# Original papers Nanostructured, nanoscale materials and nanodevices

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# Chemically resistant nanostructured protective coatings for metal surfaces

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**Abstract:** The aim of this study is to develop nanostructured protective coatings with high chemical resistance for metal surfaces. To modify paints and varnishes, compositions based on carbon nanotubes (CNTs), bismuth oxide nanoparticles, silicon dioxide, titanium dioxide, magnesium hydroxide and aluminum hydroxide were used. The introduction of nanomaterials into acrylic paint and varnish material helps to increase the adhesive strength of coatings, so the best effect was determined with the introduction of CNTs in the amount of 0.1 % – the adhesive strength increased from 2.0–2.2 MPa to 2.9–3.1 MPa with a change in the nature of the separation of the paint and varnish coating from the substrate from fully adhesive (100 %) to adhesive-cohesive. Evaluation of the chemical resistance of nanostructured paint and varnish coatings modified with CNTs showed that when the coatings were exposed to an herbicide solution, they retained their weight regardless of the concentration of the modifier, while their hardness did not change (H). Evaluation of the equilibrium swelling rate made it possible to determine that paint and varnish coatings modified with bismuth oxide nanoparticles and CNTs are resistant to solvents and acquire a more mesh structure in relation to coatings without additives. Scanning of various sections of the sample surfaces on atomic force microscope (AFM) showed that when modified with bismuth oxide and CNT nanoadditives, the surface becomes smoother, and the nanometer roughness decreases from 50–60 nm to 20–30 nm. A more significant reduction in protrusions is observed in coatings modified with CNTs, which indicates that the coating is strengthened by reducing the size of depressions and pores.

**Keywords:** nanomaterials; nanostructured coatings; paint and varnish coatings and materials; adhesive strength; chemical resistance; metal structures; carbon nanotubes; bismuth oxide.

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# **Химостойкие** наноструктурированные защитные покрытия для металлических поверхностей

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Аннотация: Цель работы заключается в разработке наноструктурированных защитных покрытий с высокой химической стойкостью для металлических поверхностей. Для модификации лакокрасочных материалов применялись составы на основе углеродных нанотрубок (УНТ), наночастиц оксида висмута, диоксида кремния, диоксида титана, гидроксида магния и алюминия. Введение в акриловый лакокрасочный материал наноматериалов способствовало увеличению адгезионной прочности покрытий, так наилучший эффект определен при введении УНТ, в количестве 0,1% — адгезионная прочность повышается с 2,0...2,2 до 2,9...3,1 МПа при изменении характера отрыва лакокрасочного покрытия от подложки с полностью адгезионного (100%) на адгезионно-когезионный. Оценка химической стойкости наноструктурированных лакокрасочных покрытий с УНТ показала, что при воздействии на покрытия раствора гербицида, они сохраняют свою массу вне зависимости от концентрации модификатора, при этом их твердость не изменяется (Н). Оценка равновесной степени набухания

позволила определить, что лакокрасочные покрытия с наночастицами оксида висмута и УНТ обладают устойчивостью к растворителям и приобретают более сетчатую структуру по отношению к покрытиям без добавок. Сканирование различных участков поверхностей образцов на АСМ показало, что при модификации нанодобавками оксида висмута и УНТ поверхность становится более гладкой, нанометровая шероховатость уменьшается с 50...60 до 20...30 нм. Более существенное уменьшение выступов наблюдается у покрытий модифицированных УНТ, что говорит об упрочнении покрытия за счет уменьшения размеров углублений и пор.

**Ключевые слова:** наноматериалы; наноструктурированные покрытия; лакокрасочные покрытия и материалы; адгезионная прочность; химическая стойкость; металлические конструкции; углеродные нанотрубки; оксид висмута.

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#### 1. Introduction

The surfaces of various metal products and structures operating in industrial environments are constantly exposed to aggressive action of various environments: acids, alkalis, fuels and lubricants, salt solutions, etc. Thus, one of the most important properties of protective coatings of metal surfaces is chemical resistance. The use of chemical-resistant protective coatings is widespread in the oil and chemical industries, and is also relevant, for example, for anti-corrosion protection of structures at mineral fertilizer production plants [1–3].

