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CORROSION DURABILITY OF NANOSTRUCTURED TiAlYN COATINGS, DEPOSITED BY PIII&D METHOD FROM FILTERED VACUUM-ARC CATHODIC PLASMA

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The electrochemical characteristics of nanostructured TiAlYN coatings deposited on 12X18H10T steel substrates are investigated in 3% aqueous solution of NaCl. The coatings were deposited from filtered vacuum-arc plasma by PIII&D method. Measurements of corrosion potentials E_{cor} and anodic polarization curves showed that the best protection against galvanic corrosion (GC) provides TiA1YN coating containing 0.2 at.% Y deposited on the substrate when applying pulsed bias potential of -1.5 kV amplitude. Addition DC bias potential of -150 V to pulse one leads to deterioration of the protective properties: activation potential of anodic processes (APAP) decreases in 3…4 times. Increase in TiA1YN coatings deposition rate promotes improvement their protective properties due to improved adhesion and decreased level of residual stress.

INTRODUCTION

Plasma immersion ion implantation and deposition (PIII&D) method combined with cathodic-arc plasma filtering allows fabricate high quality functional coating on various materials with good adhesion. In KIPT were developed a number of technological processes of synthesis of nanostructured nitride coatings based on TiN [1–5] by this method using the rectilinear filtered cathodic vacuum arc plasma source (RFCVAPS) of original design [6] when a pulsed high voltage bias potential is applied to the substrate. Nitride coatings of Ti-Al-Y-N system, synthesized by this method have a nanocrystalline structure with a cubic lattice of NaCl type, with the size of coherent scattering regions varied in the range of 10…22 nm [4].

High quality filtering of the plasma stream from macroparticles as implemented in the source, in combination with minimizing loss of plasma in the filtration process through the use of a new method for transporting the plasma flows from the cathode to the substrate [6] provides not only high quality of the synthesized coatings but also high productivity of the process of synthesis [7]. Using the multicomponent alloy cathodes of different elemental composition manufactured by “Real” LTD, Zaporozhye in the developed processes allows produce the nitride nanostructured coatings, in particular of Ti-Al-Y-N system, with unique physical and mechanical properties: high heat resistance and high resistance to cavitation and abrasion wear by compared to TiN coatings [8, 9].

Nitride coatings have previously been proposed to protect against galvanic corrosion (GC). In [10] the electrochemical properties of aluminum nitride as a protective coating on bioengineering materials in the model physiological solution (0.9% – NaCl) were investigated. Studies showed that the protective properties of the coatings are strongly dependent on the mode of their deposition. It is interesting to study the possibilities of using multi-component nitride coatings [8, 9] to protect against GC.

The aim of this work is to study the influence of the yttrium content as well as deposition parameters on the protective properties of TiAlYN coatings, deposited on 12X18H10T stainless steel by PIII&D method from the filtered flow of cathodic vacuum arc plasma against galvanic corrosion in a 3% solution of NaCl.

1. EXPERIMENTAL

TiAlYN coatings deposition was carried out from filtered flows of cathodic vacuum arc plasma using an improved version of RFCVAPS [5]. RFCVAPS provides high deposition rate of 10…20 μ/h at an arc current of 100 A with the heterogeneity of the coating thickness of ±5% inside the circle of 180 mm in diameter.

The coatings were deposited on the polished substrates of 12X18H10T stainless steel of 17×20×0.6 mm size. For deposition the nanostructured TiAlYN coatings with different Y content the cathodes of $Ti_{0.5-x}Al_{0.5}Y_x$ composition where x = 0; 0.002; 0.004 and 0.01 have been used.

In each deposition process the substrates were placed along the axis of the vacuum chamber in three positions at three different distances L from the RFCVAPS outlet: 1st position – L = 150 mm, 2nd position – L = 210 mm, and a 3rd position – L = 360 mm.

Before placing in a vacuum chamber, the substrates were cleaned with acetone and alcohol in an ultrasonic bath for 15 minutes. During coating deposition the substrate was supplied by pulsed bias potential of -1.5 kV amplitude, and in some experiments, in addition to the pulses had a DC potential -150 V. For coatings which were deposited only at pulsed bias potential, in the intervals between pulses the substrate was at a self-consistent “floating” potential 3…20 V. The repetition frequency and duration of high voltage pulses were 24 kHz and 5 μs, respectively. Prior to coating deposition the sample surfaces were cleaned by gas ion bombardment in a pulsed glow discharge in argon at a pressure of 4 Pa and amplitude of pulsed substrate bias of 2.5 kV for 5 minutes. Then the surface of the samples was cleaned by cathodic plasma ions bombardment at pulsed bias potential of 2.5 kV for 3 minutes at argon pressure of 0.01 Pa. Thereafter, thin interlayer of the cathode material was deposited for 3 minutes, using the same pulse potential as at the subsequent deposition of a nitride coating. Synthesis of the TiAlYN coating was performed at an arc current of 100 A in a mixture of nitrogen and argon with partial pressure of 0.13 and 0.013 Pa, respectively. Duration of deposition was of 30 minutes.

