

# Obtaining abrasives on the base of composite Ni-P coatings for surface treatment of rock materials

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**Abstract:** The electroless deposition of Ni-P coatings is well known classic method for production of metal coatings on polymers. The addition of dispersed particles of different types and sizes to the base solution for electroless deposition significantly improves the properties of the coating by increasing its wear resistance and micro-hardness. Thereby, the metallized polymers find new and different applications. By using a device, designed and developed in IPC-BAS, tribological tests of composite Ni-P coatings deposited on substrate of polyethylene terephthalate (PET) are performed. Three types of dispersed particles (diamond (D), BN and SiC) are co-deposited in the Ni-P coatings. Their application as abrasive material for surface treatment of rock materials is evaluated. The structure, morphology and elemental composition of Ni-P coatings are characterized by SEM and EDS.

**KEYWORDS:** COMPOSITE ELECTROLESS NI-P COATINGS, PET, DISPERSED PARTICLES (D, BN, SiC), SEM, EDS.

## 1. Introduction

The electroless deposition of Ni-P coatings is a method for surface modification of materials of different types and compositions. The coatings have a number of advantages, such as: good corrosion and wear resistance, hardness and evenly distribution, regardless of the complexity of the detail. The co-deposition of a non-metallic element (P) largely determines the structure and hence the properties of these coatings [1, 2].

Improvement of the basic properties of the electroless Ni-P coatings is achieved by addition of dispersed particles of different types and sizes to the base solution and hence, composite coatings are deposited [3-8].

The most commonly used dispersoid are synthetic diamond particles. They are cubic or octahedral crystals. A significant number of studies are devoted to the preparation of composite Ni-P coatings containing diamond particles of different sizes (Ni-P/D). The comparison with the Ni-P coatings shows better properties in terms of micro-hardness, wear resistance, abrasiveness, corrosion protection, etc. [3, 7, 9, 10].

Boron nitride (BN) is not present in nature as a mineral. Three of its crystal modifications are known: alpha modification (denoted as hBN due to the hexagonal graphite-like structure), beta modification with diamond-like cubic crystal structure of a sphalerite type (denoted as cBN) and gamma-BN with hexagonal crystal structure (wurtzite type). The last two modifications show hardness close to that of a diamond. The modification hBN is used as a solid lubricant. It is embedded into composite materials and the produced details show antifriction properties [3, 11].

Carbides are difficult to melt and insoluble solids. As a typical representative of covalent carbides, SiC (also called carborundum) is characterized by high hardness (about 9 on the Mooc scale), high melting temperature and chemical inertness. The addition of SiC particles as a second phase to the electroless coatings has been shown to be one of the methods for improving their basic characteristics (micro-hardness and corrosion resistance) [12-14].

Common between the three types of particles is that they find application in obtaining tools for abrasive treatment of different materials [15, 16].

The aim of the present study is to obtain composite Ni-P coatings with the three types of particles (D, BN and SiC) on a flexible substrate of PET, and to study their possible application for surface treatment of rock materials. Polyethylene terephthalate (PET) is a linear homo-polymer with a dominant composition of polyester fibers. It can be heated and processed into various forms, such as fibers, sheets, etc. [17, 18].

## 2. Experimental

Non-woven cloth of PET subjected to reinforcement by additional pressing was used as a substrate in these investigations. The samples were pre-treated employing the following technological scheme [19]: etching in alkaline solution OA-84-2 (a commercial product of Technical University-Sofia)

at  $T = 65\text{ }^{\circ}\text{C}$  and  $\tau = 15\text{ min}$ ; pre-activation in 3M HCl at  $T = 25\text{ }^{\circ}\text{C}$  and  $\tau = 5\text{ min}$ ; activation in colloidal solution of  $\text{PdCl}_2 \cdot \text{A-75-12}$  (a commercial product of Technical University-Sofia) at  $T = 25\text{ }^{\circ}\text{C}$  and  $\tau = 5\text{ min}$ ; acceleration in alkaline solution X-75-4 Accelerator (a commercial product of Technical University-Sofia) at  $T = 25\text{ }^{\circ}\text{C}$  and  $\tau = 5\text{ min}$ .

After the above pre-treatment, the samples were immersed in the solution for electroless nickel plating [18], presented in Table 1.

