# Evolution of structure and phase composition of vacuum plasma coatings alloyed by aluminium

# Evolution of structure and phase composition of vacuum plasma coatings alloyed by aluminium

**S D Latushkina[1], O I Posylkina[1], D V Kuis[2], A V Kasperovich[2], O Yu Piskunova[2]**

[1] The Physical-Technical Institute of National Academy Sciences of Belarus, 10 Kuprevich St., 220141,Minsk, Belarus
[2] Belarusian State Technological University, 13a Sverdlova St., 220006, Minsk, Belarus

E-mail: dmitrykuis@mail.ru

**Abstract**. The paper presents the investigation results of the structure and properties of coatings deposited from separated vacuum-arc discharge flows based on TiN alloyed with aluminum. The influence of the coatings elemental composition on the structure, phase composition and their mechanical properties was established. It is determined that coatings containing in the hexagonal phase $Ti_3Al_2N_2$ are characterized by high hardness, low friction coefficient, maximum values of elastic recovery and plastic deformation resistance.

## 1. Introduction

One of the topical scientific and practical problems of modern materials engineering and machine building is the development of new nanostructured materials and coatings possessing a complex of unique characteristics. The specific properties of nanostructured coatings are largely due to the their structure feature: high volume fraction of interfaces and strong binding energy of neighboring phases, absence of dislocations inside nanocrystallites, presence of intercrystalline amorphous interlayers; change in the relative solubility of components in the interstitial phases, etc. [1-3]

Widely known coatings based on nitrides of transition metals (titanium, zirconium, chromium) are both unstable at elevated temperatures and do not provide high corrosion resistance of the layers formed. The solution of this problem is the introduction of various elements (B, C, Al, Si, Cu or Cr) into coatings based on titanium nitride, which allows one to increase their hardness, durability and improve operating properties [4-8].

Of great interest in this connection is the process of coatings deposition from a separated multicomponent plasma cathode-arc discharge, which makes it possible to form coatings with a homogeneous structure, low roughness and equal distribution of elements by volume.

## 2. Materials and methods

In the present work, a modernized vacuum-arc deposition unit equipped with a particulate separator was used to form multicomponent coatings. Coatings were formed by simultaneous sputtering of two cathodes (titanium and aluminum) in nitrogen atmosphere (reaction gas pressure $0.3 \times 10^{-2}$ Pa), the bias potential on the substrate was -60 V.

The X-ray diffraction analysis was carried out using a DRON-3 X-ray diffractometer in the interval angles of 30-120 ° in filtered Cu-Kα-radiation. Using the basic characteristics of the diffraction maxima allowed us to calculate the size of the coherent scattering regions (L).

Nanoindentation probe Nano Indenter G200 "Agilent" was used to perform the nanoindentation, the measurement of mechanical parameters was carried out by the method of continuous indentation using the Oliver-Far method (the Berkovich trihedral diamond indenter with a radius of rounding at the vertex of 20 nm, depth of indentation was 500 nm).

## 3. The study of the structure, phase composition and mechanical properties of $Ti_xA_{1-x}N$ coatings

As a result of X-ray diffraction analysis of $Ti_xA_{1-x}N$ coatings with different element ratios, the main phase in the coating composition (x = 0.90-0.75) is the crystalline phase of a solid solution (Ti, Al) N with a cubic structure of NaCl type (fig. 1).