The elemental composition of the coating material was monitored by X-ray fluorescence analysis (XRA) on a vacuum scanning crystal-diffraction spectrometer SPRUT. X-ray diffraction studies were carried out on a DRON-3 device in the filtered Cu-Kα radiation. Hardness (H) and Young's modulus (E) of the coatings were measured by G200 nanoindenter using CSM (continuous stiffness measurement) mode. H value was taken at a depth of indentation of 10% of the film thickness.

The protective properties of the coatings were studied on electrochemical behavior of the system stainless steel – TiAlYN coating in a corrosive environment of 3% NaCl water solution by measuring the steady-state values of the corrosion potentials, as well as determine the electrochemical activity by recording and analizing the anodic polarization curves (at a speed of 1 mV/s from the static potential values). A standard silver chloride electrode (s.c.e.) served as the reference electrode in the electrochemical measurements.

2. EXPERIMENTAL RESULTS AND DISCUSSION

2.1. ACTIVATION POTENTIALS OF THE ANODIC PROCESS (APAP)

Measurement of corrosion potentials in a corrosive environment of 3% NaCl water solution (Table) showed that their values varied randomly over time, even for the same group of samples. During the same period of time both shift to positive potential values, and shift to negative values can be observed. This behavior is characteristic for electrochemical behavior of the materials such as stainless steel 12X18H10T (substrate material) with a surface initially covered with an oxide passivation film, which can be "ennobled" in the environment of the corrosive chloride ions and then repassivated. These phenomena appear abrupt changes of potential values from "+" to "-" and vice versa. In the case specimens under investigation these processes are affected by the character of the coating porosity (defects) and their wettability, expressed unstable corrosion potentials values and non-reproducible measurement results, even for a relatively long aging in solution (3…5 hours and more). Therefore corrosion potential values in our studies can not serve as a reliable criterion of the corrosion resistance of the samples with different coatings in the environment of 3% solution of NaCl. More information will provide anodic polarization curves. From them the activation potentials of anodic processes (APAP) were determined on the beginning of the intense anodic current growth.

From the analysis of the anodic polarization curves and the results of measuring the APAP values, which is a measure of the electrochemical activity of the samples, follows that the TiAlYN coating provides a protective effect with respect to the substrate, 12X18H10T steel. Experiments show that the protective properties of the coatings depend on their elemental composition, thickness h, and the preparation conditions, particularly deposition rate V_{dep}.

When TiAlN was doped with yttrium in an amount of 0.2…1 at.% (Fig. 1, curve 2–4 and Fig. 2, curve 3) for coatings of approximately equal thickness (h = 6 and 6.5 μm), APAP increase (i.e. decrease in their electrochemical activity) from + 0.1 to 0.58 V was recorded. The smallest electrochemical activity demonstrated TiAlYN coating with the percentage of Y equal to 0.2 at.% (see Fig. 2, curve 3). For him, APAP = 0.58 V.

For coatings of the same elemental composition but with different thicknesses due to different rates of deposition, dependence of the protective properties on both the thickness and the rate of deposition was observed. As seen from the anodic polarization curves (see Fig. 2), the protective properties of the TiAlYN coating with yttrium content of 0.2 at.%, deposited at the amplitude of the pulsed substrate bias potential of U_{pulse}= -1.5 kV, increased with increasing thickness (proportional to the deposition rate).

