

# THE INFLUENCE OF DEPOSITION TECHNOLOGICAL PROCESS PARAMETERS ON SURFACE PROPERTIES OF MULTILAYER COATINGS

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The surface of material plays a basic role in determining of various processes such dyeing penetration, chemical absorption, biocompatibility and others. At present study the surface parameters investigation for various multilayer coating types TiN, CrN, (Ti,Cr)N, TiN/TiC<sub>10</sub>N<sub>90</sub>, TiN/TiC<sub>20</sub>N<sub>80</sub> and oxide Al<sub>2</sub>O<sub>3</sub> films deposited by means Arc-PVD technology and Reactive Magnetron Sputtering (RMS) methods on the stainless steel (1H18N9) has been made. The surface thickness, roughness, topography, surface free energy (SFE) was evaluated for next conclusions about surface properties of multilayer coatings.

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## 1. INTRODUCTION

Surface properties such as composition, roughness, topography, wettability can influence events at material interface. Modern techniques are widely used for surface parameters modification and multilayer organic-inorganic (also with nano structural properties) coatings deposition: modified Arc-PVD Method, magnetron sputtering (MS) and ion-beam assisted deposition (IBAD) Methods [1]. The surface free energy (SFE) is one of the basic surface material properties and very important for various optical, environmental and biological applications [2]. The effects of materials composition, surface chemistry and surface topography on cell adhesion and proliferation have been largely studied [3]. The aim of the study was the comparative analysis of the surface parameters of various multilayer coating types for next industrial applications.

## 2. MATERIALS AND METHODS

At present study the surface parameters investigation for various multilayer coating types TiN, CrN, (Ti,Cr)N, TiN/TiC<sub>10</sub>N<sub>90</sub>, TiN/TiC<sub>20</sub>N<sub>80</sub> deposited by means Arc-PVD technology on the stainless steel (1H18N9) and also the same coatings with additional oxide Al<sub>2</sub>O<sub>3</sub> film deposited by Reactive Magnetron Sputtering (RMS) methods has been made.

The substrates for deposited coatings were stainless steel 1H18N9 samples. The substrates were cleaned in ultrasonic bath with standard technology. The TiN coatings were deposited by means Arc-PVD Method. The main parameters of the process were described in our previous study [4] reactive atmosphere – N<sub>2</sub>, deposition pressure  $8 \times 10^{-2}$  Pa, arc current – 100 A, substrate bias – 150 V, deposition temperature – 593 K, the thickness of deposited coatings 2...5 μm. Al<sub>2</sub>O<sub>3</sub> (RMS) coating deposition was performed in high vacuum pumping system with the base pressure about  $10^{-5}$  mbar. A pure aluminum target served as sputtered aluminum source. Power to sputter cathode was applied using DC power supply. For the presented system the stoichiometric

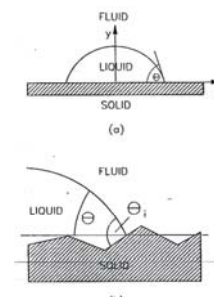
composition region size was nearly 30x30 cm and the distance to substrate 30 cm for all regimes. The main process parameters were: magnetron discharge power 1...8 kW, oxygen source power 1kW, deposition rate 8 μm/hour [1].

The analysis of surface parameters such as surface roughness, wettability, surface free energy were made. The surface roughness was measured by means of profilometer Hommel T-2000. The contact angles were measured by means of tensiometric method in Kruss K12 Tensiometer at temperature 20°C [5]. Prior to contact angle measurements, samples were ultrasonically cleaned in acetone and deionised water and dried. The standard liquids with well-known values of surface tension, component of dispersion and polar interaction such as water, formamide, diiodo methane, ethylene glycol, α-bromo naphthalene and glycerol were used. Also the surface free energy (SFE) and its polar and dispersion components were determined by means of Wu and Owens-Wendt-Rabel-Kaelble methods [6,7].

