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Helicon-Arc Ion-Plasma Synthesis of AlN-Based Film Coatings on the Steel 3 and Aluminium Substrates

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Using a hybrid helicon-arc ion-plasma reactor, a coating based on aluminium nitride (AlN) is deposited on the steel 3 and aluminium substrates. Regardless of the substrate, it is found that a coating with a thickness of ≅ 2.0 μm is formed during 30 minutes of deposition; increasing the process time to 45 minutes allows to obtain a coating two and a half times thicker (≅ 5 μm). As determined, the selected deposition time does not allow obtaining a coating of stoichiometric composition of AlN on the substrates used. As a rule, a compound with Al excess is formed. As shown, in cases where a plastic material (for example, aluminium) is used as a substrate, the formation of the coating can only be the last technological operation.

Key words: film coatings, microstructure, stoichiometric composition, aluminium, aluminium nitride.

За допомогою гібридного геліконно-дугового йонно-плазмового реактора було сформовано плівкові покриття на основі нітриду Алюмінію (AlN) на підкладинці із криці 3 й алюмінію. Встановлено, що, незалежно від використаної підкладинки, за 30 хвилин технологічного процесу було сформовано покриття товщиною у ≅ 2,0 мкм; збільшення часу процесу до 45 хвилин уможливило одержати покриття в два з половиною рази більше

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(≅ 5 мкм). З'ясовано, що обраний час технологічного процесу не дав змоги одержати покриття стехіометричного складу AlN на використаних у роботі підкладинках. Як правило, утворювалася сполука з надлишком металевої складової. Показано, що у тих випадках, коли у якості підкладинки використовується «м'який» пластичний матеріял (наприклад алюміній), формування покриття може бути тільки останньою технологічною операцією.

Ключові слова: плівкові покриття, мікроструктура, формульний склад, алюміній, нітрид Алюмінію.

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1. INTRODUCTION

To date, there are three main methods of protecting metallic materials from the negative effects of external factors [1]. The first one is changing the composition of the material surface by high-energy methods [2, 3]. The second one is the influence on the phase and/or structural state of the material, due to additional alloying [4, 5]. Moreover, the third one is the deposition of a protective coating on the surface of the material, which differs in chemical composition from the base [6]. Although all these techniques have already found their application in industry, the method of forming protective coatings is considered the most effective [7].

Protective coatings are already widely used in industry, and their use gives a huge economic effect [6, 8]. The methods of their production are very diverse: electrolytic deposition of metals and alloys, vacuum evaporation with subsequent condensation on the cathode substrate, the method of gas transport reactions, ion implantation, chemical-thermal treatment, *etc.* [1]. The compositions of protective coatings are also extremely diverse: oxides, borides, nitrides, carbides, silicides and so on [9, 10].

Among the existing variety of protective layers, coatings based on aluminium nitride (AlN) occupy a special place due to their physical [11–13] and optical properties [14, 15]. As noted in Refs. [16, 17], aluminium nitride is a wide-band-gap semiconductor with a wide band gap (of 6–6.2 eV) and sufficient dielectric strength (high relative permittivity of 8–9). AlN also has a rather high hardness and a coefficient of thermal expansion close to silicon.

The above properties and one of the highest thermal conductivity coefficients (for high-quality AlN layers, the thermal conductivity coefficient reaches $120\ \mathrm{W\cdot m^{-1}\cdot K^{-1}}$ at room temperature) allow AlN to be used to remove excess heat from powerful electronic devices. AlN can also be an effective thermal interface material (TIM) [12, 18–21].

AlN coatings on flexible polymer substrates are promising for the manufacture of multiband blocking filters in the infrared spectral range [13, 22].

In addition, due to its piezoelectric properties, AlN is used in microwave acoustic resonators [23, 24]. AlN has been widely used in packaging electronic devices and circuits, including those of RF and microwave electronics [25]. Moving up to mm-wave, the relatively high dielectric constant (k) of AlN compared to that of quartz, glasses or polymers can be turned into an advantage in reducing the interconnect size in microwave monolithically integrated circuits (MMICs), which are necessary for high-density phased arrays.

In addition, aluminium nitride has proven itself well as a protective barrier layer to reduce hydrogen permeability (for example, on titanium), especially, when operating under low-pressure conditions [5, 26]. The above properties make it an ideal candidate for practical application in many industries, so, further research into the synthesis of coatings based on AlN is needed.