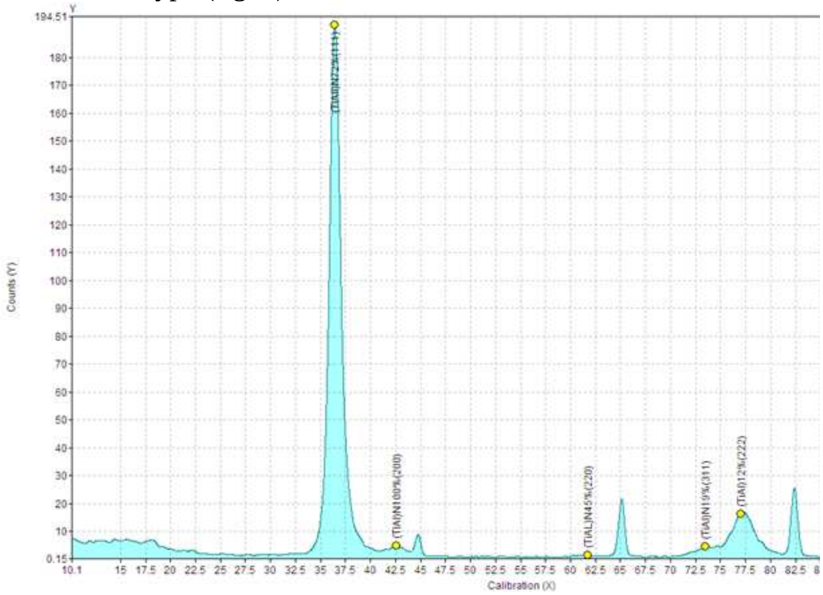



**Figure 1.** Diffractogram of $Ti_{0,90}\,Al_{0,10}\,N$ coatings

An increase in the amount of aluminum in the plasma stream leads to the formation of a $Ti_xAl_{1-x}N$ coating (x = 0.70) containing the hexagonal phase of $Ti_3Al_2N_2$ (Fig. 2).

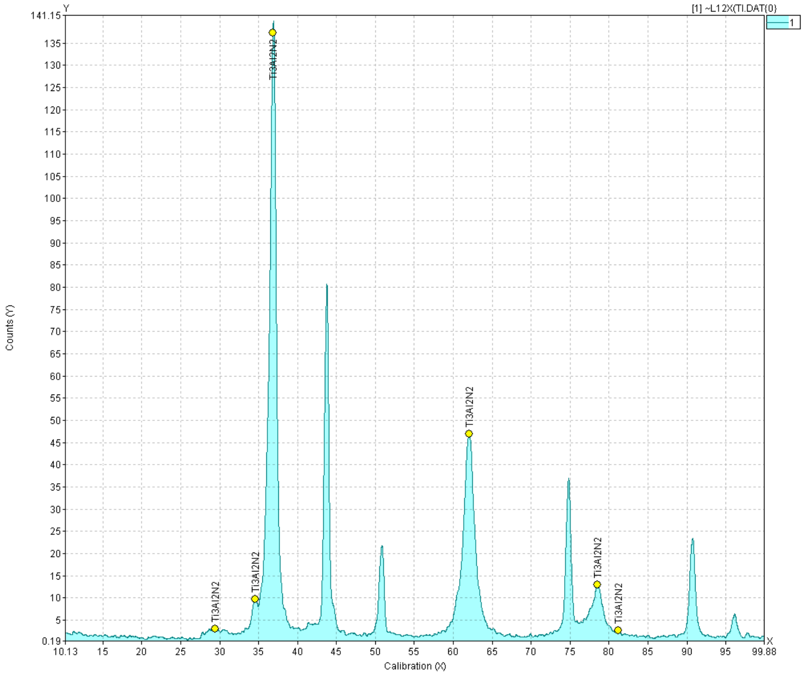



**Figure 2.** Diffractogram of $Ti_{0,70}\,Al_{0,30}\,N$ coating

As a result of the investigation of $Ti_xAl_{1-x}N$ coatings (x = 0.45-0.60), it was revealed that the hexagonal phase of $Ti_3Al_2N_2$ decays with the formation of TiN and AlN phases (fig. 3). An increase in the volume fraction of the AlN phase was observed with an increase in the concentration of aluminum in the coating (fig. 4).

