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Formation of TiN coatings by air plasma spraying

Titanium nitride (TiN) coatings were obtained on the surface of 12Kh18N10T steel by air plasma spraying (APS) of TiN powders using an arc plasmatron made by the authors. The plasmatron has a node of circular input and gas-dynamic focusing of the powder and the output apertures of the nozzle-anode are made in the form of rectangular narrowing-expanding channels (No.34334 RK: IPC H05H 1/42). A study of operation modes of a plasmatron for spraying of powder coatings was carried out. The structural-phase state, microhardness and wear resistance of TiN coatings were systematically investigated. The optimum APS operating mode for deposition of TiN powder was determined: current 250 A, voltage 68 V, argon gas flow 34 L/min, spraying distance 150 mm. To reduce the oxidation of TiN powder in the APS process, a method of creating a nitrogen environment at the outlet of the anode nozzle, nitrogen flow rate 2.3 bar was used. The results of structural analysis showed that TiN is the main phase of the coating. The mechanism of formation of TiN structures was characterized by analyzing SEM results of TiN coating surface morphology and TiN droplets sprayed on the surface of the sample. The results showed that the TiN(1) coating has better wear resistance than the TiN(2) and TiN(3) coatings. The cross-sectional and longitudinal microhardness of the TiN coating was investigated. The highest cross-sectional hardness of TiN coating is 1250 HV0.1, which is in accordance with mode 1.

Keywords: plasmatron, air plasma spraying, TiN, wear resistance, SEM analysis, anode nozzle.

Introduction

In recent years in all industrially developed countries technologies of creation of wear-resistant materials based on nitrides and methods of coating from them are intensively developed. Titanium nitride (TiN) has excellent wear resistance, erosion resistance, heat resistance and low friction coefficient [1-3], so it is widely used in some areas as the solid, wear-resistant coatings. The choice of methods for applying TiN coatings is determined by the geometric parameters of the coated parts and products, their design and technological features, conditions of future operation, as well as the required thickness of the functional protective coating [4,5]. The only factor uniting all the available methods is thermal influence in the process of applying a protective coating, necessary for formation of a stable adhesive bond of coating with a substrate.

The following coating deposition methods are currently most widely used in industry [6-9]: detonation spraying; high velocity oxide-fuel spraying (HVOF); air plasma spraying. Each of the above methods has its own advantages and disadvantages determining its effective area of application, but the first two methods can be implemented only in the presence of special chambers and gas communications. Moreover, their application is also limited by energy problems arising during heating of large-sized parts, since the required density and adhesion are achieved by the subsequent heat treatment of the formed layer. Therefore, one of the most economical and easy-to-implement methods of applying TiN coatings is the air plasma spraying method, which allows to form and melt the coating in one operation.

The essence of air plasma spraying is that powder particles pass through a zone of ionized gas (plasma) formed by an electric gas discharge, are melted and deposited on the substrate surface heated in the contact zone in the course of coating formation [10, 11]. The main element of technological equipment is a plasma torch that combines the functions of a plasma source and atomizer of disperse material. Plasma-forming gas (argon, nitrogen, air) passes through the electric arc zone, is ionized and exits through the nozzle of the plasma gun in the form of a plasma jet (flow). A significant disadvantage of this technology so far is the low thermal and overall efficiency of the process, amounting to only 3-8 %, which is an urgent problem of air-plasma spraying. In most technological processes of plasma treatment of materials, electric arc linear plasmatrons are most commonly used, which, compared to plasmatrons of other circuits, have a simple design, a

relatively long life of electrodes, the possibility of controlling the discharge power not only by changing the arc current, but also by changing the voltage at the arc.

Thus, the purpose of this study is to obtain wear-resistant nitride coatings by air plasma spraying.

Technology

Depending on the set tasks, an experimental air plasma spraying installation designed for surface treatment with plasma and application of powder coatings at atmospheric pressure was developed for protective coatings application.

The installation includes power supply, control panel, switching module with start-up unit, dispenser and autonomous plasma torch cooling unit. The installation is equipped with a plasma torch, which can be used in both manual and mechanized versions.