The aim of the present study was to determine the features of the formation of aluminium nitride-based coatings on substrates with similar or different chemical compositions, which were deposited in a hybrid helicon-arc ion-plasma reactor with the aluminium-based consumable cathode.

2. EXPERIMENTAL/THEORETICAL DETAILS

Aluminium and steel 3 were used as substrates. Rectangular 25×15×2 mm samples were used. Aluminium and AMG-6 alloy were selected as consumable cathode materials. AMG-6 alloy was selected based on previous studies [27], according to which the addition of a certain amount of magnesium to aluminium expanded the range of the residual radiation band (Reststrahlen band).

For the formation of AlN-based coatings, unique vacuum-technological equipment based on a hybrid helicon-arc ion-plasma reactor [28–31] was used that has been developed at the G. V. Kurdyumov Institute for Metal Physics, N.A.S. of Ukraine. It contains high-frequency helicon and magnetically activated plasma-arc plasma sources, which allows low-temperature ion-plasma synthesis of coatings. This equipment provides unique technological characteristics, in particular, low temperatures of the substrate (30–300°C) and the formation of a flow of ions of the working gas of the helicon source with a plasma density in the substrate region of 5–10 mA/cm^2, and energies up to 100–150 eV. A magnetically activated vacuum-arc source (plasma-arc accelerator) provides the generation of an accelerated plasma flow of consumable cathode materials with a density of up to 10–20 mA/cm^2, and ion energies of up to 100–150 eV.

The technology of depositing a layer based on aluminium nitride on rectangular samples in a hybrid helicon-arc ion-plasma reactor also provided preliminary plasma cleaning of the sample surface. First of all, the working volume was evacuated to a pressure of $\cong 1\cdot 10^{-7}$ MPa. Further, in an argon atmosphere, the surface of the working chamber and samples was cleaned of contaminants, and the surface of the samples was activated to provide the adhesion of the coatings when supplying HF power of 450 W. The cleaning lasted about 15 minutes, after which the argon was pumped out and nitrogen was introduced into the chamber to a pressure of $2.7\cdot 10^{-6}$ MPa. After that, using a plasma arc accelerator, the ion plasma component of the cathode material was transferred to the surface of the substrate at an arc current of 40 A. To achieve uniformity of coating, the substrate was rotated in a horizontal plane during deposition. The time of the technological process of 30 and 45 minutes was chosen to determine the correlation between the deposition time and the thickness of the coating. After the deposition was completed, the sample was cooled in the chamber in a nitrogen atmosphere.

Metallographic studies were performed by scanning electron microscopy at a VEGA3 TESCAN microscope equipped with an EDX XFlash610M detector (Bruker).

The coated samples were cut in half by the electrical discharge technique. At the same time, it was revealed that, in order to use this technique, the samples required mechanical indentation (it was necessary for the wire to have contact with the base). The samples were polished with Cr_2O_3 suspension using Struers LaboPol-21 and LECO Spectrum System 1000 units.

3. RESULTS AND DISCUSSION

Using scanning electron microscopy, the peculiarities of the formation of a nitride coating based on the AMG-6 alloy on aluminium were determined, depending on the time of the technological process (Fig. 1). From the presented microstructure (Fig. 1, *a*), it can be seen that thirty minutes of the process allowed forming a coating with a thickness of $\cong$ 2.0 µm. Increasing the time of the technological process to 45 minutes (by 1/2) made it possible to deposit a coating two and a half times thicker up to 5 µm (Fig. 1, *b*).

This indicates, however, that the coating did not grow linearly with the time of deposition. In the first 30 minutes, the average rate of coating growth was $\cong$0.06 µm/min, and in the period of 30–45 minutes, it was already $\cong$0.2 µm/min. It can be assumed that, during the formation of the coating, there was a certain period of time (most likely, the very beginning of synthesis), during which its thickness did not increase.