**Table 1.** Composition of the solution and working conditions

Composition of the solution	
$\text{NiSO}_4 \cdot 7\text{H}_2\text{O}$ , $\text{g l}^{-1}$	25.0
$\text{NaH}_2\text{PO}_4 \cdot 2\text{H}_2\text{O}$ , $\text{g l}^{-1}$	22.0
$\text{CH}_3\text{COONa}$ , $\text{g l}^{-1}$	20.0
Lactic acid (80 %), $\text{g l}^{-1}$	20.0
Stabilizer 2, $\text{mg l}^{-1}$	1.0
Working conditions	
pH	4.6 – 4.8
$T$ , $^{\circ}\text{C}$	82-85

As dispersoids were used particles of: diamond with dimensions of  $3/7\text{ }\mu\text{m}$  (corresponding to a mixture of D particles sized from 3 to  $7\text{ }\mu\text{m}$ ) and  $63/75\text{ }\mu\text{m}$ ; cBN –  $63/80\text{ }\mu\text{m}$  and hBN  $1/5\text{ }\mu\text{m}$ ; SiC –  $7/10\text{ }\mu\text{m}$  and  $60/70\text{ }\mu\text{m}$ . Prior to addition to the solution, all particles were subjected to preliminary treatment in aqueous solution of sodium laurylsulphonate (SLS) with concentrations of  $0.01\text{ g l}^{-1}$  at room temperature.

The composite coatings were deposited with different time duration to ensure the incorporation of the different sized particles. For more evenly distribution, the solution was stirred by air flow agitation ( $100\text{ ml min}^{-1}$  for 250 ml solution) under different modes, respectively noted in the experimental results.

The mass of the deposited nickel coating ( $\Delta m$ ) was determined gravimetrically – based on the difference in the weights of the samples prior and after the deposition of the coating.

The morphology and the structure of the coatings, as well as the particles' distribution over their surface were studied by means of scanning electron microscopy (SEM) (JSM 6390, Japan). The average amount of co-deposited particles ( $N\text{ cm}^{-2}$ ) was determined by the counting in three arbitrarily chosen zones at a magnification 200x.

A device, designed and manufactured at IPC-BAS was used to perform tribological tests [20]. The specimens with a diameter of 7 cm were fixed on a support disk, flat-parallel to the surface of the counter body of limestone. The load on the working disk was 1 kg; the speed of rotation 1000 rpm, and a successive change of the disks in the direction from larger to smaller size of the dispersed particles. The working time of the every disk was 1 min.

The roughness parameters  $R_a$  and  $R_z$  were measured by a profilograph-profilometer Perthometer C3A (Dr. Ing. Perthometer GmbH).

### 3. Results and discussion

#### 3.1. Conditions for obtaining composite Ni-P coatings on PET

In our previous papers [8, 15, 16], the influence of the concentration of the dispersoid in the solution, the deposition time and the hydrodynamic regime on the inclusion of the particles, as well as their adhesion to the coating were studied. In this section are presented only the conditions for obtaining the composite Ni-P coatings with which the tribological tests are performed.

All composite coatings were deposited at a dispersoid concentration of  $5.0 \text{ g l}^{-1}$  regardless of the particle size.

Data on the mass of composite coatings and the number of the incorporated particles during 1 h deposition time are given in Table 2.

**Table 2.** Influence of the type and the size of the particles on the Mass of the composite coating ( $\Delta m$ , g) and number of deposited particles ( $N/\text{cm}^2$ ) at a deposition time of 1 h.

	D	D	hBN	cBN	SiC	SiC
$\mu\text{m}$	3/7	63/75	1/5	63/80	7/10	60/70
$\Delta m$ , g	0.3937	0.4355	0.4360	0.2964	0.5014	0.4739
$N/\text{cm}^2$	-	2200	-	11600	-	1200

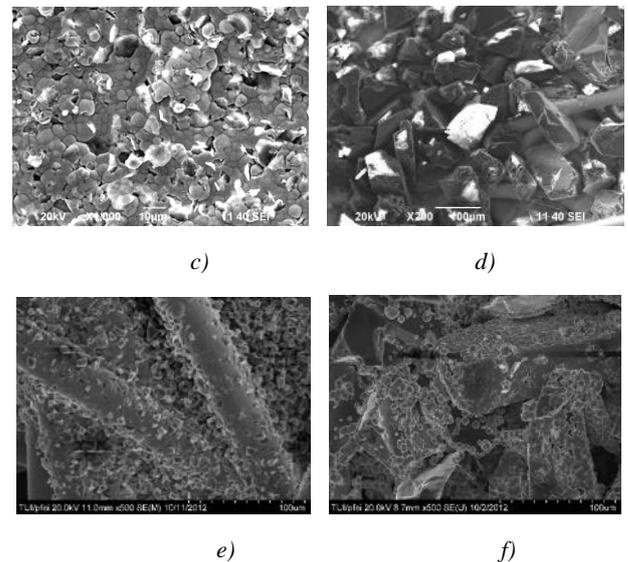
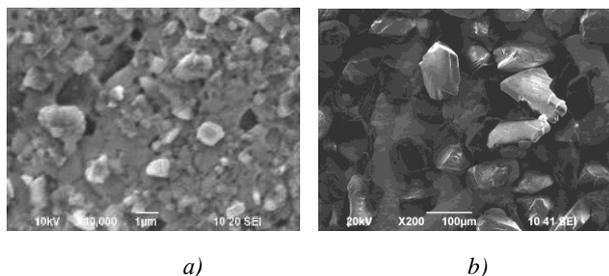
For the small particles, the time of stay of the specimens in the solution of 1 h is quite sufficient to obtain coatings with good characteristics. No attempt is made to estimate their number because of their small size and the coverage of some of them by Ni-P coating. For the larger particles, the composite coating deposited at that time has a mass insufficient to obtain good attachment of the particles to the coating. Therefore, for composite coatings with larger particles, the time of deposition of 5 h is more appropriate (Table 3).

