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INFLUENCE OF NANO-DIAMOND PARTICLES ON THE TRIBOLOGICAL CHARACTERISTICS OF NICKEL CHEMICAL COATINGS

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Abstract: *Friction and wear of 10 types Ni chemical coatings, with and without heat treatment, containing nano-diamond particles of various size – 0;5 nm; 100 nm; 200 nm and 250 nm, are studied in the paper. Procedure and laboratory device for friction investigation in starting regime were developed. Experimental results for the influence of the particle size on the static friction force and the change of friction coefficient have been obtained. Abrasive wear has been studied by means of the procedure developed by the authors for the study of above coatings under conditions of dry friction on surfaces with fixed abrasive. The obtained results are related to the parameters linear wear, wear rate and wear-resistance. A part of this study is connected with the tasks on the 7 FP Project „Acom In (Advanced Computing Innovations)" coordinated by the Institute of Information and Communication Technologies at the Bulgarian Academy of Sciences, and the other part is carried out under the Project ДУНК-01/3 "University R&D Complex for innovation and transfer of knowledge in micro/nano-technologies and materials, energy efficiency and virtual engineering" funded by the Bulgarian Ministry of Education and Science.*

Keywords: *tribology, nano-diamond particles, coatings, friction, wear*

1. INTRODUCTION

Ni chemical coatings are obtained through the method of electro-less chemical deposition known in the literature as „Electroless Nickel".

From chemical point of view, chemical deposition is a deoxidization process which develops between positive charged metal ions M^{z+} and negative electrons *e*:

$$M_e^{z+} + ze \rightarrow Me \quad (1)$$

where *z* is the valence of the metal ion.

Coatings obtained through chemical deposition differ in the methods for procurement of the electrons necessary for the deoxidization.

In the galvanic (electrolyte) methods, electric current is passed through the solution of the metal salt (electrolyte) and the metal ions are reduced to the corresponding metal atom *Me* on the cathode (the coated detail). The cathode renders, and the anode obtains electrons, which are provided by external source – the electric current.

At chemical Ni deposition an external source is not needed for providing electrons. The necessary electrons are obtained as a result of chemical reactions going between the solution and the surface of the detail to be coated. As a consequence the Ni metal ions of the solution obtain a given number of electrons depending on their valence passing thus in state of neutral atoms (*Me*). The atoms gradually build the crystal grid of the coating. In this case, the role of „supplier of electrons" is realized by different substances (chemical agents) called *reducers (deoxidizers)* from the solution [1].

Imbedding of micro- or nano-sized particles of various natures in the Ni matrix changes the physico-mechanical and the tribological characteristics of the coatings.

In connection with the improvement of the resource of tribosystems, a special interest for nanotribology represent Ni chemical coatings containing in their structure particles of the nanosize scale [2,3]. Imbedding of nano-sized particles in the solution for the production of the Ni coating brings changes in the character of the contact interactions on three levels: interaction of nanoparticles with Ni ions into the solution with the electrons, interaction of the built atom with the surface of the detail and formation of the crystal grid of the coating [4].

The purpose of the present work is to study some characteristics of contact friction and wear for Ni chemical coatings, without and with nanodiamond particles of different size: 4 nm, 100 nm, 200 nm and 250 nm.

2. NICKEL CHEMICAL COATINGS

Ten types of coatings are studied, gathered in 5 series with number given in Table 1.