In addition, there is a transition zone (dark layer) between the coat-

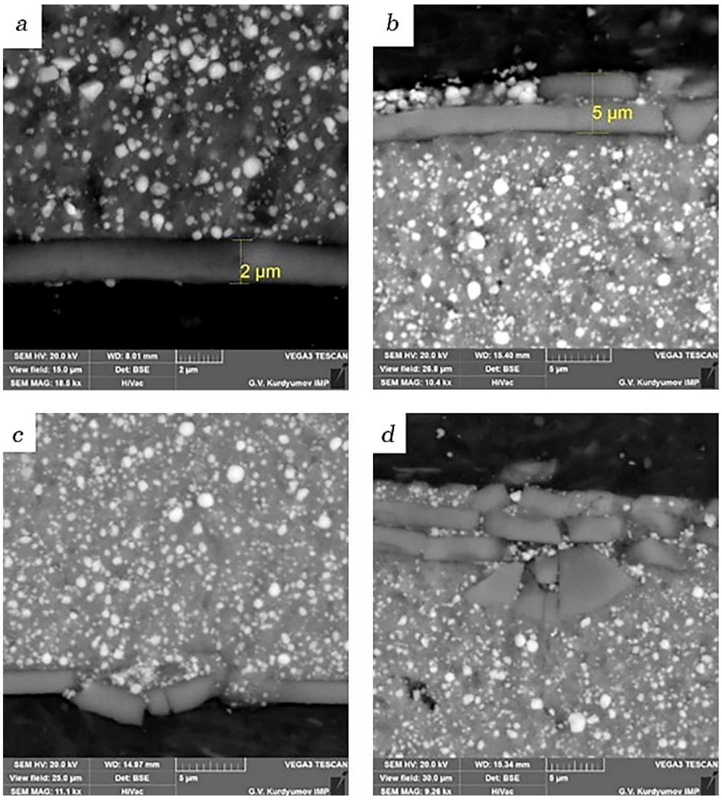

Fig. 1. Microstructure of an aluminium sample with a nitride layer based on the AMG-6 alloy deposited for: *a*, *c*—30 minutes; *b*, *d*—45 minutes.

ing and the substrate (Fig. 1). This, to a certain extent, allows us to state that at the initial stage of coating growth, there was some interaction between the substrate and the layer being formed. The obtained result well correlates with the data presented in Ref. [19]. According to these data, the size of the transition layer can reach $\cong 11.5\%$, when an aluminium nitride coating with a thickness of $\cong 2.0$ μm is deposited.

It was also found that during mechanical processing of the samples (cutting, grinding and polishing), there was a certain failure of the formed coating (Fig. 1, *c*, *d*). Moreover, as can be seen in the presented microstructures (Fig. 1, *c*, *d*), an increase in the thickness of the coating led to its more significant failure. It can be assumed that this phenomenon is associated with a significant difference in hardness between the substrate (the hardness of aluminium by the Mohs hardness scale is of 2.75) [32] and the coating.

As can be seen in the presented microstructure (Fig. 1, *c*), during mechanical processing of the sample, plastic deformation occurred in the substrate; as a result, cracking and failure of the coating occurred. However, delamination of the deposited layer from the substrate was not observed. This indicates a fairly high adhesion between the coating and the substrate. It should be noted that, before making the sections, the samples were cast in a bakelite mould to prevent their deformation (since rather thin samples were used in the study) and changes in the angle of inclination during mechanical processing.

In addition, this mould also performed the function of a holder, which made it possible to preserve the coating to a certain extent. It can be assumed that mechanical processing of such a sample, without a holder, will lead to a fairly rapid fracture of the coating. That is, in cases, where the coating is deposited to aluminium, such samples cannot be subjected to mechanical processing.

To clarify the above, a nitride coating based on AMG-6 alloy was deposited on steel 3 (Fig. 2) with the same parameters used for the aluminium substrate for their correct comparison.

The technological process of 30 minutes allowed depositing a coating with a thickness of $\cong 2$ µm (Fig. 2, *a*). Increasing the time to 45 minutes allowed obtaining a coating with a thickness of $\cong 5$ µm (Fig. 2, *b*), which completely coincides with the data obtained, when forming a coating on aluminium. Determination of certain regularity between the technological process time and the thickness of the formed layer will allow in the future depositing a coating of the required thickness, focusing on the application time. In addition, it can be stated that, when forming a coating, its thickness is mainly influenced by deposition time, and is practically not affected by the substrate used. In addition, it was established from the microstructures presented in Fig. 2 that, during mechanical processing of the samples (which was carried out under identical conditions with Al substrate samples), no failure of the coat-

