% NaCl solution at 25 °C using a conventional three-electrode electrochemical cell consisting of:

- working electrode: investigated alloy sample with an exposed area of 0.64 cm²;
- reference electrode: Ag/AgCl;
- counter electrode: platinum electrode.

Potentiodynamic polarization measurements were performed to determine the corrosion potential (E_{corr}) and corrosion current density (I_{corr}). The corrosion parameters were calculated using the Tafel extrapolation method.

3. Results and Discussion

3.1 Chemical composition analysis (XRF)

The chemical composition of the investigated alloy in the as-cast condition after electroslag remelting (ESR) and after subsequent rolling (ESR-R) was determined by XRF analysis. The results are presented in [Table 1](#). The material is a multicomponent Fe-based alloy containing Cr, Ni, Ti, and Mo as the main alloying elements. In both conditions, Fe is dominant and exceeds 90 wt.%.

The detected alloying elements may contribute to improved mechanical, tribological, and corrosion-related properties. Cr and Ni can improve corrosion resistance, Mo may enhance resistance to localized corrosion, while Ti and V may participate in the formation of strengthening phases.

The slight differences in elemental contents between the ESR and ESR-R samples are most likely related to local chemical heterogeneity, surface condition, and the XRF measurement area, rather than to changes in the bulk composition caused by rolling. Overall, XRF analysis confirmed the Fe-based nature of the alloy and the presence of functional alloying additions.

Table 1. Chemical composition of the ESR and ESR-R alloys determined by XRF analysis.

Element	ESR alloy, wt. %	ESR-R alloy, wt. %
Fe	93.47 ± 0.02	91.35 ± 0.73
Cr	2.05 ± 0.01	2.33 ± 0.15
Ni	1.66 ± 0.02	3.13 ± 0.52
Ti	1.15 ± 0.03	1.38 ± 0.01
Mo	0.49 ± 0.00	0.62 ± 0.02
V	0.15 ± 0.003	0.18 ± 0.006
Cu	0.08 ± 0.005	0.14 ± 0.01
Co	0.05 ± 0.01	0.03 ± 0.03
Nb	0.01 ± 0.001	—
W	—	0.02 ± 0.017
As	—	0.02 ± 0.001

3.2 Phase composition and structural analysis (XRD)

[Fig. 1](#) shows the XRD patterns of the alloy in the ESR and ESR-R conditions. In the initial ESR state, the diffraction pattern is dominated by intensive peaks corresponding to the α -Fe crystalline phase. The main reflections located at $2\theta \approx 44$ – 45° , 65° , and 82° correspond to the (110), (200), and (211) crystallographic planes, respectively. The relatively narrow and symmetric diffraction peaks indicate a coarse-grained structure with a comparatively low level of internal stresses.

After plastic deformation by rolling (ESR-R condition), a significant modification of the diffraction pattern is observed. In addition to α -Fe reflections, additional peaks associated with oxide phases (Fe_2O_3 and Fe_3O_4), as well as possible carbide and intermetallic compounds such as Fe_3C , are detected. Moreover, noticeable peak broadening and slight peak shifts are observed, indicating grain refinement, increased dislocation density, lattice distortion, and the formation of residual stresses.

The broadening of diffraction peaks after rolling is associated with strain hardening and accumulation of crystal lattice defects during plastic deformation. The appearance of secondary phases may be related to redistribution of alloying elements and activation of diffusion processes during rolling.

Thus, the XRD analysis demonstrates that the transition from the cast ESR condition to the deformed ESR-R state is accompanied by substantial structural transformation involving grain refinement, increased defect density, and secondary phase formation, which significantly affects the mechanical, tribological, and corrosion properties of the alloy.

