Modelling and Optimization of Technological Process for Magnetron Synthesis of Altin Nanocomposite Films on Cutting Tools

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Abstract. The paper highlights the results of the research on developing the mechanism to model the technological process for magnetron synthesis of nanocomposite films on cutting tools, which provides their specified physical and mechanical characteristics by controlling pulsed plasma parameters. The paper presents optimal conditions for AlTiN coating deposition on cutting tools according to the ion energy of sputtered atoms in order to provide their specified physical and mechanical characteristics.

Introduction
Modern nanostructured coatings enhance cutting tool wear resistance providing tool lifetime to be increased and metals to be machined at higher speeds. Improvement of technical characteristics (hardness, viscosity) of cutting tools with nanocomposite thin films results in sufficient increase in labor efficiency and reduction of costs of the products manufactured.

Application of modern protective thin films allows employing tools with nanocoatings and refurbishing tools up to 12 times.

The greatest potential for improving the performance characteristics of cutting tools, such as hardness HB, durability σv, heat endurance Tq, abrasion resistance Tasr, oxidation resistance Tox, consists in the right choice and calculation of properties of the chemical composition and the structure of the coating for specific operating conditions [1], as well as in improving technology to form nanostructured wear resistant coatings. Cutting tool performance characteristics are mainly determined by behavior of thin films deposited as they interact with the processed surface.

One of the methods to solve the problem to obtain nanocomposite coatings with specified physical-mechanical characteristics, such as indentation hardness H, elasticity modulus E, degree of coating adhesion to substrate HF etc., is improvement of physical and technical processes of their synthesis. Therefore, the leaders in the production of metal-cutting tools such as Sandvik Coromant, Balzers Aerlicon, Iscar, Mitsubishi, Dormer, Walter, and others, are actively developing in this direction.

Currently, the most promising methods of coating deposition are the vacuum ion-plasma technologies. Among them one can single out the method of magnetron sputtering, as the drift current of electrons in crossed electric and magnetic fields used in it allows providing uniformity of the coatings applied on large areas of sputtering, as well as forming fine- and superfine-dispersed structures.
One of the main problems of applying thin-film coatings on metal cutting tools by the method of magnetron sputtering is the inability to predict accurately their composition and structure, because there are many factors affecting the properties of the resulting coating [2], [3]. One of the most important parameters of technological process of nanocomposite coating synthesis is the ion energy of sputtering atoms.

Ion energy and ion flow of sputtering atoms have been studied by many Russian and foreign scientists [4] - [8]. However, the majority of works are devoted to the study of influence of the current-voltage characteristics on the magnitude of ion energy and ion flux distribution. Until now there have not been studies considering influence of such pulse parameters as pulse frequency and pause time. Monitoring the amount of ion energy and distribution of ion fluxes of sputtering atoms of the growing thin films by varying the frequency characteristics can be used for simulation and improvement of structure and properties of films with specified physical and mechanical characteristics of the coating and for intensification of magnetron sputtering process. In this connection, it is necessary to determine exactly how the pulse frequency and the pause time affect the physical and mechanical characteristics of high quality coatings obtained.

**Analysis of field of application and obtaining of nanocomposite coatings for metal-cutting tools**

The most promising method for deposition of thin films is the method of dual unbalanced magnetron ion-plasma sputtering with a closed type field, since it allows synthesis of coatings of almost any composition with high productivity while ensuring a durable adhesion against the substrate, and also allows increasing degree of plasma ionization in the area of coating synthesis on the product. Methods of nanocomposite coating deposition on cutting tools are presented in Fig.1. [3].

As the research showed, currently, the magnetron-sputtering system using unbalanced dual magnetrons with a closed type field operating in a pulsed mode is the perfect system [4].

In this system, cathodes are alternately operated, increasing the density of the ion current near the target. It allows to obtain a high degree of plasma ionization, which has a positive effect on the properties of nanocomposite thin films applied to cutting tools, improves the performance of industrial plants by increasing the size of the "effective" area of the coating deposition, it is possible to synthesize thin films on products of large sizes and complex configurations.
The results of the analysis of physico-mechanical properties of the coatings show that the mechanical, thermal and tribological properties of hard thin films can be significantly optimized through a well-designed microstructure, driven by the energy of ion bombardment, by varying the pulse plasma parameters [5].
Optimization of technological process of AlTiN coating deposition

As a result of research the analysis of the technological process of AlTiN coating deposition was carried out according to the scheme presented in the table format, where \( P_b \) is the base pressure, Pa; \( P_w \) is the working pressure, Pa; \( U_d \) is the discharge voltage on the magnetrons, B; \( I_d \) is the discharge current on the magnetron, A; \( Bias \) is the offset voltage, B/ is the bias current, A.