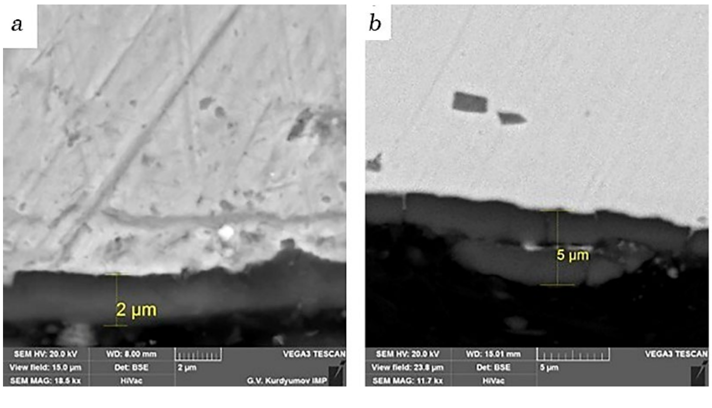

Fig. 2. Microstructure of a steel 3 sample with a nitride layer based on the AMG-6 alloy deposited for: *a*—30 minutes; *b*—45 minutes.

ing was observed. One of the reasons for this phenomenon is most likely a smaller difference in hardness between the coating and the substrate. In these samples, at the boundary of the coating-base distribution, a certain layer was also observed, which differed from them in colour. It can be considered an intermediate layer or substrate. Most likely, this layer also in some way reduces the difference in mechanical properties at the substrate–coating boundary. Unfortunately, to date, it has not been possible to establish with certainty the product, of which reaction the layer formed between the surface of the substrate (which was cleaned before the formation of the coating) and nitrogen in the chamber or the base and the AMG-6 alloy. The result obtained, regarding the absence of failure of the protective layer during mechanical processing, is an excellent confirmation that in the case of using ductile substrates (for example, aluminium), the formation of the coating can only be the last operation.

In addition to all above mentioned, it was important to determine whether the exposure time and/or the substrate affected the chemical composition of the formed coating. Using scanning electron microscopy (Figs. 1 and 2) and EDX analysis, the chemical compositions of the obtained coatings were determined (Table 1).

From the presented data, it was established (Table 1) that the chemical composition of the coatings is primarily influenced by the exposure time. In addition, regardless of the time of the technological process and the substrate used, it has not yet been possible to obtain a phase of stoichiometric composition in the coating. In 30 minutes, a phase was formed with the stoichiometric composition $(Al_{0.94}Mg_{0.06})(N_{0.73}O_{0.27})_{1.15}$ on an aluminium substrate and $(Al_{0.92}Mg_{0.08})_{1.83}(N_{0.58}O_{0.42})_{3.17}$ on steel 3. From the obtained data, it can be stated that, during the formation of the coating, within 30 minutes on an aluminium base, a phase based on the AlN compound with some excess of the non-metallic component was formed, while on steel 3, over the same period of time, a phase based on the Al_2O_3 compound was formed also with some excess of the non-metallic component. Because of such a significant difference in the chemical compositions of the formed coatings obtained over the

TABLE 1. Chemical compositions of the formed coatings.

Basis	Process time, min.	Elements, ±0.03 at.%			
		Al	Mg	N	O
Aluminium	30	43.54	2.93	39.06	14.47
	45	51.80	3.92	31.30	12.98
Steel	30	33.67	2.87	36.92	26.54
	45	50.87	4.21	32.22	12.70

same period of time, but on different substrates, they will have different physical and mechanical properties. One of the reasons for such a difference in the chemical composition of the obtained layers may be that, in the case of using an aluminium substrate, it also makes a certain contribution to the formation of the coating. Although in reality, there can be quite a lot of reasons for such a discrepancy in the chemical composition of the formed coating: the purity of the atmosphere used; the degree of contamination of the cathode used and its wear; the total time of contact of the sample with the atmosphere used, *etc*.

Increasing the process time to 45 minutes led to a significant change in the chemical composition of the resulting coatings. When using steel 3 as a substrate, the type of compound formed also changed. The stoichiometric composition $(Al_{0.93}Mg_{0.07})(N_{0.71}O_{0.29})_{0.8}$ was obtained on an aluminium base and $(Al_{0.92}Mg_{0.08})(N_{0.72}O_{0.28})_{0.82}$ on steel 3 base. The obtained almost identical composition of the applied layer indicates that the substrate no longer affected the formation of the coating, and the formation process was stabilized.

Regardless of the substrate used, a phase based on the AlN compound with an excess of the metal component was formed. Most likely, there might be several reasons for such a rather significant difference in the chemical composition of the coatings obtained only in an additional 15 minutes of the technological process, namely, a decrease in the volume fraction of nitrogen in the atmosphere used, as well as an increase in the flow of the metal component.