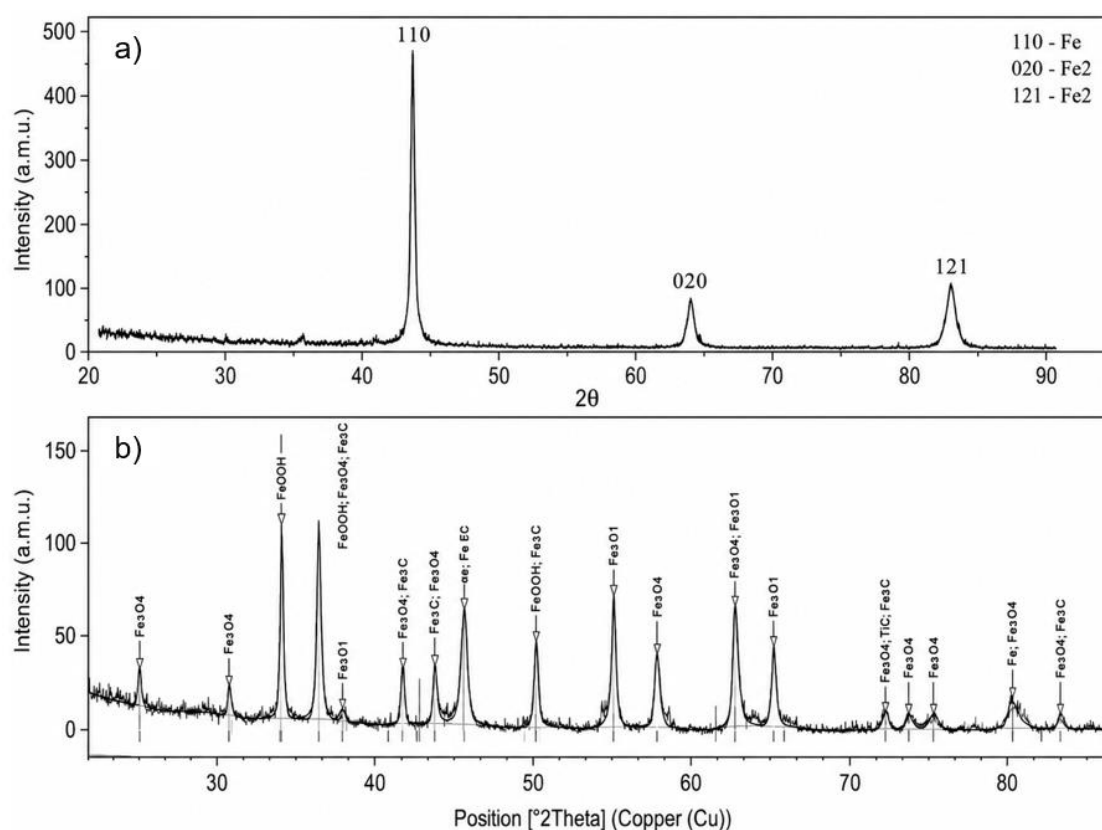


Fig. 1. XRD patterns of the investigated alloy in the ESR and ESR-R conditions.

3.3 Microstructure and elemental distribution (SEM/EDS)

SEM micrographs and EDS elemental mapping results for the alloy in the ESR condition are presented in [Fig. 2](#). The microstructure is characterized by structural heterogeneity with the presence of bright and dark regions corresponding to different phases and inclusions. Elemental mapping confirms that Fe is uniformly distributed within the matrix, while localized regions enriched with Cr, Ni, Mn, and Si are also observed.

Certain inclusions demonstrate enrichment in Ti, V, and Mo, indicating the possible formation of carbide or intermetallic phases. The observed chemical heterogeneity suggests the preservation of segregation effects typical for cast materials after electroslag remelting.

Following rolling deformation ([Fig. 3](#)), the microstructure becomes more oriented and homogeneous. The Fe-based matrix demonstrates a more uniform elemental distribution, while alloying elements are dispersed more evenly throughout the material. In addition, the size and frequency of local segregated regions are reduced, indicating a decrease in chemical heterogeneity after plastic deformation.

The obtained results suggest that rolling contributes to structural homogenization and redistribution of alloying elements within the alloy matrix.