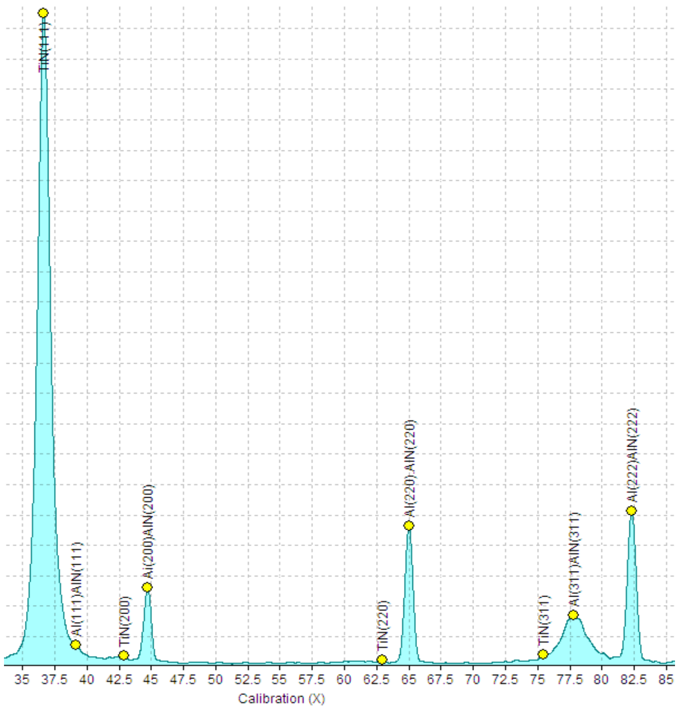



**Figure 3.** Diffractogram of $Ti_{0,60}\,Al_{0,40}\,N$ coating.

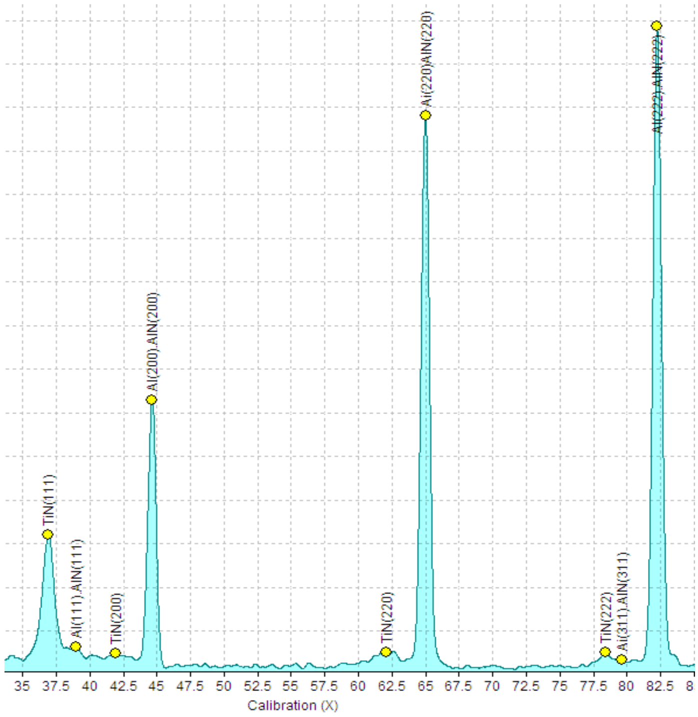
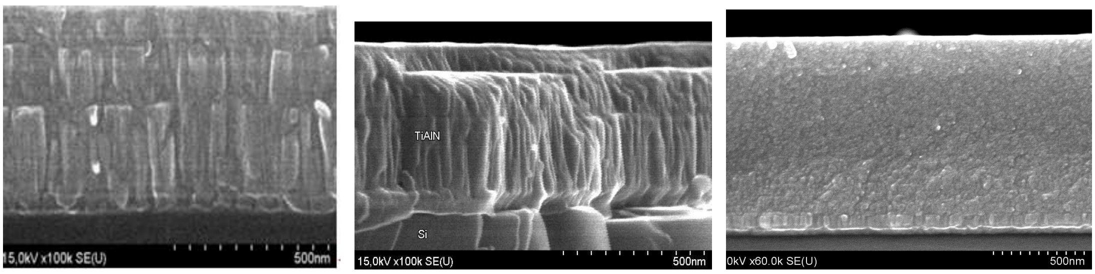