The need to address the issues of developing chemical-resistant protective coatings for metals is explained by a number of factors [1, 3–5]:

- corrosion damage (active exposure to liquids and gases in chemical and petrochemical plants, under the influence of severe temperature loads, negatively affects the condition of metal structures, pipes, tanks and other equipment);
- economic losses caused by corrosion damage, leading to the need to replace or repair parts and equipment;
- threat to human safety, including in the event of damage to bridges, oil pipelines, aviation equipment;
- negative impact on the environmental system and human health due to uncontrolled destruction, which can lead to leaks of hazardous substances into the environment.

Hence, the use of chemically resistant coatings can increase the service life of individual elements, reduce the costs of repair work and reduce the likelihood of the development of corrosion changes inside structures.

One of the most effective methods for improving technological and operational characteristics of materials is their nanomodification, which has become the basis for this study and was discussed in the works of many researchers, who presented the results of using nanomodifiers to create highly

effective composite materials for various purposes [3–17].

Thus, the study aims to develop nanostructured protective coatings with high chemical resistance for metal surfaces.

#### 2. Materials and Methods

#### 2.1. Materials

Acrylic and pentaphthalic paints and varnishes (AC-182, PF-115 - manufactured by LKZ Kolorit Ltd., Russia) were used in the tests. They are widely used for painting metal products and structures and their choice was justified in other studies [3, 18, 19]. The paints and varnishes were modified using compositions based on carbon nanotubes (CNT) (Tuball Matrix 203 – Russia, OcSiAl), bismuth oxide nanoparticles (BO) (Dongguan SAT nano technology material Co., Ltd. - China), silicon dioxide (SiO<sub>2</sub>) (Tarkosil T-20 - Russia), titanium dioxide (TiO<sub>2</sub>) (Zhengzhou Kelai Chemical Co., Ltd - China), magnesium hydroxide (Mg(OH)<sub>2</sub>) and aluminum hydroxide (Al(OH)<sub>3</sub>) (Zibo Aotai New Material Technology Co., Ltd. - China). The method of dispersing nanomaterials is mechanical mixing [3].

# 2.2. Control methods

The chemical resistance of paint and varnish coatings was assessed in accordance with Russian Standard 9.403-2022. The adhesive strength of paint and varnish coatings was determined in accordance with Russian Standard 32299–2013. The hardness of the coatings was assessed in accordance with Russian Standard R 54586–2011.

Also, one of the simple and reliable methods for determining the chemical resistance, as well as the topological structure of coatings is the equilibrium swelling method or the Flory-Rehner method, based on the swelling of the coating in a solvent, followed by an assessment of the equilibrium swelling rate [16]:

$$\Delta \mu = RT \left( \ln(1 - \varphi) + \varphi + x_1 \varphi^2 + \frac{\rho_2 V}{M_c} \left( \varphi^{1/3} - \frac{2\varphi}{f} \right) \right), (1)$$

where  $\rho_2$  is polymer density, g·cm<sup>-3</sup>; V is volume of absorbed liquid, cm<sup>3</sup>; f is mesh functionality;  $M_c$  is average molecular weight of chain segments;  $x_1$  is polymer-solvent interaction parameter (known for different polymer-solvent systems); RT is the energy required to transfer one molecule of solvent into the polymer;  $\varphi$  is volume fraction of polymer in the swollen sample.

The volume fraction of the polymer in the swollen sample is found using formula (2):

$$\varphi = \frac{m_0/\rho}{m_0/\rho + V_1},\tag{2}$$

where  $V_1$  is volume of solvent absorbed at equilibrium at the end of the experiment, cm<sup>3</sup>.

The main consequences of the calculations in accordance with the Flory-Rehner equation and the tests carried out are [20]:

- mesh polymer swelling is always limited;
- the degree of swelling of a mesh polymer depends on the thermodynamic affinity of the components and the density of the mesh;
- increasing the mesh density leads to a decrease in the degree of swelling.