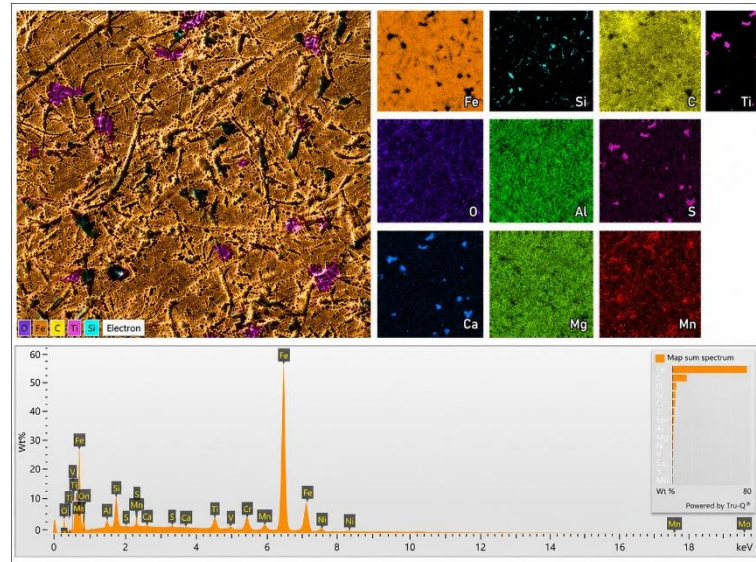


Fig. 2. SEM image and EDS elemental mapping of the alloy in the ESR condition.

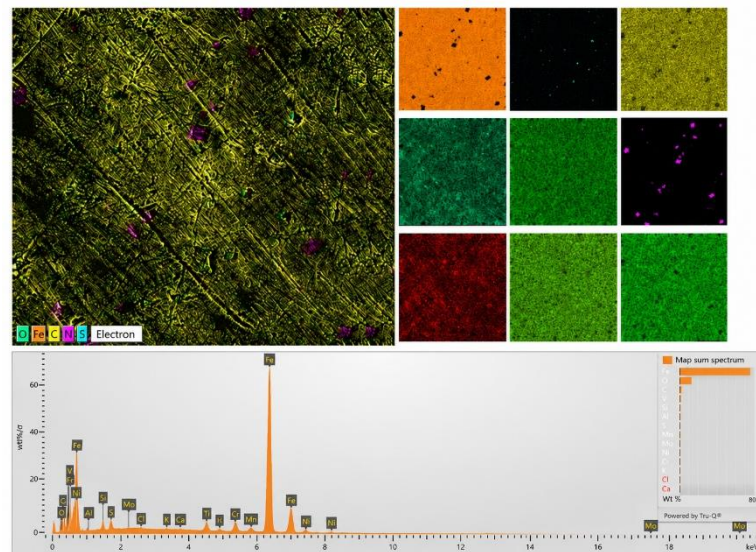


Fig. 3. SEM image and EDS elemental mapping of the alloy in the ESR-R condition.

3.4 Mechanical properties and indentation behavior

3.4.1 Microhardness

[Fig. 4](#) shows the Vickers microhardness results for the investigated alloy. In the ESR condition, relatively large indentation imprints are observed, corresponding to a lower hardness level. The average microhardness value is approximately 394 HV_{0.1}.

After rolling deformation (ESR-R), a substantial reduction in indentation size is observed, indicating increased resistance to localized plastic deformation. The average microhardness increases to approximately 914 HV_{0.1}, which is more than twice the value of the initial ESR condition.

The observed strengthening effect is mainly associated with grain refinement and increased dislocation density caused by plastic deformation. According to the Hall–Petch mechanism, grain refinement leads to enhanced resistance against dislocation motion, thereby increasing hardness and strength.

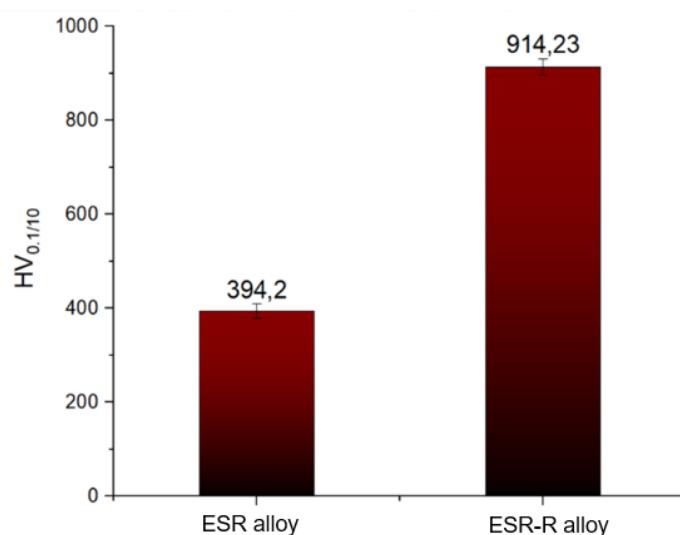


Fig. 4. Vickers microhardness values of the alloy in the ESR and ESR-R conditions.