**Figure 4.** Diffractogram of $Ti_{0,45}\,Al_{0,55}\,N$ coating.

As studies have shown, an increase in the amount of the alloying element in the coating leads to its structure change from the mononitride coatings columnar structure to a nanoscale structure (fig. 5). Also, the structure of the formed coatings is affected by the process of separation of a multicomponent plasma flow from macro-dimensional formations that are characteristic of vacuum-arc deposition. The process of separation makes it possible to significantly reduce the inhomogeneous of the coatings structure [9, 10].




a b c

**Figure 5.** Fractogram of coatings: a) – the structure of TiN coating; b) – the structure of $Ti_{0,90}Al_{0,10}N$ coating; c) – the structure of $Ti_{0,30}Al_{0,70}N$ coating.

The crystallite size in the growth direction was from 1.5 to 8 nm, depending on the aluminum concentration in the coatings (table 1). The formation of fine grained structures coatings can be a factor that reduces internal stresses and inhibits the nucleation and growth of fatigue cracks in the volume of the coating material.

Since the wear mechanism of protective wear-resistant coatings is associated with adhesion and chemical processes occurring in the contact zone, to improve the performance properties of hardened products the coatings must have high hardness, deformation resistance and low friction coefficient, which can be achieved by forming a nanostructure of condensates.

Doping of coatings on the basis of titanium nitride with aluminum increases the nanohardness of coatings to 34.4 GPa (table 1). However, to improve the coatings performance simultaneously with high hardness, coatings must have a high resistance to deformation.

In this paper, the main characteristics of the material's resistance to deformation, such as the effective modulus of elasticity (E*), the plasticity index ($H/E^*$), the elastic recovery $W_e = H^2/E^*$, the plastic deformation material $H^3/E^{*2}$ were calculated by using loading / unloading curves, obtained for coatings with different elemental composition. Based on the analysis of the calculated characteristics $Ti_xAl_{1-x}N$ coatings are characterized high values of elastic recovery and plastic deformation resistance in comparison with the hard alloy and TiN coatings (table 1). Moreover, the coatings $Ti_xAl_{1-x}N$, where x = 0.70-0.80, are characterized by a higher resistance to deformation, it seems to be associated with the formation of a more stable hexagonal phase of $Ti_3Al_2N_2$.

**Table 1.** Structural and mechanical properties of coatings

| Coating | Grain size, nm | H, GPa | $E^*$, GPa | H/E* | $W_e$, GPa | $H^3/E^{*2}$, GPa | Friction coefficient |
|---|---|---|---|---|---|---|---|
| $Ti_{80}Al_{20}N$ | 2,1 | 32,8 | 350 | 0,093 | 3,07 | 0,29 | 0,35 |
| $Ti_{70}Al_{30}N$ | 1,5 | 34,3 | 345 | 0,099 | 3,410 | 0,339 | 0,3 |
| $Ti_{60}Al_{40}N$ | 8 | 30,2 | 353 | 0,086 | 2,584 | 0,221 | 0,45 |
| Hard alloy | - | 21,5 | 665 | 0,69 | 0,032 | 0,02 | 0,87 |
| TiN | 37,8 | 25,2 | 429 | 1,60 | 0,061 | 0,097 | 0,7 |

Destruction in the zone of frictional contact develops under highly nonequilibrium conditions and can be accompanied by intense plastic deformation of the coating material, focal temperature increase, oxidation and a number of other processes. As shown by the results of tribological tests of coatings $Ti_xAl_{1-x}N$ (table 2), the minimum values of the coefficient of non-lubricating friction (0.3-0.35) were obtained for coatings, where x = 0.70-0.80, which is 3 times lower than at a basis from a hard alloy and 2 times lower than in TiN coating.

## 4. Conclusion

As a result of the conducted studies it was shown that the variation of the elemental composition of $Ti_xAl_{1-x}N$ coatings formed by the vacuum-arc deposition method from separated plasma flows leads to the evolution of their structure and phase composition. It has been established that composed of the hexagonal phase $Ti_3Al_2N_2$ coatings are characterized by high hardness, low friction coefficient, maximum values of elastic recovery and plastic deformation resistance.

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