Corrosion potential E_{cor} and APAP of the samples TiAlYN coatings of thickness h with various Y content, deposited on the 12X18H10T steel substrates at pulse substrate bias U_{pulse} = -1.5 kV and various DC potential values.
P_{N2} = 0.13 Pa, t_{dep} = 30 min

Y cont, at. %	h, μm	V_{dep}, μm/h	U_{DC}, V	APAP, V	E_{cor}, V after τ, h				E_{cor}, V
					τ =0,5	1	2	3	Ageing, (..*) day
0	6	12	float.	0.05	-0.15	–	–	–	–
0.4	6	12	float.	0.07	-0.12	-0.17	-0.11	-0.11	-0.05 (21*)
0.2	6	12	float.	0.58	-0.02	-0.02	–	–	–
0.2	3.3	6.6	float.	0.46	-0.18	-0.03	–	-0.04	-0.06 (18*)
0.2	1.5	3.0	float.	0.3	-0.01	-0.03	0.18	-0.19	-0.11 (16*)
0.2	4.9	9.8	-150	0.2	-0.11	–	–	–	-0.15 (16*)
0.2	2.9	5.8	-150	-0.03	-0.28	–	–	-0.34	-0.06 (18*)
0.2	1.1	2.2	-150	0.18	-0.02	–	0.12	0.08	-0.06 (18*)
1.0	6.5	13	float.	0.1	–			–	-0.11 (20*)

Note: * – after keeping the samples in a solution, days.

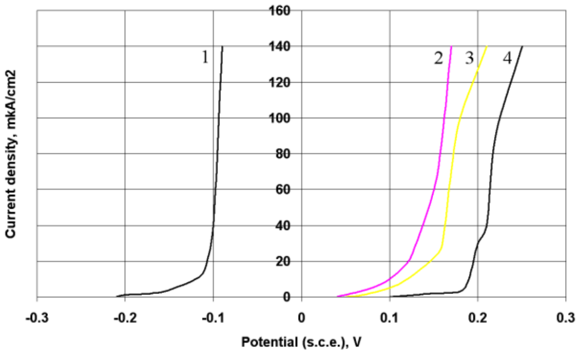

Fig. 1. Anodic polarization curves of the samples on the 12X18H10T substrates: no coating (1); coatings of thickness h = 6 µm: TiAlN (2), TiAlYN (0.4 at.% Y) (3) and TiAlYN (1 at.% Y) (4). Substrate bias during coatings deposition: U_{pulse} = -1.5 kV

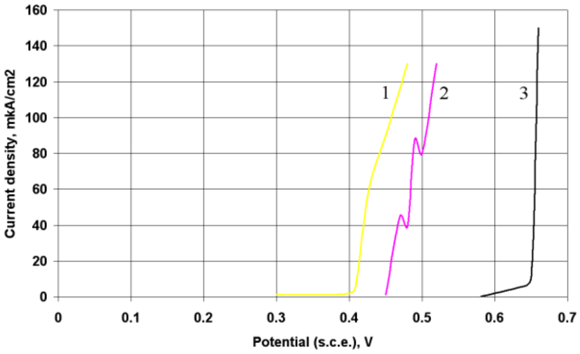

Fig. 2. Anodic polarization curves of the samples with the TiAlYN (0.2 at.% Y) coatings of various thickness h deposited on 12X18H10T substrates: h = 1.5 µm (1); h = 3.3 µm (2); h = 6 µm (3). Substrate bias during coatings deposition: U_{pulse} = -1.5 kV

The investigated electrochemical system consists of two different materials: stainless steel 12X18H10T (substrate material), originally covered with an oxide passivation film, which can be subjected to “chloride attack” and then repassivation even in the steady-state conditions at “stationary” corrosion potential, and thin protective coating layer with a low electrochemical activity. The main factors determining the electrochemical behavior of the system is porosity (defectiveness) of the coatings and wettability of their surface. Wettability is determined by the surface properties and elemental composition of the coating material, porosity (defects) – by adhesion to the substrate and the level of internal stress σ in the coating. In the case measuring the “stationary” corrosion potential the combined effect of these factors resulted in unstable, non-reproducible values of the potential.

The surface condition and residual stress level σ in the coating influence the level of adhesion to the substrate. The defects of the substrate surface are the places of pores formation in the coating. The coating will flake off when the elastic energy per unit volume at a given σ exceeds the surface fracture energy [11]:

$$\sigma^2 h/2E > 2\gamma, \quad \text{or} \quad h > 4\gamma E/\sigma^2, \qquad (1)$$

where γ is the surface fracture energy, E is modulus of elasticity of the coating.

Therefore, for $h \geq 4\gamma E/\sigma^2$ partial destruction of the coating can occurred in form the cracks or local delamination, which appears as sharp deterioration in its protective characteristics as the experiments show.

Analysis shows that the level of residual stress in the coating depends on several factors. The ion energy at the vacuum-arc deposition is of tens of electron volts, so even if the floating potential is applied to the substrate, the penetration of the ions into the subsurface layer of the coating occurs, resulting in increase of the structure defects density, and the compression stress is formed there.

