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PHYSICO-MECHANICAL PROPERTIES OF ARC-PVD NANOSTRUCTURED Ta-Ti-N COATINGS

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Ta-Ti-N coatings obtained by vacuum-arc deposition have been investigated. The structure and chemical composition of the coatings were investigated by scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS). The microhardness of the vacuum-arc Ta-Ti-N coating H_i = 24.12 GPa and the elastic modulus E = 283.47 GPa were determined. The H^3/E^2 index is 0.175, which indicates a high resistance of the coating to plastic deformation. The main increase in hardness is due to the TiN phase. The protective wear-resistant Ta-Ti-N coating due to high physical-mechanical properties is promising for deposition on various surfaces used in industry and for elements of gas turbine engines.

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INTRODUCTION

The technology of vacuum-arc coating has become widespread in various branches of mechanical engineering, mainly to increase the durability of products (including erosion and corrosion resistance) [1, 2]. Compared to traditional coatings on gas turbine engines elements (GTE), the effectiveness of a nanostructured or nanolaminate coating in the true sense will be significantly improved in terms of strength, toughness, wear resistance and thermal barrier.

Highly effective nanostructured protective coatings can have two-dimensional or three-dimensional architectures, and they can be formed from nanometer-thick bilayers, columnar nanostructures, nanograins surrounded by a fabric phase (nanocomposites), and mixtures of nanograins with different crystallographic orientations [3, 4]. Compounds that exhibit a certain affinity for each other tend to provide high thermal stability and durability in service, and they are often super strong and have unusual mechanical and physical properties [5–10]. The compounds involved are highly chemically active, and the system exhibits a thermodynamic driving force for phase segregation. Such coatings will contribute to the modernization of traditional industries with their improved performance and durability, and will eventually replace traditional technologies and bring good economic benefits.

In the present research Ta-Ti-N coatings obtained by vacuum-arc deposition have been investigated. The structure and physical-mechanical characteristics have been studied.

1. EXPERIMENTAL SETUP

Vacuum-arc Ta-Ti-N coatings were obtained in a modified installation of the "BULAT-6" type, the schematic diagram of which is shown in Fig. 1. The samples of steel X18H10T measuring 20x20 mm and 3 mm thick have been used. The samples were located on a metal substrate holder located at a distance of 500 mm from the evaporators. Steel samples and cutting inserts before loading into the vacuum chamber were washed with an alkaline solution in an ultrasonic bath and degreased with Nefras C2-80/120.

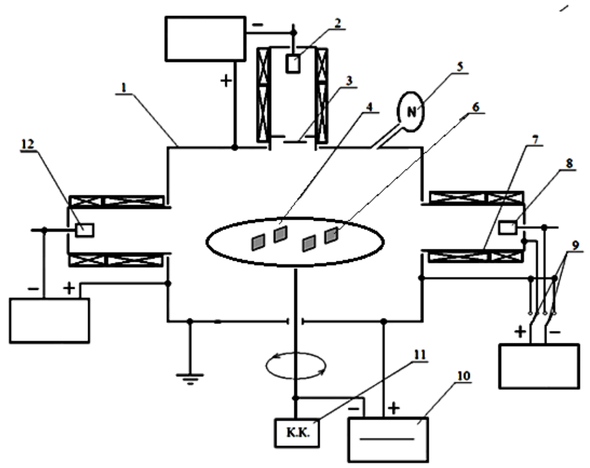


Fig. 1. Schematic diagram of a vacuum-arc installation of the "BULAT-6" type for applying nitride coatings with preliminary cleaning in a gas discharge: 1 – vacuum chamber; 2 – vacuum-arc evaporator; 3 – metal screen that does not allow ions of the evaporated metal to pass through; 4 – sample substrate holder; 5 – nitrogen inlet; 6 – samples; 7 – vacuum-arc evaporator housing – anode for gas discharge; 8 – cathode of the first material; 9 – switching relay; 10 – constant voltage source; 11 – command controller; 12 – cathode of the second material

After pumping the vacuum chamber to a pressure of $P = 0.3 \cdot 10^{-3}$ Pa, ion cleaning and activation of the surface of the samples were performed in a gas vacuum arc discharge, which allows cleaning with accelerated ions of a gas plasma (without metal ions) in order to obtain good adhesion.