Fig. 1 shows schematically the installation for powder coatings, showing: plasmatron 1 which consists of anode 2, cathode 3, interelectrode ceramic insert 4, tubes 5 for feeding plasma gas — argon 6, tubes 7 for feeding inert gas — nitrogen 8 and sprayed powder 9, fittings for inlet 10 and outlet 11 of cooling fluid 12, powder batcher 13, power supply 14, off-line unit of water cooling 15.

The installation works as follows. Before starting work, the cooling system is switched on. Distilled water is used to cool the plasmatron, which enters the cavity of the anode assembly housing through a fitting and then the heated water is discharged through a fitting. The coolant is in circulating mode and the temperature of the coolant is automatically regulated by an autonomous water cooling module. Then the plasma-forming gas-argon is fed through the radial feed tube of the cathode assembly, to the discharge area of the plasmatron. When voltage is applied to the electrodes, an electric arc occurs between the nozzle-anode and the cathode and the plasma-forming gas – argon is ionized and exits the nozzle-anode at high speed, forming a plasma stream. Then the metering device is connected and through the inert gas channel the powder to be sprayed is fed into the plasma stream.

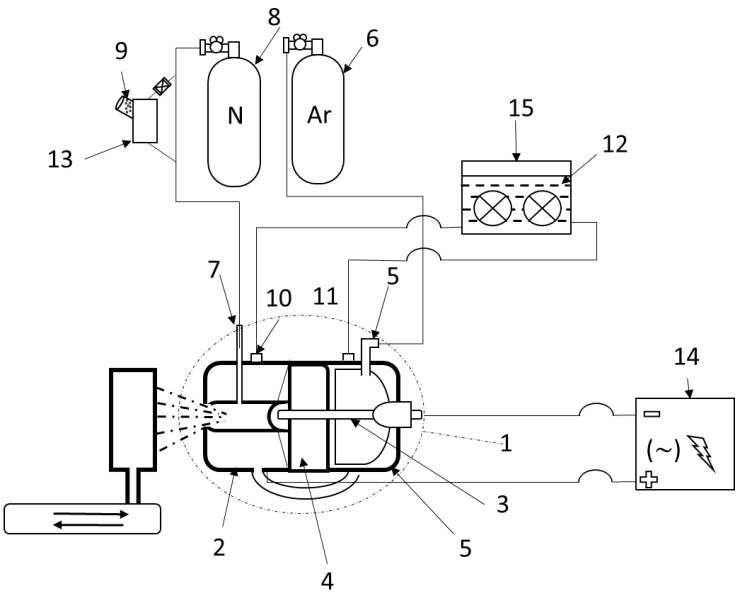
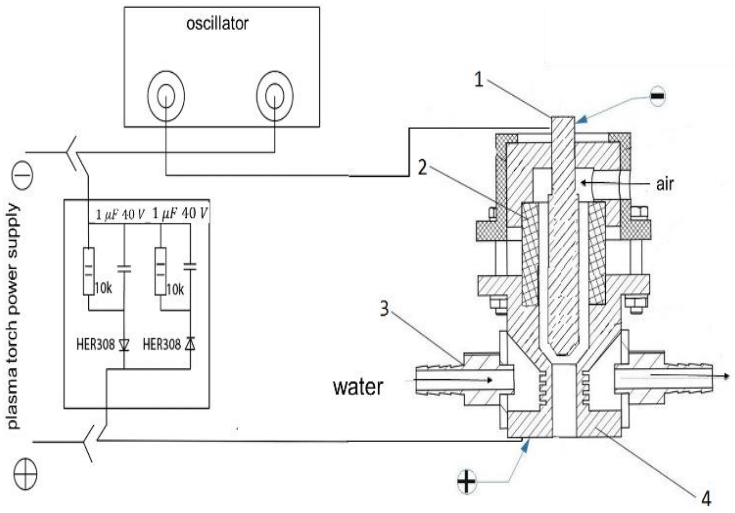


Figure 1. Schematic diagram of a powder coating installation

Plasmatron (Figure 2) is a thermal plasma generator of DC coaxial design with tungsten cathode with diameter of 5 mm, embedded in the copper rod in the center, and all-welded nozzle anode of copper, which has a radiator profile, which will allow to disassemble and assemble the plasmatron during repair work without deteriorating its quality. The key element of the plasmatron design is a node of annular input and gas-dynamic focusing of the powder, as well as the outlet holes of the nozzle-anode are made in the form of rectangular tapering-expanding channels. This design scheme provides input of the sprayed powder into the axial high-temperature and high-speed part of the plasma flow, which significantly increases the efficiency of heating and acceleration of particles and sputtering productivity [12]. The lifetime of the plasma torch is more than 200 hours, the cathode is replaced after 50 hours of operation. The plasma torch is capable of sta-

tionary operation with various media, primarily with oxidizing media. AC electric arc plasmatron operates in argon (nitrogen, air, gels) with a maximum power of up to 34 kW at a low pressure of about 0.01 MPa.