## 3. RESULTS AND DISCUSSION

The parameters of multilayers coating's thickness and roughness were evaluated (see Table 1)

The Young equation is a fundamental relation between contact angle and the surface tension of liquids, solids and interfacial tension of solid-liquid (see Figure).



*Definition of the contact angle and SFE for a rough surface*

The calculation of surface free energy of solids from the measurements of contact angle is based on the surface energy balance condition:

$$\gamma_s = \gamma_l \cos \theta + \gamma_{sl}, \quad (1)$$

$\gamma_{sl}$  - interfacial tension between liquid and solid (interfacial free energy),

$\gamma_s$  - surface tension of solids,

$\gamma_l$  - surface tension of liquid.

The equation has two measurable values - surface tension of liquids and contact angle and also two unknown values  $\gamma_{sl}$  and  $\gamma_s$ . In thermodynamics the work needed to divide the solid-liquid surface is adhesion energy  $W_a$  (2) and after substitution into Young equation (1) we obtain Young-Dupre equation (3)

$$W_a = \gamma_l + \gamma_s - \gamma_{sl}, \quad (2)$$

$$W_a = \gamma_l (1 + \cos \theta). \quad (3)$$

According to Wu [6] surface interfacial energy is the sum of harmonic mean dispersion and polar interaction multiplied by 4. Complete SFE of solid and liquid is defined as a sum of dispersion and polar interaction.

$$\gamma_l = \gamma_l^d + \gamma_l^p, \quad (4)$$

$$\gamma_s = \gamma_s^d + \gamma_s^p, \quad (5)$$

$$\gamma_{ls} = \gamma_l + \gamma_s - \frac{4\gamma_l^d \gamma_s^d}{\gamma_l^d + \gamma_s^d} - \left( \gamma_l^p \gamma_s^p \right)^{\frac{1}{2}}, \quad (6)$$

where  $\gamma_l^d$  and  $\gamma_s^d$  is a part of SFE contributed by dispersion interaction (the dispersion component),  $\gamma_l^p$  and  $\gamma_s^p$  is a part of SFE contributed by polar interaction (the polar component). The Wu method can be used for the surface free energy estimation by contact angle measurements of two liquids.

Table 2. SFE, its polar and dispersion parts and fractional polarity by Owens-Wendt-Rabel-Kaelble' methods for liquid system:  $\alpha$ -bromonaphthalene- formamide-ethylene glycol-diiodomethane- glycerol-water

Symbol of coatings	Component of surface free energy [mN/m]			
	Dispersion part $\gamma^d$	Polar part $\gamma^p$	Total $\gamma$	Fractional polarity $\gamma / (\gamma^d + \gamma^p)$
(TiCr)N	41.2	3.9	45.1	0.086
(TiCr)N/Al <sub>2</sub> O <sub>3</sub>	33.0	9.1	42.1	0.22
CrN	38.6	4.0	42.6	0.093
CrN/ Al <sub>2</sub> O <sub>3</sub>	30.8	7.7	38.5	0.2
TiN	39.0	7.0	46.0	0.152
TiN/ Al <sub>2</sub> O <sub>3</sub>	34.8	9.9	44.7	0.22
TiN/TiC <sub>10</sub> N <sub>90</sub>	39.7	8.2	47.9	0.17
TiN/TiC <sub>10</sub> N <sub>90</sub> /Al <sub>2</sub> O <sub>3</sub>	31.6	9.9	41.5	0.24
TiN/TiC <sub>20</sub> N <sub>80</sub>	39.2	4.7	43.9	0.107
TiN/TiC <sub>20</sub> N <sub>80</sub> / Al <sub>2</sub> O <sub>3</sub>	33.2	7.1	40.3	0.18

#### 4. CONCLUSIONS

In spite of the large amount of data about structural parameters and composition of multilayer coatings more