Gas vacuum-arc discharge is a discharge in which the positive arc column is divided into two stages, the first of which is a vacuum arc with a cold cathode (2), and the second stage – the positive arc column is located behind a metal screen (3), which does not allow ions of the evaporated metal to enter the chamber with the samples. The quality of cleaning and activation of the surface of the samples before the deposition of vacuum-arc nitride coatings depends on the ion current on the sample, which is associated with the density of the gas plasma of the vacuum-arc discharge, and the heating temperature of the samples depends on the applied potential at the time of cleaning. The cleaning was performed at a cathode current of I_k = 80 A (cathode 2), an anode current of I_A = 100 A (anode 7). The voltage U_s on the samples varied from -100 to -1100 V (substrate holder 4) using a power source (10). Nitrogen pressure during cleaning was P_N = 0.6 Pa, the sample temperature was 550…590 ºC, and the duration was 10 min.

After cleaning and heating, layers of pure metals Ta and Ti were deposited for 1 min with constant rotation of the samples. The deposition of vacuum-arc Ta-Ti-N coatings was then performed for 60 min under the following conditions: I_{Ti} = 85 A, I_{Ta} = 170 A, U_s = –200 V, and P_N = 0.4 Pa.

X-ray structural studies were performed on a DRON-4M diffractometer with digital data acquisition and Cu-Kα radiation. The “Match!4.2” program was used to decipher and calculate the diffractograms.

Instrumental indentation of the coatings was performed on a Nanoscan-4D device with a Berkovich pyramid at a load of 500 mN and a holding time of 15 s. The average values of the indicators were determined from at least 15 measurements.

Structure and chemical composition of the coatings were investigated by scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS) by using ZEISS EVO® MA10.

2. RESULTS AND DISCUSSION

The morphology of the surface of Ta-Ti-N coatings at different magnifications is shown in Fig. 2. The presence of a droplet phase was detected, which occurs during the deposition of coatings by the vacuum-arc method. It was found that the surface of the coatings has a cellular structure. The droplet phase is evenly distributed over the surface of the samples. Most of the droplet phase has dimensions less than 1 μm, however, there are drops of molten metal up to 7 μm in size.

Fig. 3 shows typical EDX spectra from the surface of the Ta-Ti-N coating, and Fig. 4 shows EDS mapping of selected chemical elements from the surface of one of the coated samples.

The elemental composition of the vacuum-arc coating was determined, in particular: the titanium content was 31.93 at.%, tantalum – 32.26 at.%, nitrogen – 35.81 at.%. The average error in estimating the elemental composition of the coating is ~ 4 at.%. Analysis of cartographic images of the elemental composition of vacuum-arc Ta-Ti-N coatings showed a relatively uniform arrangement of elements on the surface of the coating, even in the droplet phase.

The elemental composition of the surface of the droplet phase was studied (Fig. 5). It was determined that the surface of the droplet phase is depleted in nitrogen, which may be due to the conditions of coating deposition. The droplet phase almost does not form nitrides during the deposition process, the nitride phase is present only on the droplet surface.