1 — cathode, 2 — insulator, 3 — cooling connector, 4 — anode
Figure 2. Scheme of connection of the plasma torch to the power supply

Materials and Methods

Stainless steel 12Kh18N10T (0.12 % C, 18 %Cr, 10 %Ni, Ti) was used as a substrate material. To improve the adhesion between the coating and the substrate, the flat surface of a 50×50×4 mm steel sample was cleaned in acetone and sandblasted before spraying. TiN powder with a dispersion of 15-40 μm was used for coating spraying (Figure 3).

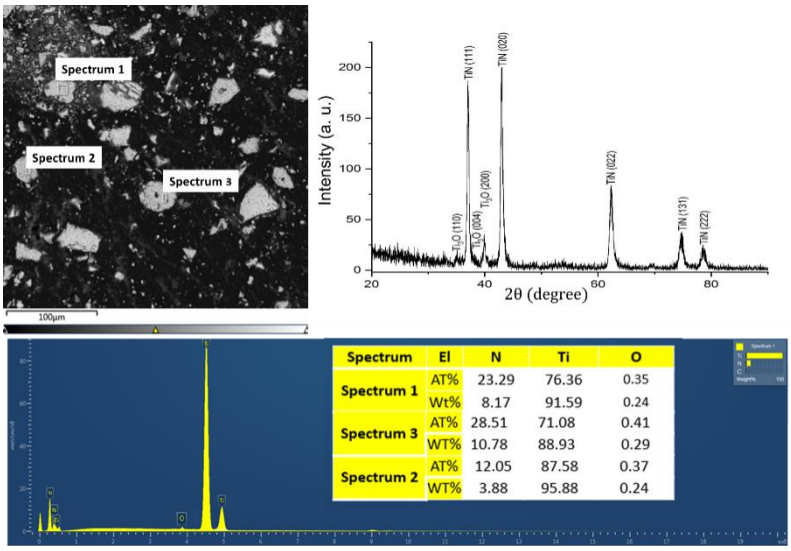


Figure 3. Structure-phase states of sprayed titanium nitride powder

An air-plasma system was used for the experiment, the design of which is shown in Figure 1. First, the substrate was preheated to 250 °C using a plasma jet, which was controlled by a HT-819 pyrometer. Then, TiN coatings were applied to the substrate using a plasma torch. The surface morphology was studied by scanning electron microscopy on a JSM-6390 scanning electron microscope with an energy dispersive spectrometer (EDS).

The microhardness of the samples was measured by a diamond indenter on the device Metolab 502 (Russia) in accordance with GOST 9450-76 [13], at a load of 100 g and exposure time of 10 s. The samples were tested for abrasive wear on the experimental stand (Figure 4) against soft stationary abrasive particles

by the scheme "rotating roller — flat surface" in accordance with GOST 23.208-79, which corresponds to the American standard ASTM C 6568 [14].

Measurements of corrosion resistance were carried out on potentiostat-galvanostat "P-150", using a three-electrode connection scheme to the electrochemical cell using a chlorosilver reference electrode and an auxiliary platinum electrode. For corrosion resistance tests, a 3 % NaCl solution in distilled water was used as an aggressive medium. Measurements were carried out at ambient temperature 20±2 °C.