Table 1. The coating's thickness and roughness parameters

Symbol of coatings	Parameters of roughness [ $\mu\text{m}$ ]			Thick ness [ $\mu\text{m}$ ]
	R <sub>a</sub>	R <sub>t</sub>	R <sub>z</sub>	
(TiCr)N	0.140	3.000	1.370	1.50
(TiCr)N/Al <sub>2</sub> O <sub>3</sub>	0.111	1.723	0.903	3.70
CrN	0.060	1.153	0.533	1.45
CrN/ Al <sub>2</sub> O <sub>3</sub>	0.053	1.263	0.473	3.40
TiN	0.097	2.300	0.853	1.80
TiN/ Al <sub>2</sub> O <sub>3</sub>	0.250	4.043	1.660	3.90
TiN/TiC <sub>10</sub> N <sub>90</sub>	0.063	1.837	0.460	1.50
TiN/TiC <sub>10</sub> N <sub>90</sub> /Al <sub>2</sub> O <sub>3</sub>	0.150	2.550	1.263	3.50
TiN/TiC <sub>20</sub> N <sub>80</sub>	0.180	4.343	1.783	1.57
Steel 1H18N9	0.040	0.890	0.237	

The geometric approach which combined dispersion and polar components has been used for calculation of SFE by Owens-Wendt-Kaelble method [7]. The model is based on the assumption that total SFE is a sum of dispersion and polar components. The interfacial free energy is a sum of geometric means of dispersion and polar contributions multiplied by factor 2.

$$\gamma_{ls} = \gamma_l + \gamma_s - 2\left(\gamma_l^d \gamma_s^d\right)^{\frac{1}{2}} - 2\left(\gamma_l^p \gamma_s^p\right)^{\frac{1}{2}}. \quad (7)$$

The SFE and its polar and dispersion parts, fractional polarity estimations were made by Wu method for two liquids system and by Owens-Wendt-Rabel-Kaelble' methods for liquid system:  $\alpha$ -bromonaphthalene-formamide-ethylene glycol-diiodomethane- glycerol-water (Table 2).

studies are necessary to obtain the details of specific technological processes and the surface parameters of deposited coatings.

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## ВЛИЯНИЕ ТЕХНОЛОГИЧЕСКИХ ПАРАМЕТРОВ ПРОЦЕССА НАНЕСЕНИЯ МНОГОСЛОЙНЫХ ПОКРЫТИЙ НА ИХ ПОВЕРХНОСТНЫЕ СВОЙСТВА

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Поверхность материалов играет определяющую роль в прохождении многих процессов, таких как окрашивание, химическая абсорбция, биосовместимость и другие. Представлены результаты проведенных исследований поверхностных параметров многослойных покрытий TiN, CrN, (Ti,Cr)N, TiN/TiC<sub>10</sub>N<sub>90</sub>, TiN/TiC<sub>20</sub>N<sub>80</sub>, а также оксидных Al<sub>2</sub>O<sub>3</sub> пленок, нанесенных методами Arc-PVD и реактивного магнетронного распыления (RMP) на сталь (1H18N9). Проведена оценка толщины, шероховатости, топографии, поверхностной энергии и других поверхностных свойств многослойных покрытий.

## ВПЛИВ ТЕХНОЛОГІЧНИХ ПАРАМЕТРІВ ПРОЦЕСУ НАНЕСЕННЯ БАГАТОШАРОВИХ ПОКРИТТІВ НА ЇХ ПОВЕРХНЕВІ ВЛАСТИВОСТІ

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Поверхні матеріалів відіграють значну роль в проходженні багатьох процесів, таких як фарбування, хімічна абсорбція, біосумісність та інші. Представлено результати проведених досліджень поверхневих параметрів багатошарових покриттів TiN, CrN, (Ti,Cr)N, TiN/TiC<sub>10</sub>N<sub>90</sub>, TiN/TiC<sub>20</sub>N<sub>80</sub>, а також оксидів Al<sub>2</sub>O<sub>3</sub>, які нанесено за допомогою методів Arc-PVD та реактивного магнетронного розпилення (RMP) на сталь (1H18N9). Проведена оцінка товщини, шорсткості, топографії, поверхневої енергії та інших поверхневих властивостей багатошарових покриттів.