**Table 3.** Influence of the type of the particles on the mass of the composite coating ( $\Delta m$ , g) and number of the deposited particles ( $N/\text{cm}^2$ ) at a deposition time of 5 h.

	D 63/75 $\mu\text{m}$	cBN 63/80 $\mu\text{m}$	SiC 60/70 $\mu\text{m}$
$\Delta m$ , g	0.7937	0.7027	0.5292
$N \text{ cm}^{-2}$	222600	240500	90000

Composite Ni-P coatings with small particles were deposited by constant air stirring. This hydrodynamic regime is not suitable for large particles. An appropriate interrupted hydrodynamic regime for the large particles was turned out to be 2 min of stirring and 10 min without stirring according to [16, 20].

SEM images of Ni-P coatings obtained on PET under the selected composition and operating conditions of the nickel solution (Fig. 1).



**Fig.1.** SEM images of composite Ni-P coatings deposited in the presence of: Diamond particles - a) 3/7  $\mu\text{m}$  and b) 63/75  $\mu\text{m}$ ; c) hBN particles 1/5  $\mu\text{m}$ ; d) cBN 63/80  $\mu\text{m}$ ; SiC particles - e) 7/10  $\mu\text{m}$  and f) 60/70  $\mu\text{m}$ .

#### 3.2. Tribological testing of samples with different dispersoids

The object of treatment is the limestone material with dimensions  $10 \times 10 \times 10 \text{ mm}$ .

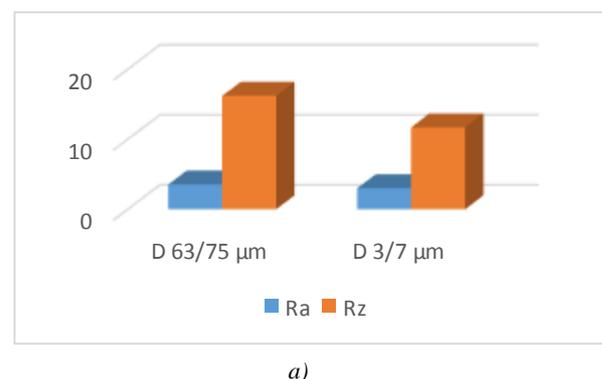
The change in the mass ( $\Delta m$ ) of the limestone samples before and after treatment with the tested disks is shown in Table. 4.

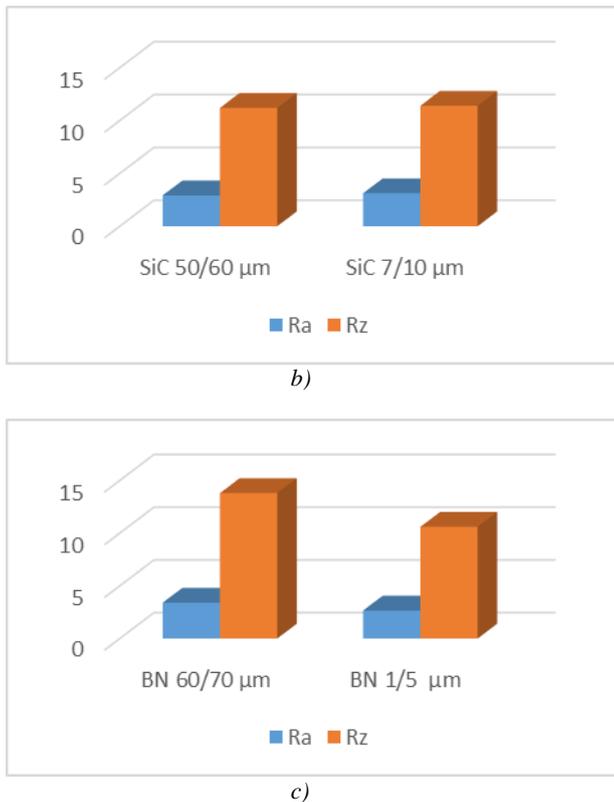
**Table 4.** Influence of the type and size of the dispersoid in the composite coating on the loss of limestone mass (1000 rpm/1 min/1 kg).