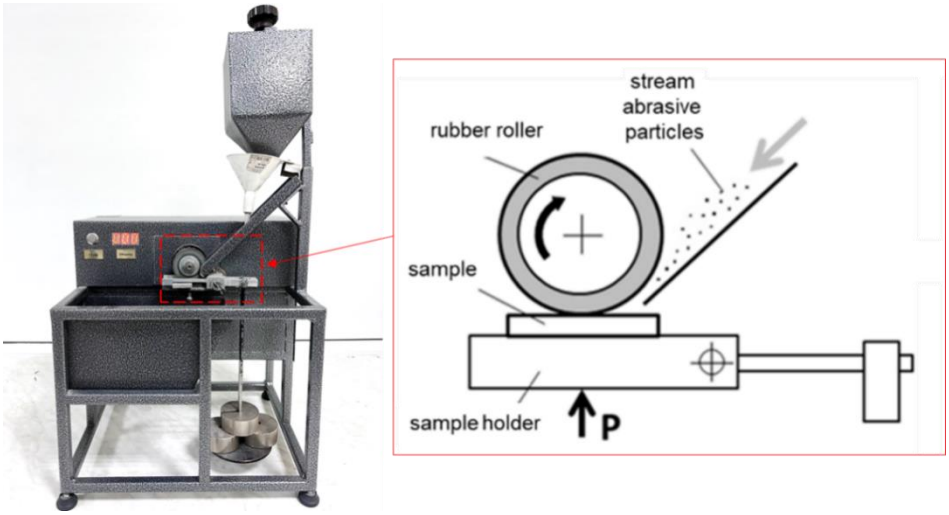


Figure 4. The experimental test stand for testing of samples abrasive wear according to the “rotating roller–flat surface” scheme

The formation of a dense homogeneous coating layer is influenced not only by the spraying parameters, but also by the quality of the powder. In addition, the main quality parameters of air plasma spraying, which include hardness, layer thickness and wear resistance, depend on several factors: spraying distance, current strength, voltage, gas pressure. Among these factors, electric current and working gas (Ar) pressure play an important role. In this regard, for the spraying modes the corresponding technological parameters listed in Table 1 were selected.

Table 1

Modes of air plasma spraying

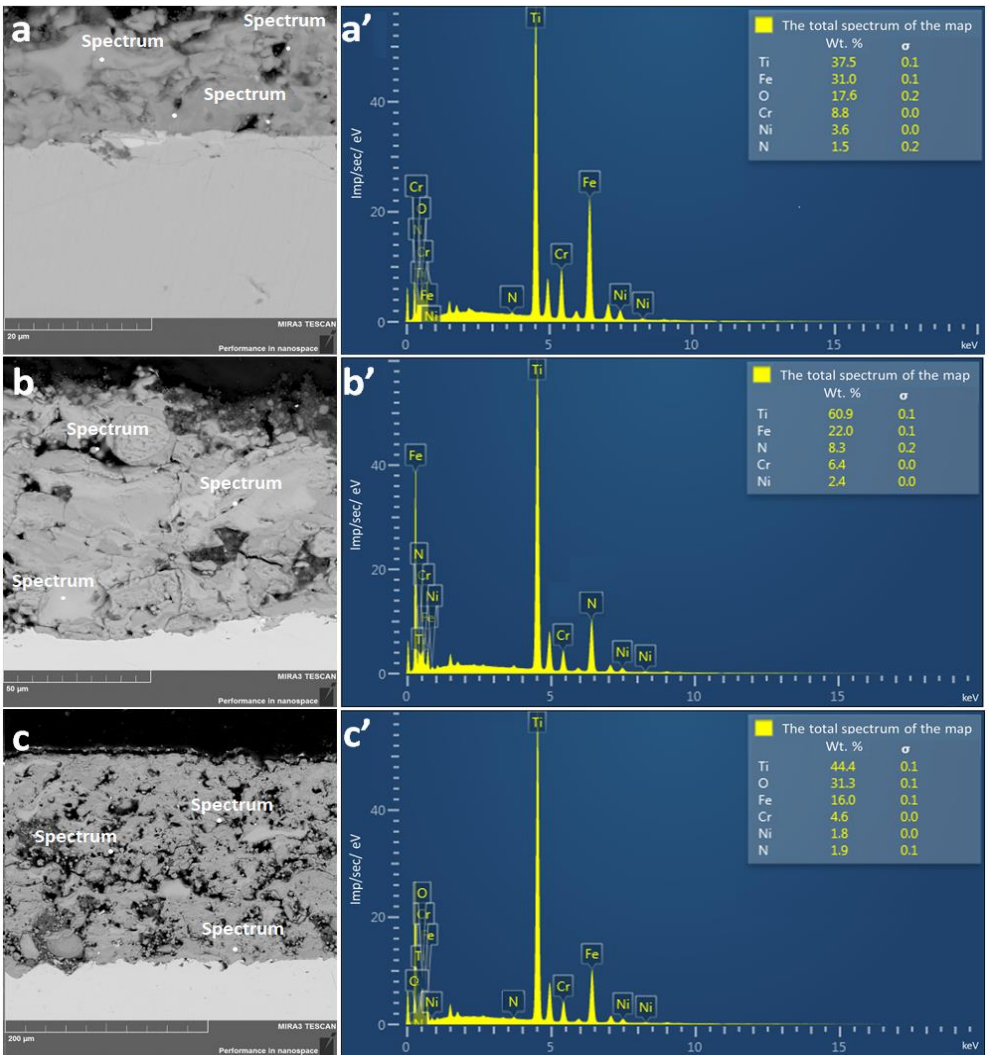
Sample	Spraying distance, mm	Gas flow rate Ar, ltr/min	Gas flow rate N, bar	Current, A	Processing time, s	Coating thickness, μm	Voltage, V
TiN(1)	150	35	2.4	250	60	~70	68
TiN(2)	150	35	2.7	350	60		
TiN(3)	150	35	3	450	60		