The level of stress is a complex function of the substrate potential during deposition, both of its DC component and pulse one (pulse amplitude, duration and repetition frequency). Also strongly influences the temperature of the coating during deposition, increase in temperature reduces the defectiveness and causes the stress relaxation.

In multi-component nitride coatings another possible cause of the stress generation may be the change in the phase composition of the coating. Thus, in the particular case TiAlN the spinodal decomposition of the solid solution can occur with the precipitation of X-ray amorphous cubic TiN and AlN clusters, having different lattice parameters [12, 13], which may cause the stress state of the coating.

Differences in thermal linear expansion coefficients of the substrate material (stainless steel) and a nitride coating, heated to a sufficiently high temperature during coating deposition, after cooling for example to room temperature, result in generating the compressive stress of thermal origin [14].

Fig. 3 shows the anodic polarization curves of the samples with TiAlYN coating containing 0.2 at.% yttrium, deposited over the same period (30 min) with different deposition rates on substrates made of stainless steel, with thicknesses of h = 1.1; 2.9 and 4.9 µm (curves 2, 1 and 3 respectively). Substrate bias potential during coatings deposition was joint DC potential of -150 V and a pulsed one with an amplitude of -1.5 kV. We see that the lowest APAP value has the sample with TiAlYN coating of h = 2.9 µm thickness (see Fig. 3, curve 1) and the largest one – the specimens with thicknesses h = 1.1; 4.9 µm (see Fig. 3, curves 2 and 3 respectively). Thus, for a given group of samples deposited with the joint DC and pulsed potential on the substrate, the protective properties of GC not correlate with the thickness of the coating.

Comparison of anodic polarization curves in Figs. 3 and 2 shows that DC potential of -150 V additional to the pulse substrate bias of -1.5 kV in TiAlYN coatings deposition reduces their APAP value in about 3…4 times as compared with the mode of only pulsed substrate bias potential.

The most probable reason for the APAP value decrease in this case may be the increase in the level of residual stress and elevated level of the defects generated in the coating synthesized in this mode.

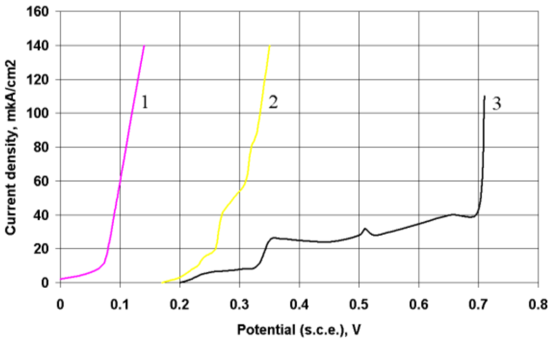

Fig. 3. Anodic polarization curves of the samples with the TiAlYN (0.2 at.% Y) coatings of various thickness h deposited on 12X18H10T substrates: h = 2.9 μm(1); h = 1.1 μm (2); h = 4.9 μm (3). Substrate bias during coatings deposition: joint U_{pulse} *= -1.5 kV and* U_{DC} *= -150 V*

In [15] it was shown that at floating value of the substrate potential the TiAlYN coatings formed with the level of residual compressive stress of 7.5 GPa. Supply to the substrate a high voltage pulsed bias potential during coating synthesis generally reduces the level of residual stress. The dependence of the stress for TiAlYN coatings on the amplitude of the voltage pulses U_{pulse} is nonmonotonic [16], when U_{pulse} increases from 0 to 1 kV the level of residual stress in the TiAlYN coatings decreases from 4.7 to 2.7 GPa. With further increase in the amplitude the residual stress level increases too and reaches the value of 6…6.5 GPa at U_{pulse} = -2.5 kV. In our experiments, the coatings were deposited with a pulse amplitude of -1.5 kV, i.e. under conditions which remote the minimum stress level formation.

Efficacy of the pulse bias potential influence on the structure of the synthesized coatings and consequently its mechanical characteristics, including residual internal stress, are determined by the ion energy and ion flux density on the substrate surface. Namely the ion current density during the pulse should play the main role in efficiency of the relaxation processes occurring in the surface layers of the deposited coating and reducing the internal stress level.