The tests were carried out by processing the obtained data by calculating the equilibrium swelling rate of specimens, excluding the estimation of the mesh density parameter. Indicators for comparing the test results of the specimens were carried out by means of formulas 3 and 4, according to which the equilibrium swelling rate is calculated.

$$\alpha = \frac{m - m_0}{m_0} 100 \%; \tag{3}$$

$$\alpha = \frac{V \rho}{m_0} 100 \%, \tag{4}$$

where m is weight (volume) of swollen specimen, g;  $m_0$  is weight (volume) of the initial specimen, g; V is volume of absorbed liquid,  $cm^3$ ;  $\rho$  is solvent density,  $kg \cdot m^{-3}$ .

According to the equilibrium swelling method, the specimens were exposed in the solvent and the changes in the parameters of mass, thickness, length and width of the specimens were monitored at regular intervals. The tests were continued until the

equilibrium state was reached, when the parameters became unchanged. Based on the Flory-Rehner theory, an increase in the mesh density leads to a decrease in the degree of swelling, i.e. the smaller the equilibrium swelling rate, the more mesh-like the coating structure.

#### 2.3. Analytic methods

The study of the linear dimensions of the elements of the micro- and nanorelief surface structures of the nanomodified coatings was carried out using an atomic force microscope (AFM) based on the INTEGRA Aura probe nanolaboratory platform of NT-MDT company. A semi-contact (intermittent-contact) method was used to produce images; to increase the goodness of cantilever and, consequently, to increase the oscillations sensitivity, reliability, and validity the measurements of weak forces between the probe and the specimen, the study was carried out under low vacuum conditions.

#### 3. Results and Discussion

When determining the effect of nanomaterials on the physical and mechanical properties of the resulting coatings, it was found that the best adhesion strength has coatings based on acrylic paint material (AC-182) than on the basis of pentaphthalic (PF-115). At the same time, the introduction of nanomaterials contributes to an increase in the adhesive strength of the coating. So, the best effect is determined by the introduction of CNTs, in the amount of 0.1 % – the adhesive strength increases from 2.0–2.2 to 2.9–3.1 MPa with a change in the nature of detachment of the paint coating from the substrate from fully adhesive (100 %) to adhesion-cohesive (Table 1).

Tests on the chemical resistance of paint coatings were carried out by exposing paint coatings to various chemical media (solvents, fuels and lubricants, acids, alkalis, etc.). At the first stage of the research rational concentration of chemical solutions was determined. So, for example, 40 % aqueous solution of herbicide (a mixture of dimethylamine, potassium, sodium salts) was chosen, at which changes in the properties of coatings occur, at which it is possible to most adequately trace the different effects of chemical solution on the coating. Nanostructured paint coatings based on AS-182 enamel showed the greatest chemical resistance, the results of the studies of which are presented in Table 2.

**Table 1.** Results of adhesive strength of paint and varnish coatings

Introduced additives	Adhesion strength, MPa / Detachment character (adhesive (A) - cohesive (K			
introduced additives	AC-182	PF-115		
No additives	1.7 – 1.9 / A100 – K0	1.6 - 1.8 / A100 - K0		
BO – 0.5 %	2.6 - 2.8 / A30 - K70	2.4 - 2.6 / A30 - K70		
BO – 1 %	2.9 - 3.1 / A10 - K90	2.5 - 2.7 / A10 - K90		
BO - 2 %	2.4 - 2.6 / A30 - K70	2.3 - 2.5 / A20 - K80		
$SiO_2 - 0.5 \%$	2.0 - 2.2 / A80 - K20	1.9 - 2.1 / A80 - K20		
SiO <sub>2</sub> – 1 %	2.3 - 2.5 / A80 - K20	2.1 - 2.2 / A80 - K20		
SiO <sub>2</sub> – 2 %	2.0 - 2.2 / A80 - K20	1.9 - 2.1 / A20 - K80		
CNTs - 0.01 %	2.7 - 2.9 / A90 - K10	2.4 - 2.6 / A70 - K30		
CNTs − 0.05 %	2.9 - 3.1 / A90 - K10	2.8 - 3.0 / A70 - K30		
CNTs − 0.1 %	3.2 - 3.4 / A50 - K50	3.0 - 3.2 / A75 - K25		
$Mg(OH)_2 - 0.5 \%$	1.8 - 2.0 / A100 - K0	1.6 - 1.8 / A90 - K10		
$Mg(OH)_2 - 1 \%$	2.0 - 2.2 / A80 - K0	1.7 - 1.9 / A100 - K0		
$Mg(OH)_2 - 2\%$	1.9 - 2.1 / A90 - K10	1.8 - 2.0 / A100 - K0		
Al(OH) <sub>3</sub> – 0.5 %	1.6 - 1.8 / A100 - K0	1.7 - 1.9 / A100 - K0		
Al(OH) <sub>3</sub> – 1 %	1.7 - 1.9 / A100 - K0	1.6 - 1.8 / A90 - K10		
Al(OH) <sub>3</sub> – 2 %	1.5 - 1.7 / A100 - K0	1.7 - 1.9 / A100 - K0		