Table 1 Technological process of AlTiN nanocomposite coating deposition

| Seq. No | Operation                              | Time min. | Values of assigned parameters                                                                 |
|---------|----------------------------------------|-----------|-----------------------------------------------------------------------------------------------|
| 1       | Loading items into the chamber         | 10        | Surface preparation for coating deposition                                                     |
| 2       | Pumping the vacuum chamber to base pressure | 30-60     | \( P_b=0.001 \) Pa                                                                          |
| 3       | Ion cleaning                           | 10        | \( P_b=0.12 \) Pa; \( U_d=650/650 \) V; \( I_d=1/0.5 \) A; \( Bias=800 \) V/40 A            |
| 4       | Deposition of the metal sub-layer       | 30-50     | \( P_w=0.12 \) Pa; \( U_d=650/650 \) V; \( I_d=6/0.5 \) A; \( Bias=40 \) V/40 A            |
| 5       | Deposition of reactive layer            | 50-75     | \( P_w=0.1-0.15 \) Pa; \( QN2=5-15 \)%; \( U_d=600-800 \) V; \( I_d=1-6 \) A; \( Bias=40-50 \) V/40-50 A |
| 6       | Cooling-down                           | 15        | \( P_w=0.12 \) Pa                                                                           |
| 7       | Items unloading                        | 10        | -                                                                                           |
Based on the model offered by Davis and Vanderslice a mathematical model to measure the ion energy distribution was developed, taking into account the length of free path of an electron in crossed electric and magnetic fields in relation to the process of ionization and frequency of electron-atom collisions:

\[
\frac{d\gamma}{d\varphi} = \left(\gamma \cdot I_0 \cdot \mu_\perp \cdot \varepsilon_0\right)^{\frac{1}{2}} \left[\frac{\omega}{(\lambda \cdot \nu)}\right] \left[\frac{U \cdot \left(\gamma^{\frac{1}{2}} - 1\right)}{U_k} + 1\right],
\]

where \(j_i\) is the ion flux, A/m²; \(I_0\) is the discharge current; \(\gamma\) is the Townsend coefficient; \(\mu_\perp\) is electron mobility in the direction perpendicular to the magnetic field, m²/B·sec; \(\varepsilon_0\) is the dielectric constant; \(\omega\) is the cyclotron frequency, Hz; \(\lambda\) is the average track length of the electron, m; \(\nu\) is the frequency of electron-atom collisions, Hz; \(U\) is the electric field strength, B; \(U_k\) is the cathode potential drop, B.

In order to account for possible ionization in the magnetron sputtering process made the following assumptions and limitations are taken:

- the mean free path of electrons is considered to be constant;
- edge effects are not considered;
- given that, the degree of ionization of low-temperature plasma of gas discharge is small (of order \(10^{-5}\)), one neglects the collisions of electrons with any particles, besides atoms of the working gas.
- the x-axis is perpendicular to the cathode and directed to the anode, and the y-axis is parallel to the cathode and directed along the magnetic field line;
- within the cathode layer the field is homogeneous;
- acceptable value of ion energy is \(j(U)\leq U_k\).

To determine the energy spectrum of the ions, bombarding the cathode unbalanced magnetron, the value of cathode potential drop was determined according to the formula:

\[
U_k = 1.2 \cdot 10^{-29} \cdot \left[\frac{\gamma \cdot I_0 \cdot T}{P \cdot B}\right]^{\frac{1}{2}} \cdot \sigma^{\frac{3}{2}} \cdot \left(\gamma^{\frac{1}{2}} - 1\right),
\]

where \(T\) is the gas temperature; \(P\) is the gas pressure in the chamber; \(B\) is the magnetic flux density; \(\sigma_i\) is the cross section of ionization in the magnetron discharge, \(\sigma_i=2\cdot10^{-19}\) m².

Having solved differential equation (1) for the ion flux density under the initial condition \(j(U_k)=0\) we get the expression describing the energy distribution of the ions bombarding the cathode:

\[
\frac{\omega \cdot (U - U_k)}{\lambda \cdot \nu} \cdot \sqrt{\frac{\omega}{\lambda \cdot \nu}} \cdot \sqrt{\mu \cdot I_0 \cdot \varepsilon_0} \cdot \left(U + U_k - \sqrt{\gamma \cdot U + \sqrt{\gamma \cdot U_k}}\right)
\]

The results of mathematical simulation proved that the largest contribution to the sputtering process make not high-energy ions, since their density in the flow is small, but the ions with average energy values. Increasing discharge voltage results in increase in the value of the cathode potential drop. As a result, ions are accelerated with greater speed and, consequently, their kinetic energy increases, which is transferred to the target atoms, which has a positive effect on sputtering rates.

**Influence of pulse parameters on the properties and structure of AlTiN coating**

Performing a research on the influence of pulse parameters on properties and structure of AlTiN coatings the values of the pause time (time of return) and the values of the frequency of the pulses
varied. Based on mathematical simulation, the following values of process parameters were chosen: pulse frequency 100KHz and 350KHz (the minimum allowed and maximum possible) the pause time of 1.0 µs to 5.0µs.

