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Technological Features of Creating Nanostructured Decorative-Protective Coatings

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Abstract

Nanotechnologies in transport engineering are particularly related to the creation of different protective coatings as a result of particle flows acting upon structural materials in vacuum. Nature of effect depends on the type of particles (electrons, ions, atoms, molecules), on their energy and chemical activity as well as on product materials (metals, semiconductors, dielectrics). The work analyses the properties of decorative, protective multi-component coatings on the basis of Ti-Al-N, which are obtained by vacuum ion-plasma sputtering. There has been carried out a series of experiments aimed to obtain single-layer coatings according to a certain pattern of sputtering modes in argon and nitrogen environment. The obtained coatings have a wide variety of colors. Different properties of the created coatings including spectral characteristics, at different angles of incidence of light as well as chemical composition have been investigated.

KEY WORDS: *ion-plasma deposition in vacuum, decorative and protective coatings, technological features of creating*

1. Introduction

The protection of metals and composite materials from corrosion and erosion is a topical issue that affects all areas of the world's economy [1-4]. The method of ion-plasma sputtering is widely used in industry to create coatings for different purposes. Existing technologies allow to create coatings based both on fine metals and fine metal compounds (nitrides, carbides). Titanium-based coatings, for instance, have good corrosion- and wear-resistant properties. In atmospheric air conditions, water, many salt solutions, inorganic and organic acids, in terms of corrosion resistance, titanium is substantially superior to many other metals, while under the effect of sea water, for instance, it is inferior only to noble metals (gold, platinum, etc.). Besides, the chemical activity of the metal allows it to be used in many compounds, and makes it possible to obtain coatings in a wide range of colours. Thus, golden spectrum coatings (titanium nitride) allow to simulate noble metals. In turn, on the basis of aluminum, it is possible to create, for instance, reflective coatings. Being sputtered by ion-plasma method, aluminum has good technological and adhesive properties. Compounds based on Ti-Al-N system are more and more widely used for creating wear-resistant coatings. Such compounds are prospective also from the point of view of applying them as decorative or protective decorative coatings.

The work analyses the properties of Ti-Al-N based protective decorative single-layer coatings obtained by the method of vacuum ion-plasma sputtering [1, 5-7]. Vacuum ion-plasma sputtering is a final operation, so subsequent mechanical treatment of protective decorative coatings is excluded. The quality of coatings in this case is determined by the quality of workpiece surface (roughness, texture etc.), as well as the quality of preliminary cleaning (presence of contamination, oxide films, etc.).

2. Technological Equipment

Were conducted a series of experiments to create multi-component ion-plasma coatings by vacuum deposition on Ti-Al-N basis.

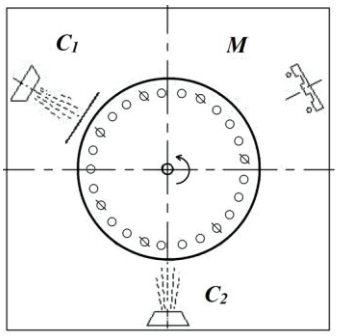

Fig. 1 The vacuum chamber deposition scheme of the ion-plasma installation NNV-6,6-I1. C_1 – first electric arc vaporizer (Ti) with a protective screen; C_2 – second electric arc vaporizer (Ti); M – magnetron (Al).

Ion-plasma sputtering of the coatings was carried out with the help of a modernized vacuum installation NNV6.6-II [5-7]. The experimental investigation involved the use of two sources of plasma – arc evaporator (Ti) and magnetron (Al) (Fig. 1). Electromagnetic shift stabilization, cathode spot focusing in the end face of the cathode being evaporated as well as separation of sputtered material flow from the drop phase were provided in the process of sputtering by using the arc evaporator. The use of two independent sources of deposition as well as controlled feeding of reaction gas (N) into the vacuum chamber made it possible to adjust the composition of the multicomponent layer being deposited.

3. Spectral Characteristics of Protective Decorative Coatings

The colours of protective decorative coatings are usually presented in so called 'Cielab' units received through processing the data of coating reflection from the source of light which is the closest to the spectral distribution of daylight (of 'C' type) based on the following parameters: L* (coating lustre); +a* (red colour component); -a* (green colour component); +b* (yellow colour component); -b* (blue colour component).

The spectral analysis of decorative coatings was carried out according to RGB (Red, Green, Blue) additive colour measurement system by using graphics editor Paint.NET v3.5.6. RGB parameters characterize the saturation of red, green and blue shades of a colour, respectively, and vary within the range from 0 to 255.