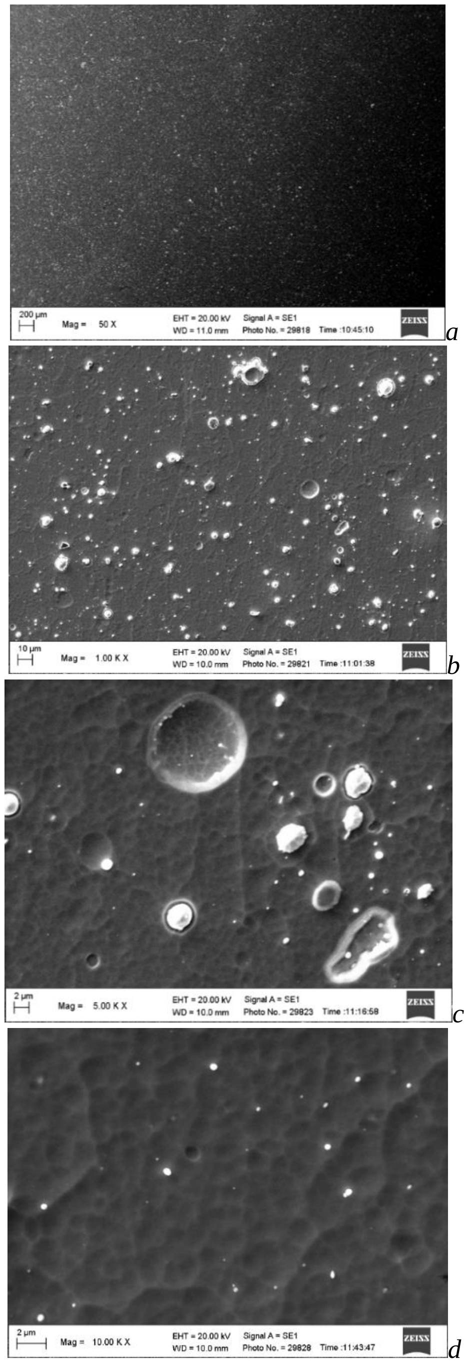


Fig. 2. Surface morphology of vacuum-arc Ta-Ti-N at different magnifications

When calculating the phase composition of the vacuum-arc Ta-Ti-N coating, the values of the 2Θ angle were shifted internally to calculate the quantities, intensity scaling factors, and the figure of merit (FoM).

The formation of four phases in the Ta-Ti-N coating was established. The TiN phase with a cubic Fm-3m lattice and a unit cell size a = 4.2200 Å is predominantly formed.

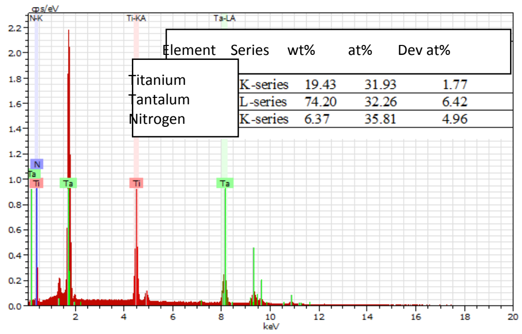


Fig. 3. EDX spectra from the surface of the Ta-Ti-N coating

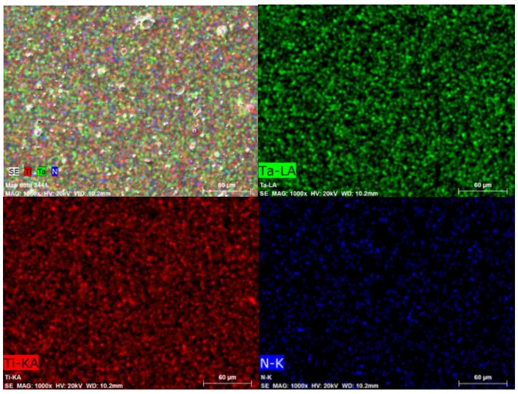
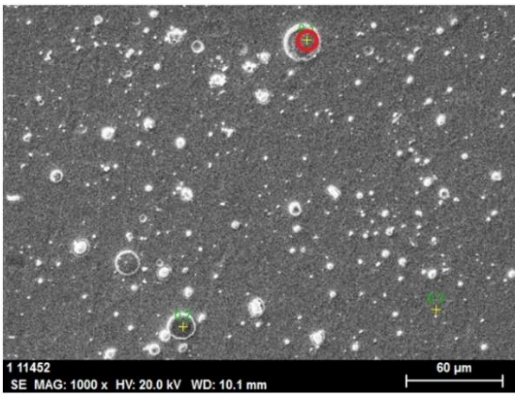
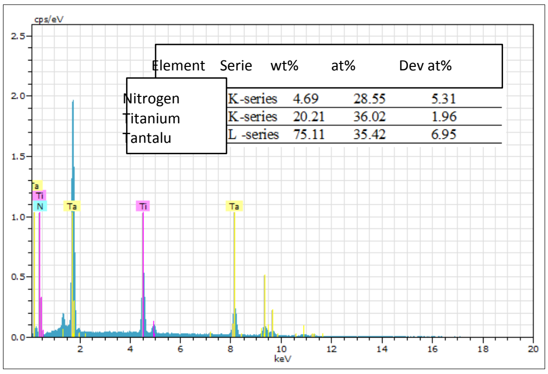


Fig. 4. EDS mapping of selected chemical elements from the surface of the Ta-Ti-N coating



a



b

Fig. 5. SEM-surface image (a) and EDX spectra (b) of vacuum-arc Ta-Ti-N coating with droplet phase

However, when forming the TaN coating, three phases are formed: cubic TaN (Fm-3m) with a lattice parameter a = 4.3580 Å, hexagonal TaN (P-6m2) with lattice parameters a = 2.9380 Å and c = 2.8830 Å, hexagonal Ta_5N_6 (P63/mcm) with lattice parameters a = 5.1760 Å and c = 10.3530 Å.