**Table 2.** Chemical resistance of nanostructured coatings based on enamel AC-182 when exposed to herbicides

		Befo	ore testing		Aft	er testing
Additive type	Weight, g	Hardness	A micrograph specimen (X230)	Weight, g	Hardness	A micrograph specimen (X230)
1	2	3	4	5	6	7
No additives	2.173	НВ		2.159	В	
CNTs – 0.01 %	2.104	Н		2.104	Н	
CNTs – 0.05 %	2.063	Н		2.063	Н	

# Continuation of Table 2

1	2	3	4	5	6	7
CNTs – 0.1 %	2.116	2Н		2.115	2Н	
Mg(OH) <sub>2</sub> – 0.5 %	2.169	Н		2.162	Н	
Mg(OH) <sub>2</sub> – 1 %	2.189	Н		2.186	НВ	
Mg(OH) <sub>2</sub> – 2 %	2.113	Н		2.108	НВ	
Al(OH) <sub>3</sub> – 0.5 %	2.150	2Н		2.143	2Н	
Al(OH) <sub>3</sub> – 1 %	2.138	2Н		2.136	2Н	
Al(OH) <sub>3</sub> – 2 %	2.127	3Н		2.120	2Н	

Continuation of Table 2

1	2	3	4	5	6	7
SiO <sub>2</sub> – 0.5 %	2.084	Н		2.080	Н	
SiO <sub>2</sub> – 1 %	2.126	2Н		2.120	Н	
SiO <sub>2</sub> – 2 %	2.160	2Н		2.151	Н	
BO – 0.5 %	2.191	2Н		2.190	Н	
BO – 1 %	2.166	2Н		2.166	Н	
BO – 2 %	2.132	2Н		2.132	Н	

Table 2 presents data on chemical resistance of paint coatings with different additives before and after exposure to herbicide. The results of data analysis revealed that the mass of the specimen without additives decreased by 0.014 g, also the coating became softer, as evidenced by the change in hardness from HB to B. Nanostructured paint

coatings with carbon nanotubes retain mass according to the test results, and regardless of the concentration of the additive.

At the same time the hardness of such coatings does not change (H), which indicates high chemical stability of CNTs nanostructured coating. Also modification of the paint material with bismuth oxide

nanoparticles leads to chemically stable coatings, as evidenced by the mass retention of the specimens and insignificant change in their hardness from 2H to H.

According to the results of studies of the equilibrium swelling rate (Tables 3, 4, Fig. 1), it was also determined that the paint coating modified with

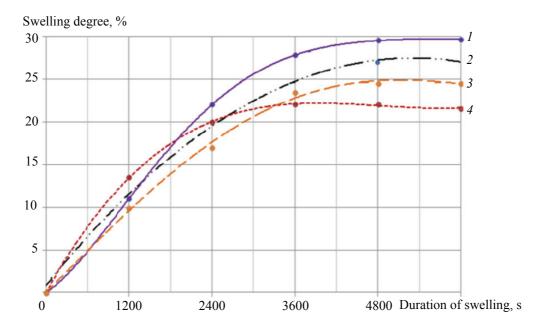
bismuth oxide nanoparticles and carbon nanotubes has resistance to solvents, and also acquires a more reticulated structure in relation to coatings without additives, which also depends on the amount of introduced additives.