The results of the analysis of two and three-dimensional surface morphologies of AlTiN films deposited under different asynchronous pulsing conditions, were measured using atomic force microscopy (AFM) and scanning electron microscopy (SEM) (Figure 3). Change of ion energy and ion flux in the plasma by pulsation shows that the increase of ion energy and ion flux leads to a change in AlTiN film texture from (200) to (111). In the study, the films deposited at relatively low ion energy bombardment (e.g., a mode 100/1,0 with total ionic energy 72eV) support orientation (200) to minimize the surface energy. With increase of ion energy and ion flux in the plasma increased strain energy in the film and re-sputtering due to the high ion bombardment cause the growth of the coating in the orientation (111) to reduce the strain energy (for example, 350kHz/1,4µs with a total ion energy of 200eV).

Figure 3. SEM cross micrographs of AlTiN films, deposited under different conditions of asynchronous pulsing: a) 100KHz and 1.0µs (72eV); b) 100KHz and 2.5µs (84eV); c) 100KHz and 5.0µs (122eV); d) 350KHz and 0.4µs (177eV); e) 350KHz and 1.0µs (180eV); f) 350KHz and 1.4µs (200eV)

Pulsed ion energy and ion flux have significant impact on the micro-structural development of AlTiN film. The use of controlled ion bombardment to maintain the maximum ion energy at the level less than 120eV and increase the ion flow in the central field of ion energy "B", will increase adatom mobility, reinforce coating density and reduce the size of the film columnar crystals.
Mechanical and tribological properties of $AlTiN$ films obtained under different conditions of deposition are presented in Table 2.

Hardness of the $AlTiN$ film has increased from 34 to 41 HPa under growth of the total ionic energy from 72eV (100KHz/1.0µs) to 122eV (100KHz/5.0µs) in the asynchronous pulse mode. This is due to increasing density of the coating and reduction of grain size under conditions of increased ion energy and ion flux bombardment. Strain hardening became significant and excessive, with a total ion energy 177-200eV and the pulse frequency 350KHz.

### Table 2 Mechanical and tribological properties of $AlTiN$ films

| Film properties            | Pulse parameters |
|----------------------------|------------------|
|                            | 100/1.0          | 100/2.5         | 100/5.0         | 350/0.4         | 350/1.0         | 350/1.4         |
| $Al/(Ti+Al)$               | 58.5             | 57.3            | 60.4            | 60.9            | 67.5            | 69.7            |
| Film thickness, mm         | 1.2              | 0.8             | 1               | 14.1            | 0.9             | 1.2             |
| Surface roughness, nm      | 5.45             | 2.43            | 1.04            | 2.16            | 2.01            | 2.25            |
| Residual stresses, HPa     | -3.4             | -4              | -5.6            | -7.3            | -12.2           | -12.8           |
| Nanoidentation hardness, HPa| 34.3±3.8         | 37±3.9          | 41.1±3.3        | 42.4±2.9        | 43.6±2.8        | 48.0±3.6        |
| The Young's Modulus, HPa   | 370.8±26.3       | 385.6±31.9      | 411.4±32.5      | 432.4±30.4      | 450.5±28.4      | 515.5±43.7      |
| $H/E$                      | 0.092            | 0.096           | 0.01            | 0.098           | 0.097           | 0.093           |
| Friction coefficient       | 0.38             | 0.43            | 0.46            | 0.46            | 0.87            | 0.63            |
| Wear rate                  | 2.9              | 3.2             | 3.4             | 3.9             | 3.8             | 4               |

As a result, the optimal combination of mechanical (hardness) and tribological properties can be achieved only when saving the average ion energy between 70-120eV with high ion fluxes. The work of $Ti$ and $Al$ targets at 100kHz frequency and pause time of 5.0µs resulted in formation of $AlTiN$ films, which showed high hardness (~ 41HPA, viscosity of 0.10) and good wear resistance (coefficient of friction 0.40-0.46; rate of wear $3.4 \times 10^{-6}$ mm$^3$H$^{-1}$ m$^{-1}$).

### Conclusion

According to the data obtained concerning the influence of the frequency characteristics of the pulse on the properties of the $AlTiN$ coating a model was prepared for technological process of magnetron sputtering, providing specified physical and mechanical properties of nanocomposite coatings of metal-cutting tools. Cutting tools coated with AlTiN nanocomposite coating obtained using the developed technology of magnetron sputtering were tested, as well as it was compared with analogues (Fig.4).
Based on the research recommendations for improving efficiency of the process of magnetron sputtering were formulated:

- Discharge voltage on the target should be chosen in accordance with the percentage of asymmetric pulse to maintain the reverse positive pulse voltage in the range of 20-80V. It is preferable to use a longer time of return at relatively low pulse frequencies (e.g., 100-200KHz) for reactive pulsed sputtering, for the purpose of obtaining appropriate values of the pulsed ion energy and increasing of ion flux.

- Since pulsed plasma has already provided relatively high values of ion energy in the plasma, it is proposed to use relatively small displacement of the substrate (not more than -50 B) to avoid excessive ion bombardment.

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