3.4.2 Instrumented indentation

Additional instrumented indentation tests were performed to determine Martens hardness (HM), nanohardness, and elastic modulus values (Fig. 5). The ESR sample exhibits HM values of approximately 4500 N/mm² and nanohardness values of about 600–650 HV, while the elastic modulus is approximately 236–237 GPa.

After rolling, the HM values increase significantly to approximately 7400–7500 N/mm², whereas nanohardness reaches 1100–1150 HV. In contrast, the elastic modulus changes only slightly and remains within 228–229 GPa.

The obtained hardness values are in good agreement with the Vickers microhardness measurements, confirming the reliability and consistency of the experimental results.

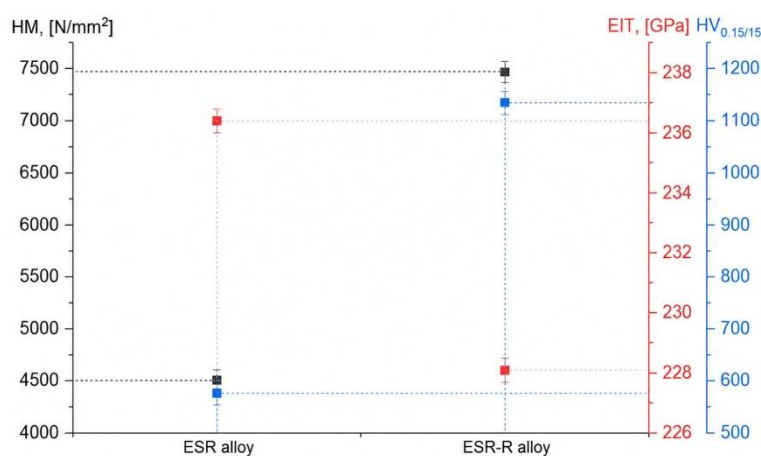


Fig. 5. Instrumented indentation results for the alloy in the ESR and ESR-R conditions.

3.5 Tribological behavior

3.5.1 Friction coefficient

The friction coefficient curves obtained using the ball-on-disk method (ASTM G99) are presented in Fig. 6. In both cases, the friction coefficient stabilizes after the running-in stage. However, the ESR sample demonstrates a higher average friction coefficient (~ 0.65) compared to the ESR-R sample (~ 0.59).

The reduction in friction coefficient after rolling indicates lower wear intensity and more stable friction behavior. The improved tribological performance is associated with increased hardness and structural homogenization after plastic deformation.