**Table 3.** Results of tests of nanostructured coatings by the swelling method. Part 1

Specimen	Soaking time of specimens in solvent, min	Change in specimen weight, g	Change in specimen thickness, mm
	0	0.47	1.13
	20	0.57	1.21
No additives	40	0.60	1.23
	60	0.63	1.23
	80	0.63	1.24
	0	0.45	1.03
	20	0.55	1.12
CNTs 0,05 %	40	0.59	1.17
	60	0.60	1.17
	80	0.60	1.17
	0	0.51	1.23
	20	0.62	1.30
CNTs 0,1 %	40	0.67	1.33
	60	0.69	1.35
	80	0.69	1.35
	0	0.59	1.33
	20	0.70	1.40
TiO <sub>2</sub> 0.5 %	40	0.78	1.45
	60	0.80	1.47
	80	0.81	1.47
	0	0.63	1.56
	20	0.69	1.64
TiO <sub>2</sub> 1 %	40	0.77	1.66
2	60	0.79	1.75
	80	0.81	1.81
	0	0.64	1.46
	20	0.74	1.535
BO 0.5 %	40	0.79	1.615
	60	0.82	1.72
	80	0.84	1.72
	0	0.42	1.0
	20	0.5	1.03
BO 1 %	40	0.53	1.07
	60	0.54	1.1
	80	0.56	1.1

**Table 4.** Results of tests of nanostructured coatings by the swelling method. Part 2

Specimen	Soaking time of specimens in solvent, min	Change in specimen length, mm	Change in specimen width, mm	Volume equilibrium swelling rate ( $\alpha$ )
	0	26.30	9.65	
	20	27.05	9.90	
No additives	40	27.60	10.20	0.27
	60	27.80	10.35	
	80	28.20	10.40	
	0	26.70	9.55	
	20	27.30	9.80	
CNTs 0.05 %	40	27.90	10.50	0.22
	60	27.90	10.50	
	80	27.90	10.50	
	0	26.50	9.68	
	20	27.40	9.77	
CNTs 0.1 %	40	27.50	10.20	0.21
	60	27.50	10.30	
	80	27.50	10.30	
	0	26.30	9.83	
	20	26.60	10.25	
TiO <sub>2</sub> 0.5 %	40	27.65	10.55	0.27
1102 0.0 /0	60	27.70	10.80	
	80	27.70	10.80	
	0	26.60	9.58	
	20	27.50	9.64	
TiO <sub>2</sub> 1 %	40	28.20	9.81	0.29
<del>-</del>	60	28.30	10.00	
	80	28.50	10.00	
	0	26.55	10.10	
	20	27.45	10.30	
BO 0.5 %	40	27.70	10.45	0.24
	60	27.80	10.60	
	80	28.00	10.60	
	0	26.70	10.18	
	20	26,8	10,82	
BO 1%	40	27,00	11,00	0.24
	60	27,6	11,05	
	80	27,6	11,15	



**Fig. 1.** The degree of swelling of modified acrylic protective coatings, depending on the duration of swelling:  $I - \text{modification TiO}_2$  1 %; 2 - without modifier;  $3 - \text{Bi}_2\text{O}_3$  1 % modification; 4 - CNTs modification 0.1 %

When bismuth oxide was added to the enamel, the meshiness of the coating increased, as indicated by the volume equilibrium swelling rate, which decreased from 0.27 to 0.24 at 0.5 and 1 % additives.

A more significant effect on the formation of dense molecular bonds was exerted by adding CNTs into the composition, and the index of the volume equilibrium swelling rate decreased from 0.27 to 0.22 at 0.05 % CNTs and to 0.21 at 0.1 % CNTs in the enamel composition.