Fig. 6 shows the X-ray diffraction spectrum of vacuum-arc Ta-Ti-N coating. The diffraction peaks have a significant widening, which indicates a high level of dispersion of the crystal structure or residual stresses. Using the Silyakov-Scherrer formula, the size of the crystallites of the phases was calculated. For cubic TaN, the size of the crystallites is 14.6 nm, for hexagonal TaN – 12.8 nm, for Ta_5N_6 – 6.5…14.6 nm.

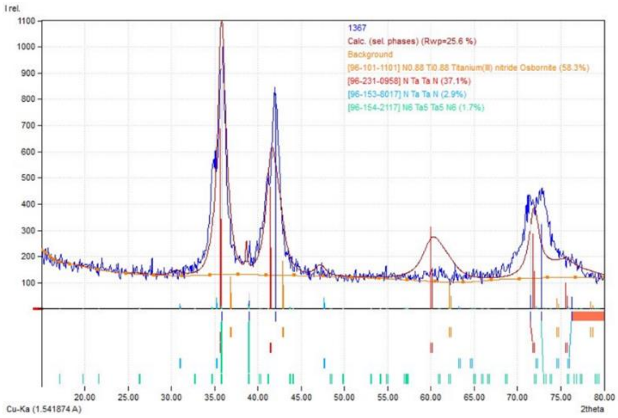


Fig. 6. XRD spectrum of vacuum-arc Ta-Ti-N coating

The presented nitride spectrum and the similarity of the crystallite sizes indicate an evolutionary path of coating formation, the individual phases of which are transformed from each other, which is consistent with known ideas about the phase composition of equilibrium systems [4]. With an increase in the amount of nitrogen in the system, first nitrides Ta_2N_x are formed, which under non-equilibrium conditions are transformed into Ta_5N_6, then into hexagonal TaN with a rearrangement into a more equilibrium cubic TaN. Ta_5N_6 probably remains in a small amount at the separation boundaries where local supersaturation with nitrogen is preserved.

The microhardness of the vacuum-arc Ta-Ti-N coating H_i = 24.12 GPa and the elastic modulus E = 283.47 GPa were determined. The H^3/E^2 index is 0.175, which indicates a high resistance of the coating to plastic deformation.

CONCLUSIONS

The vacuum-arc Ta-Ti-N coating with a hardness H_i = 24.12 GPa, an elastic modulus E = 283.47 GPa, and a ratio of H^3/E^2 = 0.175 has been developed, which has high resistance to plastic deformation due to a reduction in the average crystallite size to 7…14 nm.

The elemental composition of the vacuum-arc Ta-Ti-N coating has been determined, in particular: titanium – 31.93 at.%, tantalum – 32.26 at.%, nitrogen – 35.81 at.%.

The main increase in hardness is due to the TiN phase. In turn, tantalum increases the thermal stability of the system, reducing the diffusion mobility of atoms in the coating, and has high chemical resistance and high resistance to oxidation.

Therefore, the protective wear-resistant Ta-Ti-N coating with high hardness is a promising solution for deposition on various surfaces used in industry and for gas turbine engine elements as well.

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ФІЗИКО-МЕХАНІЧНІ ВЛАСТИВОСТІ ВАКУУМНО-ДУГОВИХ НАНОСТРУКТУРОВАНИХ ПОКРИТТІВ Ta-Ti-N

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Досліджено покриття Ta-Ti-N, отримані методом вакуумно-дугового осадження. Структуру та хімічний склад покриттів досліджували за допомогою скануючої електронної мікроскопії (СЕМ) та енергодисперсійної спектроскопії (ЕДС). Визначено мікротвердість вакуумно-дугового покриття Ta-Ti-N H_i = 24,12 ГПа та модуль пружності E = 283,47 ГПа. Індекс H^3/E^2 становить 0,175, що свідчить про високу стійкість покриття до пластичної деформації. Основне збільшення твердості зумовлене фазою TiN. Захисне зносостійке покриття Ta-Ti-N завдяки високим фізико-механічним властивостям є перспективним для нанесення на різні поверхні, що використовуються в промисловості, та для елементів газотурбінних двигунів.