By reducing the ion current density (deposition rate) at a fixed U_{pulse} value it is possible decrease the coating surface temperature during deposition, the maximum attainable for the duration of the pulse, which leads to slowing the relaxation processes in the coating. As a result, the residual stress in a coating at low deposition rates remains fairly high. Supply a 150 V DC potential to the substrate leads to its additional heating. After cooling to room temperature, the coatings deposited at a sufficiently high temperature undergoes additional increase in the compressive stress of thermal nature due to significant difference of thermal linear expansion coefficients of the substrate material (stainless steel) and a nitride coating [14]. For large coating thickness the stress σ in it, according to (1), may lead to failure. Therefore, reducing the thickness of the coating under low stress relaxation during its deposition plays a positive role in the improvement of its protective ability (see Fig. 3, curve 2).

It was also noted that with increasing duration of exposure of the sample in the solution its electrochemical index can “improve”, i.e. the area of electrochemical passivity increase, what is characterized by greater APAP value. This is probably due to additional spontaneous passivation of both the coating material and the underlayer or the substrate itself in the pores or cracks.

CONCLUSIONS

1. TiAlYN nanostructured coatings were deposited on 12X18H10T steel substrates by PIII&D method from filtered vacuum arc plasma and their electrochemical characteristics in a 3% aqueous solution of NaCl investigated.
2. It was shown experimentally that deposition of TiA1YN coatings in the modes providing a reduction of residual stress, improves the protective properties of these coatings against galvanic corrosion (GC).
3. Experimentally shown that the best protective characteristics against GC has TiA1YN coating with Y concentration of 0.2 at.% deposited when applying during deposition pulsed substrate bias potential of negative polarity with the amplitude of -1.5 kV.
4. It was found that supply of the substrate with DC bias potential of -150 V, in addition to the pulse one with the amplitude of -1.5 kV, in the deposition of TiAlYN coatings leads to deterioration of the protective properties against GC. At that the activation potential of the anodic processes is reduced to approximately 3…4 times as compared with the case where the substrate is supplied with pulse potential only.
5. Increasing the deposition rate in the mode of supplying the substrate with pulse potential bias of -1.5 kV amplitude improves the protective properties of the TiA1YN coatings against GC due to improved adhesion and decrease in the level of residual stresses in the coatings.

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КОРРОЗИОННАЯ СТОЙКОСТЬ НАНОСТРУКТУРНЫХ TiAlYN-ПОКРЫТИЙ, СИНТЕЗИРОВАННЫХ PIII&D-МЕТОДОМ ИЗ ФИЛЬТРОВАННОЙ ВАКУУМНО-ДУГОВОЙ КАТОДНОЙ ПЛАЗМЫ

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Исследованы электрохимические характеристики наноструктурных TiAlYN-покрытий на стали 12Х18Н10Т в 3% водном растворе NaCl. Покрытия осаждались из фильтрованной вакуумно-дуговой плазмы методом PIII&D. Измерения потенциалов коррозии $E_{кор}$, а также анодные поляризационные кривые показали, что наилучшую защиту от электрохимической коррозии (ЭХК) обеспечивают TiA1YN-покрытия с содержанием Y 0,2 ат.%, осажденные при подаче на подложку импульсного потенциала смещения амплитудой -1,5 кВ. Подача дополнительно постоянного потенциала смещения -150 В приводит к ухудшению защитных свойств: потенциал активации анодных процессов (ПААП) уменьшается в 3–4 раза. Повышение скорости осаждения способствует улучшению защитных свойств TiA1YN-покрытий, что обусловлено улучшением адгезии и снижением уровня остаточных напряжений.

КОРОЗІЙНА СТІЙКІСТЬ НАНОСТРУКТУРНИХ TiAlYN-ПОКРИТТІВ, СИНТЕЗОВАНИХ PIII&D-МЕТОДОМ З ФІЛЬТРОВАНОЇ ВАКУУМНО-ДУГОВОЇ КАТОДНОЇ ПЛАЗМИ

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Досліджено електрохімічні характеристики наноструктурних TiAlYN-покриттів на сталі 12Х18Н10Т в 3% водному розчині NaCl. Покриття осаджувались з фільтрованої вакуумно-дугової плазми методом PIII&D. Вимірювання потенциалів корозії $E_{кор}$, а також анодні поляризаційні криві показали, що найкращий захист від електрохімічної корозії (ЕХК) забезпечують TiA1YN-покриття з вмістом Y 0,2 ат.%, осаджені при подачі на підкладку імпульсного потенціала зміщення амплітудою -1,5 кВ. Подача додатково постійного потенціала зміщення -150 В призводить до погіршення захисних властивостей: потенціал активації анодних процесів (ПААП) зменшується в 3–4 рази. Підвищення швидкості осадження сприяє поліпшенню захисних властивостей TiA1YN-покриттів, що зумовлено поліпшенням адгезії та зниженням рівня залишкових напружень.