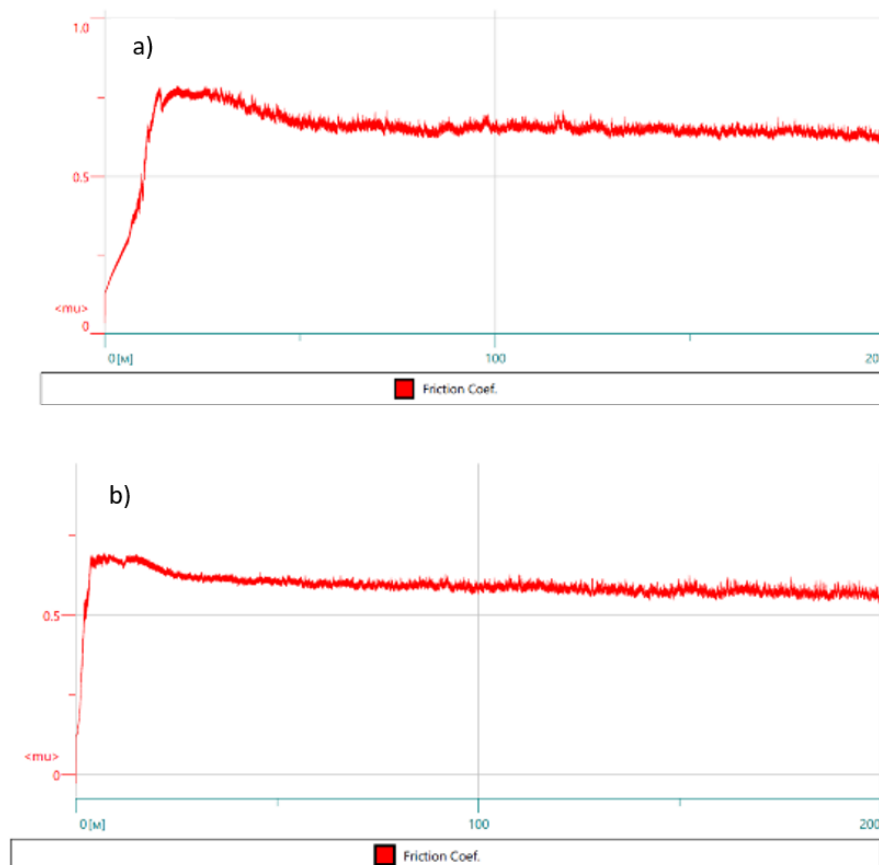


Fig. 6. Friction coefficient as a function of sliding distance for the ESR and ESR-R samples.

3.5.2 Wear track morphology and wear resistance

Wear track surface profiles obtained after tribological testing are shown in Fig. 8. The ESR sample exhibits a wider and deeper wear track with pronounced surface relief changes, indicating severe material removal and intensive plastic deformation during sliding.

In contrast, the ESR-R sample demonstrates a narrower and smoother wear profile with significantly lower wear depth. The cross-sectional wear area decreases from $2528.3 \mu\text{m}^2$ for the ESR sample to $621.7 \mu\text{m}^2$ for the ESR-R sample, corresponding to an approximately 4.1-fold reduction.

Simultaneously, the wear rate decreases from 1.588×10^{-5} to $3.906 \times 10^{-6} \text{ mm}^3/(\text{N}\cdot\text{m})$. The improved wear resistance is attributed to strain hardening, increased hardness, and enhanced structural uniformity of the rolled material.

4. Conclusion

This study investigates the effect of plastic deformation on the microstructure, mechanical, tribological, and corrosion properties of an alloy produced by the electrosag remelting (ESR) process.

It has been established that plastic deformation leads to a significant change in the material's microstructure: grain refinement, an increase in defect density, and a more uniform distribution of alloying elements are observed. This is accompanied by a significant increase in microhardness and an improvement in tribological characteristics, including a reduction in the coefficient of friction and wear rate.

It has been shown that the deformed state is characterized by higher hardness and resistance to local penetration; however, the increase in defect density contributes to increased brittleness and higher electrochemical activity of the material. Electrochemical studies in a NaCl solution confirmed an increase in the rate of corrosion processes following plastic deformation.

Thus, plastic deformation has a complex effect on the properties of the alloy under study: it promotes hardening and increased wear resistance, but simultaneously reduces corrosion resistance. The results obtained can be used in selecting operating conditions and developing processing technologies for materials produced by the ESR method.

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Conflict of interest

The authors declare that they have no competing interests.