Results and Discussion

The microstructure of the cross-sectional microslip is shown in Figure 5. The cross-sectional microslip shows porosity in the applied layer, probably having a diffusion character. The microstructure of 20 μm thick TiN(1) coating is characterized by high density, low porosity, smooth boundary with the substrate. The volume fraction of the pores formed at large distances from each other is 0.13 % of the surface area of the coating. There are also very small pores formed, apparently, as a result of degassing of the molten material of the particles during their crystallization.

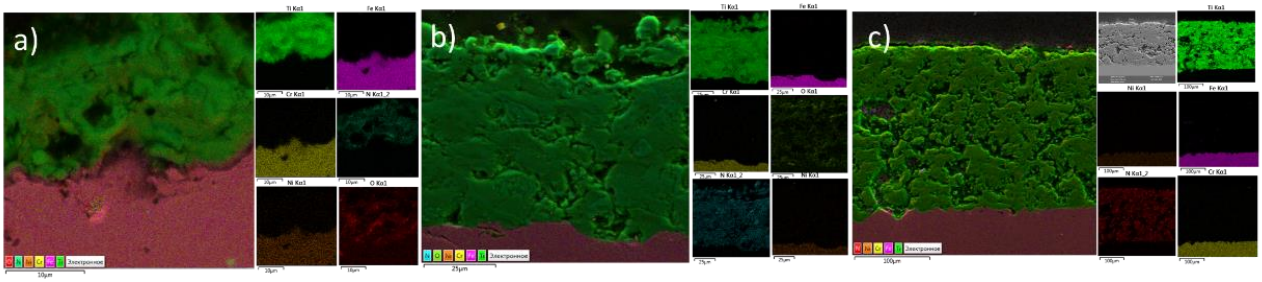
Fig. 5 b, c shows the microstructure of TiN(2) and TiN(3) coatings, which is characterized by the presence of porosity throughout the thickness, the pores have an irregular shape and sizes from a few microns to 10 μm. The porosity of the TiN coating is 2.96 % and 8.04 % at 75 and 200 μm thickness, respectively. The coating section at the border with the substrate is characterized by a more uniform structure. The structure with few pores should be attributed to the gas that exists between the tiny liquid droplets and does not have time to be released during coating formation, which seems to contribute to the formation of small character-

istic micro-cracks in the thick coating structures seen in Figure 5 b, c. Therefore, to this day, the problem of reducing pores and cracks, as well as improving the structure of coatings remains an urgent task that requires thorough research.