The results of the spectral analysis of decorative coatings according to RGB system for 8 studied samples are shown in Table 1. The experimental data are presented in the order of Ti content increase.

Main colour characteristics include the following (see Table 1):

- H_{ue} determines a shade for red, green and blue components of a selected colour within the range from 0 to 360;
- Saturation (S_{at}) varies within the range from 0 to 100. The higher the value of this parameter, the 'purer' is the colour, therefore this parameter is sometimes called colour purity. As the value of Sat parameter is approaching zero, the colour is approaching to neutral grey.
- Colour brightness (V_{al}) also varies within the range from 0 to 100.

Table 1

Spectral characteristics of protective decorative coatings

Sample No.	H_{ue} (0-360)*	S_{at} (0-100)*	V_{al} (0-100)*	Red (0-255)*	Green (0-255)*	Blu (0-255)*
1	43	35	49	128	116	83
2	28	35	31	81	66	52
3	213	17	54	115	125	139
4	247	20	30	68	66	83
5	206	29	37	68	82	95
6	214	22	37	74	83	96
7	109	96	61	43	43	42
8	54	11	48	124	122	110

Note: * - range of possible parameter values.

An energy dispersive electron microprobe analysis was carried out by the methodology described in work [5] with the aim to determine the chemical composition of the created coatings. Due to a large relative error of the analysis in terms of nitrogen N content, the spectral analysis was carried out by two elements – Ti and Al. Table 2 contains the data on the chemical composition of the created protective decorative coatings.

Table 2

Percentage ratio of Ti and Al content in protective decorative coatings

Sample No.	Ti, (%)	Al, (%)
1	64.25	35.75
2	49.35	50.65
3	40.86	59.14
4	23.16	76.84
5	36.87	63.13
6	38.89	61.11
7	21.32	68.68
8	19.17	80.83

Table 3

Results of the evaluation of linear regression models for the values Red, Green, Blue

Variable	Effect factor	β_i coefficient		
		Red mod.	Green mod.	Blue mod.
x_1	Ti %	2.2	1.6	1.1.
x_2	$I_{el.lok.}$	2.8	3.0	1.2
x_3	I_f	-1756.3	-1575.0	-527.9
x_4	I_m	42.3	40.0	
x_5	P_{Ar}	-87659.7	-68943.6	-27756.1
x_6	P_N	47400.1	60815.2	25657.4
x_7	U_m	-	-	-1.7

The work also includes an analysis of the effect of some technological parameters on the increase of the amount of red, green and blue hues in the formation of coating colour. A statistical analysis of the data was carried out with the help of STATISTIKA 7 software package. Correlation of main components in coating composition – Ti/Al; strength of arc evaporator current ($I_{el.lok.}$), strength of magnetron current (I_m), focusing current strength (I_f), magnetron voltage (U_m), argon reactive gas pressure (P_{Ar}), nitrogen reactive gas pressure (P_N) were taken as technological parameters to be evaluated. Based on the results of statistical processing, three linear regression models were built respectively for Red, Green and Blue values. The results of model evaluation are presented in Table 3.

The adequacy of the models was evaluated by Fisher criterion (F), p-error and sum of squared deviations (R_0). The corresponding data for the three obtained models are presented in Table 4.

$$Y^{\wedge}_{Red} = 508.4 + 2.2x_1 + 2.8x_2 - 1756.3x_3 + 42.3x_4 + 87659.7x_5 + 47400.1x_6\,; \quad (1)$$

$$Y^{\wedge}_{Green} = 411.9 + 1.6x_1 + 3.0x_2 - 11575.0x_3 + 40.0x_4 - 68943.6x_5 + 60915.2x_6\,; \quad (2)$$

$$Y^{\wedge}_{Blue} = 682.6 + 1.1x_1 + 1.2x_2 - 527.9x_3 - 27756x_5 + 25657.45x_6 - 1.7x_7\,. \quad (3)$$

Table 4
Criteria for evaluating the adequacy of linear regression models

Model	F	p	R_0
Red	8.06	0.02	60.89
Green	20.28	0.05	14.74
Blue	3.36	0.08	64.66

The results of the correlation analysis of analytical models are shown in Table 5. The obtained data show that the Red value is directly proportional to Ti content in the created coating. The Green value is directly proportional to Ti content and arc evaporator current strength. The Blue value is directly proportional to the pressure of reactive gases *N* and Ar in the sputtering chamber, and inversely proportional to the strength of magnetron current and focusing current. The effect of Ti content in the coating on the Blue value is quite small.