References

1. Platonov A.D., Gurevich S.M. (1985) Electrosag Remelting of Metals. Moscow: Metallurgiya.
2. Kozlov V.A., Shapovalov V.A. (1990) Theory and Technology of Electrosag Remelting. Moscow: Metallurgiya.
3. Medovar B.I. (1974) Electrosag Remelting. Kyiv: Naukova Dumka.
4. Paton B.E., Medovar B.I. (1991) Electrosag Remelting and Casting. CRC Press.
5. Mitchell A., Smail R.M. (1979) Practical Aspects of Electrosag Remelting Technology. International Metals Reviews. 24(1), 231–264. <https://doi.org/10.1179/imtr.1979.24.1.231>
6. Dilthey U., Stein L. (2003) Electrosag Remelting Process: Fundamentals and Applications. Steel Research International.
7. Knyazev M.I., Lysenko A.A. (2001) Metallurgical Processes during Electrosag Remelting. Moscow: Mashinostroenie.
8. Holzgruber H., Steineder K. (2007) ESR Technology for High-Performance Steels. BHM Berg- und Hüttenmännische Monatshefte.
9. Ghosh A., Chatterjee A. (2008) Ironmaking and Steelmaking: Theory and Practice. PHI Learning.
10. Kablov E.N., Lukin A.I. (2012) Modern Technologies for Producing High-Quality Alloys. VIAM.
11. Li J., Thomas B.G. (2004) Modeling of Electrosag Remelting Process. Metallurgical and Materials Transactions B.
12. Wang Q., Zhang L. (2010) Inclusion Removal during Electrosag Remelting. Journal of Materials Processing Technology.
13. Kelkar K.M., Mok J., Patankar S.V., Mitchell A. (2004) Computational Modeling of Electrosag Remelting Processes. Journal de Physique IV France. 120, 325–332. <https://doi.org/10.1051/jp4:2004120048>
14. Zhukov A.A., Popov V.I. (2006) Special Methods of Metal Melting and Refining. Moscow: Metallurgiya.

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ORIGINAL STUDY

OPTIMIZATION OF REACTIVE MAGNETRON SPUTTERING
PARAMETERS FOR TION COATINGS DEPOSITED ON 316L STAINLESS
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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 $1.13 \times 10^{-7} \text{ A/cm}^2$ 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 $\text{Ar}/\text{N}_2/\text{O}_2$ 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 + N₂ + O₂ 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 cm^2 at room temperature ($25\text{ }^{\circ}\text{C}$) 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.

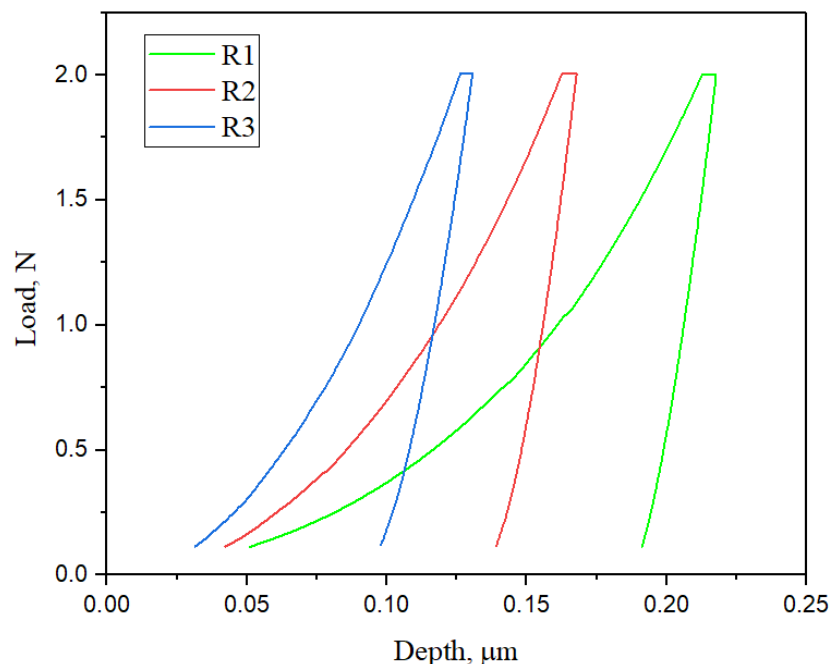


Fig. 1. Instrumented indentation load–depth curves of TiON coatings deposited under R1, R2, and R3 sputtering regimes.

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/N₂/O₂ 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/oxy-nitride 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.