Mode 1 (20 μm), (b) mode 2 (75 μm), (c) mode 3 (200 μm)
Figure 5. SEM images of cross-sectional morphology of TiN coating

From the mapping data in Figure 6 we can also conclude that the distribution of the main components (Ti, N, Cr, Ni, O and Fe) is uniform throughout the thickness of the coatings and in the substrate. In accordance with the data on the distribution of elements in the surface layer obtained both in the mapping mode and in the point mode (Figure 6), the layer consists of pronounced spectra of Ti and N, demonstrating the contrasting composition of TiN coatings. According to this, it can be assumed that the predominant phase in this area is TiN.



a)TiN(1); *b) TiN(2);* *c)TiN(3)*

Figure 6. Cross-section morphology and corresponding EDS maps of coating samples

The study of the structure was supplemented by microhardness measurements along the entire cross section of the coating. Figure 7 shows the results of microhardness measurements of TiN coatings of different modes. All coatings in general are characterized by high microhardness. Similar results were obtained in [15, 16]. The dependence of microhardness on an arrangement of measuring points on height of a layer is not found. The comparatively high hardness is observed on the surface of the coating processed by mode 1, has value 1250 $HV_{0.1}$. This may be due to the low content of pores and cracks in the structure of the sample TiN(1).

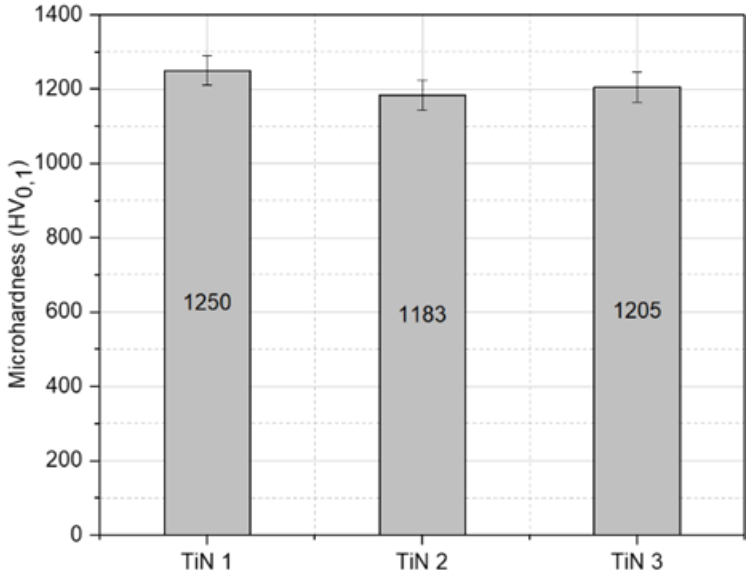


Figure 7. Results of the microhardness of TiN coatings obtained by the APS method

An abrasion test was performed to evaluate the wear resistance of the coatings. The abrasion results in terms of mass loss and relative wear resistance for the coatings are shown in Figure 8. It can be seen that there was little difference in the resistance against abrasive wear of the samples. The best material among all the coatings studied was TiN(1), which showed a higher abrasion resistance compared to the coatings obtained by the TiN(2) and TiN(3) modes, by about 83-87 %.

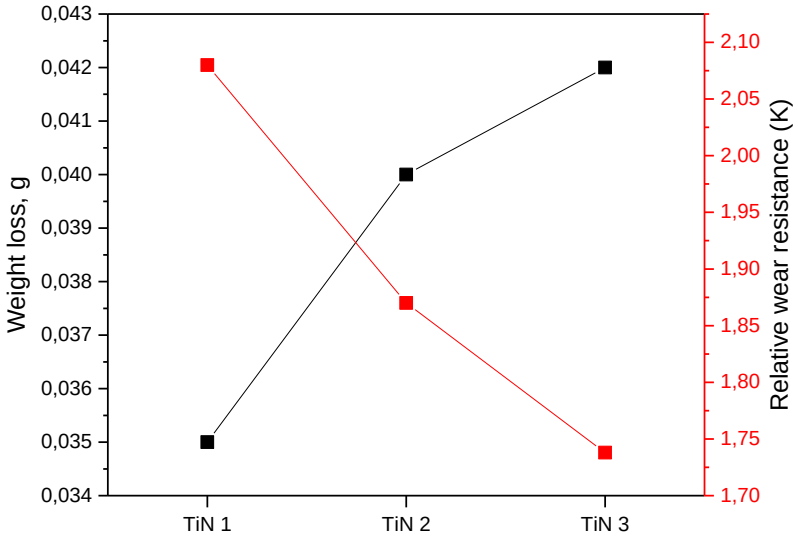


Figure 8. Results of mass loss and relative wear resistance of coatings

According to the thermodynamics of corrosion, a higher intrinsic corrosion potential can lead to greater resistance to the electrochemical reaction, indicating a stronger ability to resist the addition and loss of electrons, as well as better corrosion resistance of the material. Generally, based on corro-