In order to confirm or refute the established patterns, aluminium and steel 3 were coated with aluminium nitride using a consumable aluminium cathode. Figure 3 shows the AlN coating formed on aluminium depending on the time of the technological process. It was established (Fig. 3) that, regardless of the time of the technological process, plastic deformation of the substrate occurred during mechanical processing of the sample and, accordingly, the coating fractured. That is why, unfortunately, it was not possible to determine accurately the thickness of the formed layer. However, the obtained result fully confirms the above conclusions that it is undesirable to carry out mechanical processing of the sample with the coating. In addition, the failure of the coating occurred in the places of plastic deformation of the substrate (Fig. 3, *a*), and not by delamination from it. The obtained result confirms the above data on relatively high adhesion between the substrate and the coating.

Figure 4 shows the microstructures of samples with AlN coatings on steel 3. During mechanical processing of these samples, regardless of the thickness of the formed layer, no failure of the protective coating was observed. This somewhat confirms the assumptions made that, in cases of a small difference in hardness between the coating and the substrate, samples with a protective layer applied can be subjected to

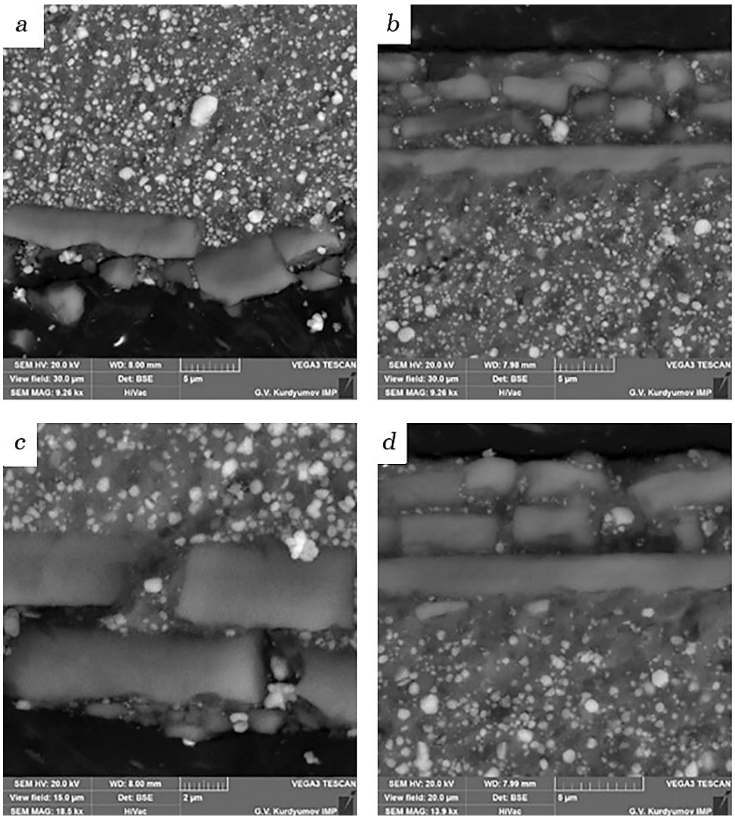

Fig. 3. Microstructure of an aluminium sample with an AlN layer deposited for: *a*, *c*—30 minutes; *b*, *d*—45 minutes.

mechanical processing. From the presented microstructures (Fig. 4), it can be seen that, in thirty minutes, it was possible to form a coating with a thickness of ≅ 2.168 μm; increasing the time of the technological process to 45 minutes allowed obtaining a coating thickness of ≅ 6 μm. The obtained data confirm the above-mentioned regularity, regarding the fact that, in the first 30 minutes, the average rate of coating formation is relatively low, and, in the interval 30–45 minutes, it increases almost by an order of magnitude. In addition, in the presented microstructures (Fig. 4, *b*), a transition zone is visible between the coating and the substrate (this zone was also observed, when forming a nitride coating on the AMG-6 alloy).

It was also important to confirm the data obtained above that the exposure time and/or the substrate affects the chemical composition of the formed coating (Table 2).