The results of studies on the example of modifiers introduction into the paint material showed that nanomodification leads to the formation of denser mesh structures of coatings, which explain the increase in their physical and mechanical properties (adhesion strength, hardness, etc.) (Tables 1, 2) [3, 18, 19].

Scanning of various surface areas of specimens of nanostructured paint coatings, before chemical action on them, on the atomic force microscope showed that the surface becomes smoother nanometer roughness decreases from 50–60 nm (Fig. 2a) to 20–30 nm (Figs. 2b, c) when modified with bismuth oxide and carbon nanotubes. Moreover, a more significant reduction of protrusions is observed in CNT-modified coatings.

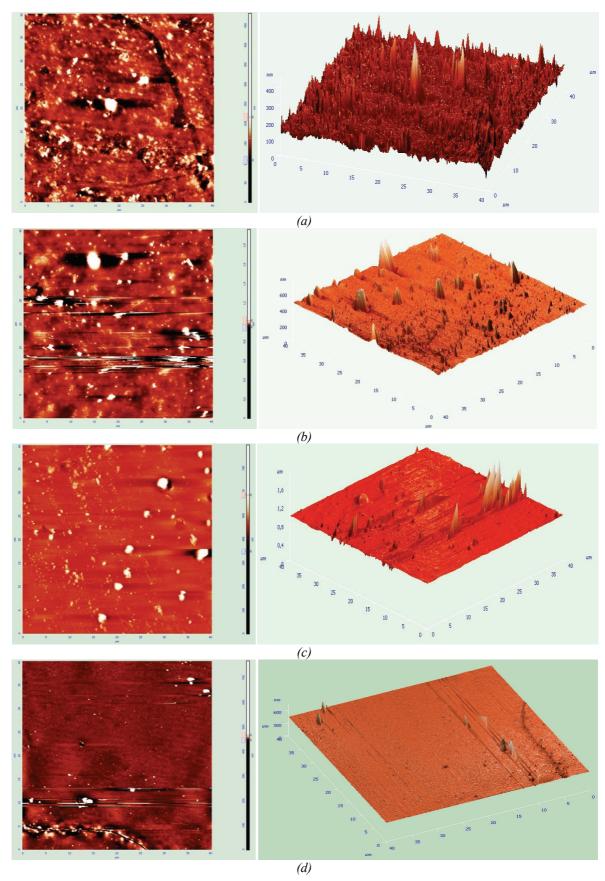
At the same time, at the joint introduction of bismuth oxide nanoparticles and carbon nanotubes, the number and size of protrusions are minimized and the *Ra* parameter decreases to 10–15 nm (Fig. 2*d*), which indicates a strengthening effect of the joint introduction and, consequently, hardening of the

coating by reducing the size of depressions and pores [3].

Thus, microstructural analysis allowed us to prove the existence of the optimal content of nanoadditives to enhance the quality parameters of protective paint coatings.

#### 4. Conclusion

According to the results of this study it was found that by using nanoscale materials it is possible to produce effective nanostructured protective coatings capable of reliably protecting metal surfaces operating in a variety of aggressive chemical environments. It is most effective to apply nanostructured coatings when adding CNT-based compositions and bismuth oxide nanoparticles. It is more preferable to use acrylic paint materials to obtain chemoresistant nanostructured protective coatings. Thus, adding CNTs and bismuth oxide, the resulting coating has high adhesive strength (more than 3 MPa at cohesive detachment character up to 50% and higher), and also increased chemical resistance that is expressed in decrease of coating equilibrium swelling rate (from 0.27 to 0.21), preservation of specimen weight and its hardness (2H-H) caused by chemical media exposure. The increase in chemical resistance of such coatings is justified by the fact that they have a more hardened mesh structure with a minimum size of recesses and micropores in the surface.



**Fig. 2.** AFM-images of nanomodified coatings: a – without additives, b – BO 1 %, c – composition with CNTs 0.1 %, d – BO 1 % + composition with CNTs 0.1 %

## 5. Supplementary Material

A number of the data presented in this study are openly available at [https://gos\_att.bstu.ru/shared/attachments/272578].

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#### 7. Conflict of interest

The authors declare no conflict of interest.

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