Table 5
Correlation coefficients of Red, Green and Blue values

Effect factor	Red	Green	Blue
Ti %	0.63	0.59	0.18
$I_{el.lok.}$	0.12	0.42	-0.29
I_f	-0.10	-0.36	-0.63
I_m	-0.22	-0.21	-0.51
P_{Ar}	-0.25	0.01	0.48
P_N	-	0.10	0.69
U_m	-	-	-

Figs. 2 and 3 show the results of the evaluation of Ti content effect on the value of Red and Green respectively, while Fig. 4 demonstrates the effect of nitrogen pressure in the sputtering chamber on the Blue value. The data are presented in comparison with linear prediction.

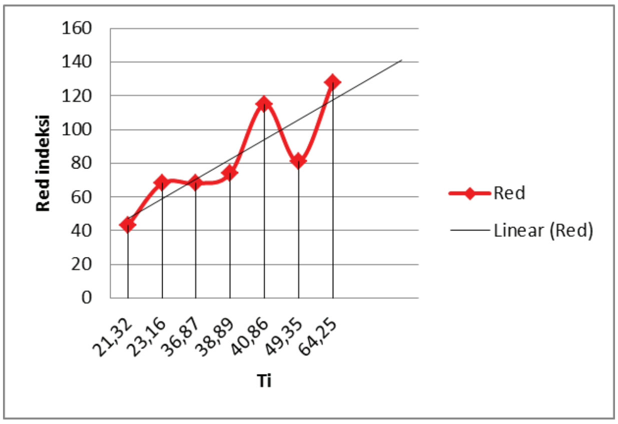

Fig. 2 The effect of Ti content on the change of Red value according to RGB

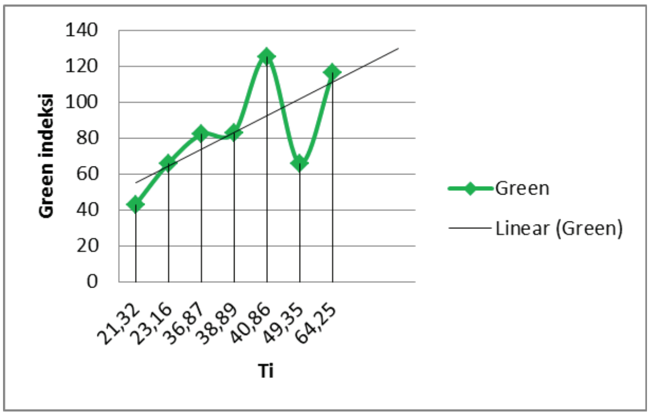

Fig. 3 The effect of Ti content on the change of Green value according to RGB

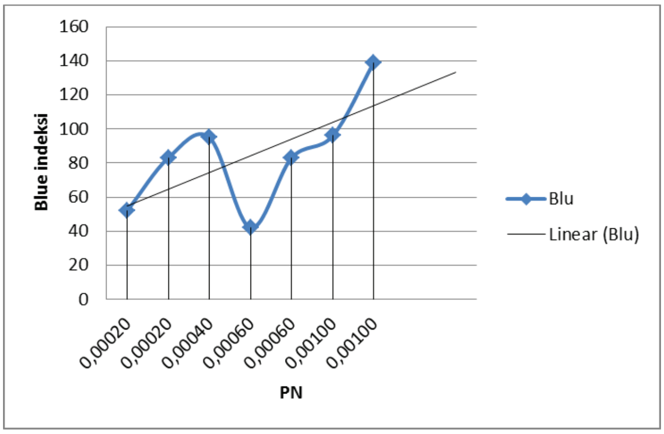

Fig. 4 The effect of nitrogen pressure in the sputtering chamber on the change of Blue value according to RGB

4. Conclusions

The work considers the peculiarities of the technology for depositing Ti-Al-N based protective decorative single-layer coatings. There has been carried out a series of experiments on the creation of single-layer multi-component ion-plasma coatings through vacuum sputtering in argon and nitrogen environment. Various coating properties – spectral characteristics, chemical composition – have been studied.

The effect of the chemical composition of protective decorative coatings on spectral characteristics determined with the help of RGB system has been revealed. The obtained coatings have a wide variety of colours.

The research results can be used in practice for the production and repair of vehicles.

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