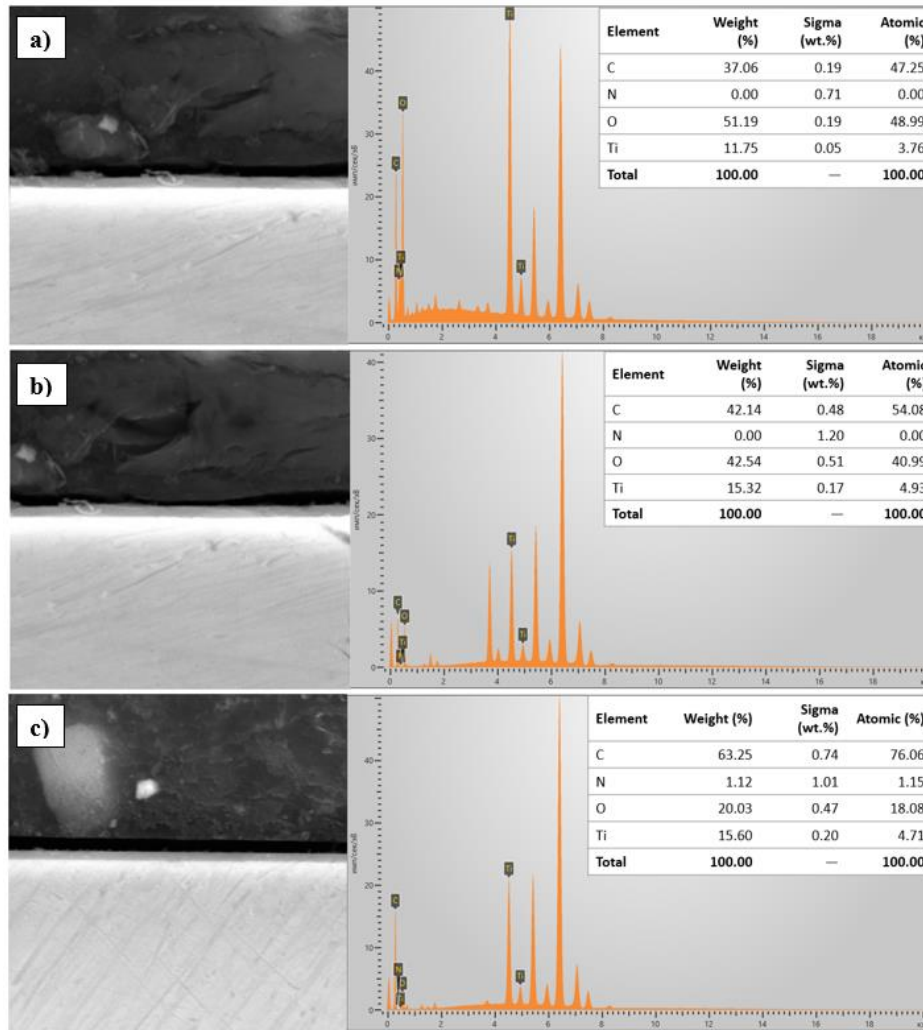


Fig. 2. Cross-sectional SEM micrographs, EDS spectra, and elemental compositions of TiON coatings deposited under different sputtering regimes: (a) R1, (b) R2, and (c) R3.

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](#).