Type of the dispersoid	$\Delta m$ , g
D 3/7 $\mu\text{m}$	0.008
D 63/75 $\mu\text{m}$	0.067
SiC 7/10 $\mu\text{m}$	0.001
SiC 60/70 $\mu\text{m}$	0.056
hBN 1/5 $\mu\text{m}$	0.001
cBN 63/80 $\mu\text{m}$	0.011

The results show that larger particles take away more mass, with the strongest effect in the case of diamond particles

The abrasive effect of the composite coatings is determined by the change of the values of the parameters characterizing the surface roughness ( $R_a$  and  $R_z$ ) (Fig. 2) and the weight loss (Table 4).





**Fig. 2:** Change of the roughness parameters  $R_a$  and  $R_z$  after treatment of the limestone samples with disks with different types and sizes of the particles: a) Diamond particles; b) SiC; c) BN. Values of  $R_a$  and  $R_z$  before treatment -  $3.96 \mu\text{m}$  and  $17.6 \mu\text{m}$ , respectively.

#### 4. Conclusions

Composite Ni-P coatings with three types of particles - diamond (D), boron nitride (BN) and silicon carbide (SiC) of different sizes are deposited on a flexible substrate of polyethylene terephthalate (PET). The deposited samples are characterized by good adhesion of the particles to the coating and to the substrate. Tribological studies show that composite coatings are promising in terms of their use in abrasive tools for surface treatment of rock materials. The next step would be to optimize the tribological conditions - load on the abrasive disk and duration of the treatment.

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#### 5. References

- G. Gavrilov, Ts. Nikolov, "Chemical nickel plating and disperse coatings", (1985).
- Hr. Petrov, "Galvanization of plastics", (1982).
- J.N. Balaraju, T.S.N. Sankara Narayanan, S.K. Seshadri, J. Appl. Electrochem. 33, 807-816 (2003).
- A. Sharma, A. K. Singh, J. Mater. Eng. Perform, 22, 176-183 (2013).
- V.V.N. Reddy, B. Ramamoorthy, P. Kesavan Nair, Wear, 239, 111-116 (2000).
- V. Chakarova, M. Georgieva, M. Petrova, Special Issue of Bulg. Chem. Commun., 49 (F), 30-36 (2017).
- H. Xu, Z. Yang, M.-Ke Li, Y.-Li Shi, Y. Huang, H.-Lin Li, Surf. Coat. Technol, 191, 161-165 (2005).
- M. Petrova, M. Georgieva, V. Chakarova, Ek. Dobрева, Arch. Metall. Mater., 61 (2), 493-498 (2016).
- Y.L. Shi, Z. Yang, H. Hu, M.K. Li, H.L. Li, J. Mater. Sci., 39, 5809-5815 (2004).
- H. Mazaheri, S. Reza Allahkaram, Appl. Surf. Sci., 258, 4574-4580 (2012).
- O.A. Leoh, M.H. Staila, H.E. Hintermann, Surf. Coat. Technol., 108-109, 461-465 (1998).
- M. Islam, M.R. Azhar, N. Fredj, T. David Burleigh, Surf. Coat. Technol., 236, 262-268 (2013).
- Y.S. Huang, X.T. Zeng, X.F. Hu, F.M. Liu, Electrochim. Acta, 49, 4313-4319 (2004).
- M. Islam, M.R. Azhar, Y. Khalid, R. Khan, H.S. Abdo, M.A. Dar, O.R. Oloyede, T. David Burleigh, J. Mater. Eng. Perform, 24, 4835-4843 (2015).
- M. Georgieva, M. Petrova, Ch. Jakob, M. Fritz, V. Chakarova, WoMag, 10 (3), 26-27 (printed, short version) and 6 pages (on-line) (2014).
- V. Chakarova, M. Georgieva, M. Petrova, E. Dobрева, D. Stoychev, Trans. Inst. Metal Finishing, 94 (5), 269-264 (2016).
- J. Karl Fink, Book Chapter, 347-390 (2008).
- M. Georgieva, M. Petrova, V. Chakarova, Bulg. Chem. Commun., 45 A, 116-121 (2013).
- BG Patent № 65604 (2009).
- D. Stoychev, E. Dobрева, N. Razkazov, M. Stoycheva, N. Koteva, Bulg. Chem. Commun., 46 (2), 283 (2014).