sion kinetics, the lower the intrinsic corrosion current density, the higher the corrosion resistance [17, 18]. Figure 9 shows that the corrosion resistance of TiN(1) coatings is better than that of TiN(2) and TiN(3) coatings. In the coatings obtained by TiN(2) and TiN(3) modes the corrosion rate Rcorr =0.905 cm/year and Rcorr =0.486 cm/year respectively is higher than that of TiN(1) coating with value Rcorr=0.078 cm/year (Table 2). It was determined that the corrosion resistance reaches the highest value when spraying under the regime TiN(1).

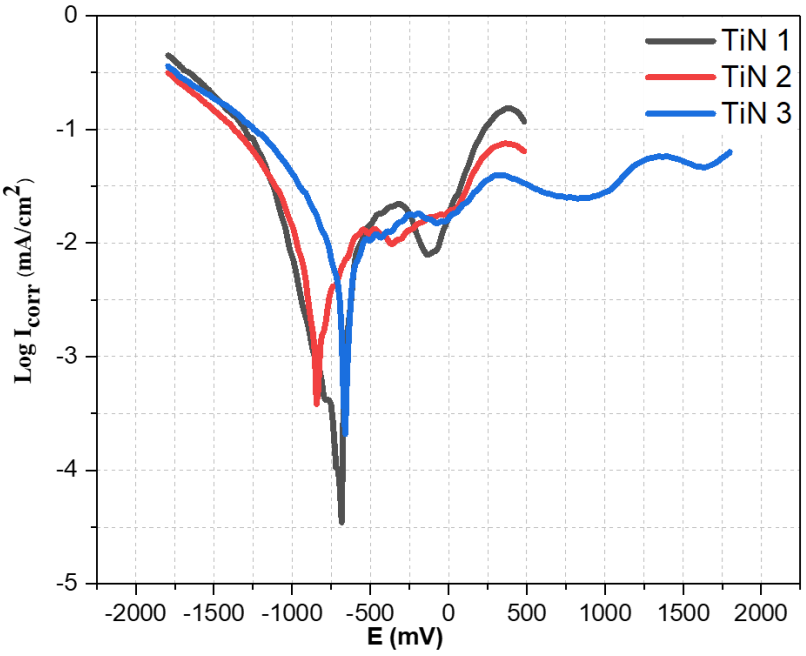


Figure 9. Polarization curves of coatings obtained by the APS method

Table 2

Correlation table of obtained results

Sample	Microhardness, HV0.1/10	Abrasive tests		Corrosion tests		
		Weight loss, g	Wear resistance coefficient	m, g	l, cm	R_{corr} cm/year
TiN 1	1250	0.035	2.08	0.317	0.078	0.078
TiN 2	1183	0.040	1.87	3.556	0.905	0.905
TiN 3	1205	0.042	1.73	1.910	0.486	0.486

Conclusion

This article presents the results of research work on obtaining coatings from titanium nitride by air plasma spraying, as well as an analysis of the evaluation of microhardness values, corrosion and abrasion characteristics. The received results allow to draw the following conclusions. It is established that the use of inert gas at the APS of thin-film coatings from titanium nitride allows to change the chemical composition of the coating and its structure. Under optimal gas pressure conditions of deposition, thin film coatings of stoichiometric composition with improved antifriction properties are formed. At nitrogen pressure equal to 2.3 bar (mode 1), the wear resistance coefficient of the formed coatings decreases practically 1.5 times compared to its value for modes 2 and 3. Increasing nitrogen pressure up to 3 bar causes the formation of a porous coating structure with a developed surface relief and, as a consequence, leads to a deterioration of the coatings' tribological properties.

It was determined that the value of microhardness increased and amounted to 1250 $HV_{0.1}$, which apparently contributed to a decrease in wear of TiN coatings in the realized friction conditions.

It was revealed that the application of TiN coatings improves the corrosion resistance of stainless steel 12X18H10T; in particular the corrosion rate is reduced, indicating that the coating has good stability and excellent protection.