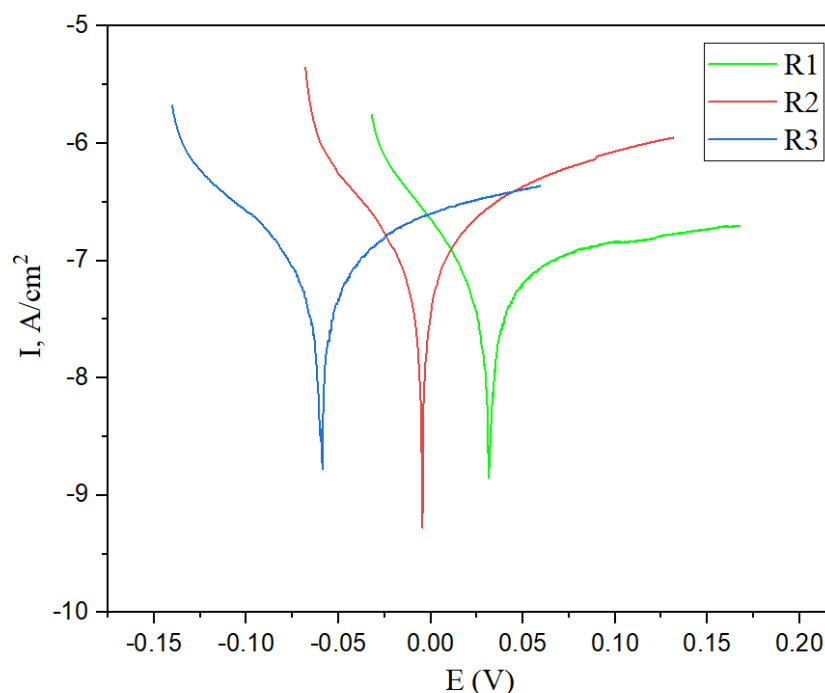


Fig. 3. Potentiodynamic polarization curves of TiON coatings deposited under different sputtering regimes in 3.5 wt.% NaCl solution.

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 (E_{corr}) 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/cm² and the lowest corrosion rate of 0.00231 mm/year. In contrast, R2 showed the highest corrosion current density (2.22×10^{-7} A/cm²) and the highest corrosion rate (0.00451 mm/year), indicating the lowest corrosion resistance. Sample R3 demonstrated intermediate behavior with an i_{corr} value of 1.79×10^{-7} A/cm² 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.

Reference

1. Kengesbekov A., Rakhadilov B., Kussainov A., Serikbaikyzy A., Askhatov A., Aringozhina Z. (2025) Formation of Titanium Oxynitride Films by Reactive Magnetron Sputtering, Their Structural Features and Properties. *Coatings*. 15(12), 1434. <https://doi.org/10.3390/coatings15121434>

2. Pană I., Braic V., Dinu M., Mouele E.S.M., Parau A.C., Petrik L.F., Braic M. (2020) In Vitro Corrosion of Titanium Nitride and Oxynitride-Based Biocompatible Coatings Deposited on Stainless Steel. *Coatings*. 10(8), 710. <https://doi.org/10.3390/coatings10080710>
3. Saqib M., Kuzmin O.S., Kraśkiewicz H., Wasyluk Ł., Cuniberti G., Ficai A.A., Pichugin V.F., Beshchasna N. (2021) Evaluation of In Vitro Corrosion Behavior of Titanium Oxynitride Coated Stainless Steel Stents. *IEEE Access*. 9, 59766–59782. <https://doi.org/10.1109/ACCESS.2021.3072597>
4. Dadafarin H., Moradi M., Shahryari A., et al. (2024) Modification of 316L Stainless Steel Surfaces for Biomedical Applications: A Review. *Molecules*. 29(20), 4927. <https://doi.org/10.3390/molecules29204927>
5. Liu C.L., Chu P.K., Lin G.Q., Yang D.Z. (2006) Anti-Corrosion Characteristics of Nitride-Coated AISI 316L Stainless Steel Stents. *Surface and Coatings Technology*. 201(6), 2802–2806. <https://doi.org/10.1016/j.surfcoat.2006.06.019>
6. Lemdani F., Belkahla S., Chihi T., et al. (2025) A Comparative Study of Titanium-Based Coatings Deposited by Magnetron Sputtering for Biomedical Applications. *Engineering Proceedings*. 81(1), 9. <https://doi.org/10.3390/engproc2025081009>
7. Buranych V.V. et al. (2023) Characterization, Mechanical and Biomedical Properties of Titanium Oxide-Nitride Coating. *Ceramics International*. <https://doi.org/10.1016/j.ceramint.2023.06.070>
8. Cubillos G.I. et al. (2021) ZrN-ZrOxNy vs ZrO2-ZrOxNy Coatings Deposited via Reactive Magnetron Sputtering: Anti-Corrosion Characterization and Biocompatibility. *Scientific Reports*. <https://doi.org/10.1038/s41598-021-98052-2>
9. Dorofeeva T. et al. (2022) Si-Al-N-O Multi-Layer Coatings with Increased Corrosion Resistance Deposited by Magnetron Sputtering. *Metals*. <https://doi.org/10.3390/met12020254>
10. ISO 14577-1 (2015) Metallic Materials – Instrumented Indentation Test for Hardness and Materials Parameters – Part 1: Test Method. International Organization for Standardization (ISO), Geneva, Switzerland.

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