As shown above, the chemical composition of the coating was significantly influenced by the atmosphere, in which it was applied. The sub-

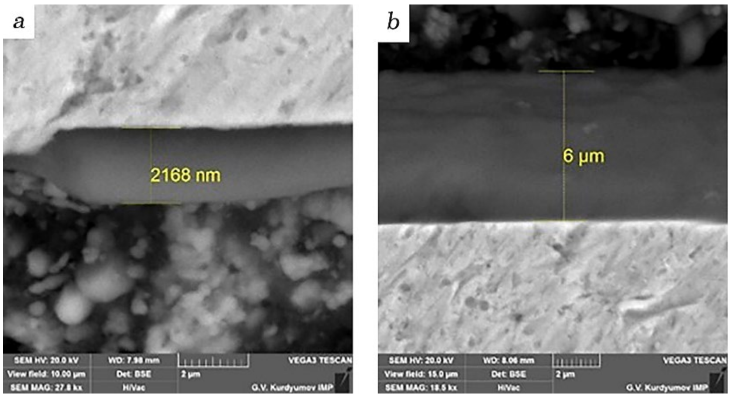

Fig. 4. Microstructure of a sample of steel 3 with an AlN layer deposited for: *a*—30 minutes; *b*—45 minutes.

TABLE 2. Chemical composition of the formed coatings.

Basis	Process time, min.	Elements, ±0.03 at.%			
		Al	Mg	N	Al
Aluminium	30	61.64	–	32.82	5.54
	45	60.93	–	32.69	6.38
Steel	30	39.29	–	38.59	22.12
	45	56.73	–	36.29	6.98

strate, on which the coating was formed, also has an effect. In addition, the regularity was confirmed that, regardless of the time of the technological process and the substrate used, it was not possible to form a stoichiometric phase in the coating. In 30 minutes, a layer was formed with the stoichiometric composition $Al(N_{0.86}O_{0.14})_{0.62}$ on an aluminium substrate and $Al_{1.96}(N_{0.64}O_{0.36})_{3.04}$ on steel 3 substrate. The obtained data coincide with the above: when the coating was applied for 30 minutes, a phase based on the AlN compound was formed on the aluminium substrate, while, on steel 3, a phase based on the compound Al_2O_3 was formed. It turned out to be interesting that increasing the technological process time on an aluminium substrate to 45 minutes had practically no effect on the change in the chemical composition of the coating, which had the stoichiometric composition $Al(N_{0.84}O_{0.16})_{0.64}$.

In the case of using steel 3 as a substrate, increasing the technological process time, on the contrary, led to significant changes in the chemical composition of the coating. At the same time, it was possible to obtain a layer with the stoichiometric composition $Al(N_{0.84}O_{0.16})_{0.76}$, which already corresponds to the phase based on the AlN compound, with some excess of the metal component. This is confirmation that the chemical composition of the coating is influenced by various factors throughout the technological process. In addition, from the presented data on the chemical composition of the coating applied to steel 3, it can be assumed that the coating formed in 45 minutes may not be homogeneous.

From the presented data (Table 1 and Table 2), it was found that, regardless of the substrate and cathode material used, the obtained coatings contained a rather large percentage of oxygen (up to 26.5 at.%). To date, it has not been possible to establish accurately the nature of this oxygen. It can only be assumed that part of the oxygen in the coating gets from the cathode material, and the other from the used atmosphere. A certain confirmation of this assumption may be the decrease in the proportion of oxygen in the coating with an increase in the time of the technological process (Table 1 and Table 2). Depending on the operating conditions of these coatings, the presence of oxygen in them can have both a positive and a negative effect.

According to data [33–36], with an increase in the oxygen content in the AlN compound, the magnitude and activation energy of high-temperature electrical conductivity increase, and, accordingly, the dielectric properties decrease. The authors of Ref. [36] note that, with the dominance of oxygen in the AlN compound, two main thermally activated contributions to electromechanical losses were observed, namely, inelastic relaxation of point defects at temperatures of 400–800 K, and electrical conductivity at $T > 800$ K.

Comparing the obtained data on the chemical composition of coatings, when using a cathode of AMG-6 alloy and pure aluminium, the following facts can be noted. First, in cases, where the substrate has a chemical composition that is identical or close to the cathode used, in some way, it participates in the formation of the coating. Second, in the case of a completely different chemical composition between the substrate and the cathode used, an increase in the process time from 30 to 45 minutes leads to a significant change in the type of coating formed.

4. CONCLUSIONS

1. It was shown that, when using ductile substrates, such as aluminium, for the deposition of aluminium nitride, to prevent the integrity of the formed layer from being compromised, the coating formation operation can only be the last one.
2. The chemical composition of the coating was primarily determined by the gaseous environment, in which it was formed. In those cases, when the chemical compositions of the substrate and the used consumable cathode were similar, the simultaneous action of the substrate and

the gas environment on the resulting coating could occur.

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