Thus, the conducted studies have shown the prospects and feasibility of using the APS technology to improve the wear resistance of stainless steel 12Kh18N10T.

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Ауа-плазмалық бүрку арқылы TiN жабындарын қалыптастыру

Титан нитриді (TiN) жабындары 12Х18Н10Т болатының бетіне TiN ұнтақтарын ауа-плазмалық бүрку (АПБ) әдісімен, өзіміз әзірлеген доғалық плазмотронда алынды. Аталған плазмотронда сақиналы енгізу және ұнтақты газодинамикалық фокустау торабы бар, сондай-ақ саптама-анодтың шығу тесіктері тікбұрышты конустық-кеңейетін арналар түрінде орындалған (№ 34334 ҚР: МПК H05H 1/42). Ұнтақты жабындарды бүрку үшін плазмотронның жұмыс режимдеріне зерттеу жүргізілді. TiN жабындарының құрылымдық-фазалық күйі, микроқаттылығы және тозуға төзімділігі жүйелі түрде зерттелді. TiN жабынын алу үшін ауа-плазмалық бүркудің оңтайлы режимі анықталды: ток 250 А, кернеу 68 В, аргон газының шығыны 34 л/мин, бүрку қашықтығы 150 мм. Ауа-плазмалық бүрку процесінде TiN ұнтағының тотығуын азайту үшін саптаманың — анодтың шығысында азотты ортаны құру әдісі қолданылды, азот шығыны 2,3 бар болды. Құрылымдық талдау нәтижелері TiN жабынның негізгі фазасы TiN екенін көрсетті. TiN құрылымдарының түзілу механизмі TiN жабынының беткі морфологиясын және үлгі бетіне шашыраған TiN тамшыларын СЭМ талдауымен сипатталды. Нәтижелер TiN(1) жабынының TiN(2) және TiN(3) жабындарына қарағанда тозуға төзімділігі жақсы екенін көрсетті. TiN жабынының көлденең және бойлық қималарының микроқаттылығы зерттелді. TiN жабынының көлденең қимасының ең жоғары қаттылығы 1250 $HV_{0,1}$ көрсетті. TiN жабынын қолдану 12Х18Н10Т тот баспайтын болаттың коррозияға төзімділігін жақсартатыны анықталды, атап айтқанда коррозия жылдамдығы төмендейді, бұл жабынның жақсы тұрақтылығы мен тамаша қорғанысы бар екенін көрсетеді.

Кілт сөздер: плазмотрон, ауа-плазмалық бүрку, TiN, тозуға төзімділік.

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Формирование TiN покрытий методом воздушно-плазменного напыления

Покрытия из нитрида титана (TiN) были получены на поверхности стали 12Х18Н10Т методом воздушно-плазменного напыления порошков TiN с помощью дугового плазмотрона собственной разработки. Плазмотрон имеет узел кольцевого ввода и газодинамической фокусировки порошка, а также выходные отверстия сопла-анода выполнены в виде прямоугольных ссужающихся-расширяющихся каналов (№ 34334 РК: МПК H05H 1/42). Проведено исследование режимов работы плазмотрона для напыления порошковых покрытий. Систематически исследованы структурно-фазовое состояние, микротвердость и износостойкость покрытий TiN. Определен оптимальный режим ВПН для нанесения порошка TiN: ток 250 А, напряжение 68 В, расход газа аргон 34 л/мин, дистанция напыления 150 мм. Для снижения окисления порошка TiN в процессе APS был использован способ создания азотной среды на выходе из сопла-анода, расход азота — 2,3 бар. Результаты структурного анализа показали, что TiN является основной фазой покрытия. Механизм формирования TiN структур был охарактеризован путем анализа СЭМ результатов морфологии поверхности покрытия TiN и капель TiN, распыленных на поверхность образца. Результаты показали, что покрытие TiN (1) обладает лучшей износостойкостью, чем полученные по режимам TiN (2) и TiN (3). Исследована была микротвердость поперечного и продольного сечений покрытия TiN. Наибольшая твердость поперечного сечения покрытия TiN составила 1250 HV 0.1, что соответствует режиму 1.

Ключевые слова: плазмотрон, воздушно-плазменное напыление, TiN, износостойкость, анализ СЭМ, сопло-анод.

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