Table 1: Description of the specimens with coatings of chemical Ni containing nanodiamond particles

№ of series	Designation of the series	№	Designation of the coating	Composition of the coating	Thickness of the coating before wear, h_1, µm
I	N	1	N-	Ni	25,56
		2	N+	Ni+T°C	11,28
II	nD4	3	nD4-	Ni+Di 4 nm	23,22
		4	nD4+	Ni+Di 4 nm +T°C	8
III	nD100	5	nD100-	Ni+Di 100 nm	27,94
		6	nD100+	Ni+Di 100 nm +T°C	7,12
IV	nD200	7	nD200-	Ni+Di 200 nm	26,24
		8	nD200+	Ni+Di 200 nm +T°C	9,14
V	nD250	9	nD250-	Ni+Di 250 nm	30,5
		10	nD250+	Ni+Di 250 nm +T°C	8,7

Each series has its designation in Latin letters, correspondingly:

* N - Nickel coating without nanoparticles;
* nD - Nickel coating with diamond nanoparticles;

The number after the letter D indicates the average size of the nanoparticles – 4 nm, 100 nm, 200 nm, 250 nm. Each series includes two groups of coatings: first group - coatings without heat treatment designated by the sign (-) and second group - with heat treatment at 360°C during 6 hours designated by the sign (+).

3. ABRASIVE WEAR

3.1 Device and procedure

Experimental study of abrasive wear of Ni coatings is realized by means of the test rig TABER ABRASER according to the kinematical scheme „disk-on-disk" (Fig.1).

The specimen 1 (the body) with deposited coating 2 is in the shape of disk and is fixed appropriately on carrying horizontal disk 3 drived by electrical motor 4 with a constant rotational speed $\omega = 1[s^{-1}]$=const. The counter-body 5 is an abrasive disk (roller) of special material CS 10 mounted on horizontal axis 6 in the device 8, by means of which is set the desired normal load *P* in the contact zone *K*. Thus, the body 1 and the counter-body 5 are located on two crossed axes. Because of the constant rotational speed of the body 1 and the constant nominal contact pressure p_a, the friction in the contact zone *K* supports constant speed of rotation of the counter-body 5.

The procedure of the experimental study on abrasive wear is realized in the following sequence of operations:

- clean-up, cleaning of lubricants and drying of the equal specimens. The specimens represent disks of diameter 100 mm and thickness 3 mm with the deposited coatings;
- measuring of roughness of the contact surfaces of the specimens before and after wear;
- measuring of specimens mass m_o before and its mass m_i after a given friction path L by electronic balance WPS 180/C/2 of accuracy 0,1 mg. At every measurement the specimens are cleaned with appropriate solution against static electricity;

- measuring of coating thickness h_1 before wear and h_2 after wear by means of *Pocket LEPTOSKOP 2021 Fe* device in 10 points of the surface; the average value is taken for thickness of the sample;

- the specimen 1 is fixed on the carrying horizontal disk 3; then the normal load P is set. The friction path L is determined by the number of cycles read by the revolution counter 8.

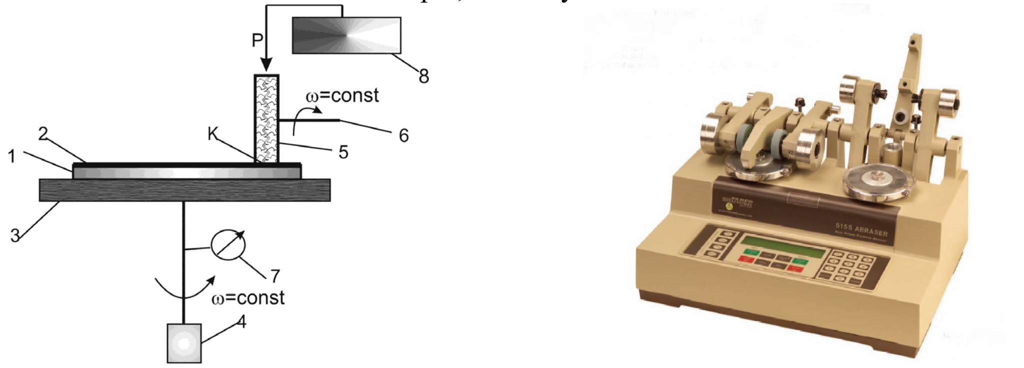

Figure 1. TABER ABRASER – device for study of abrasive wear

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Abrasive wear for all coatings is obtained by fixed equal operating conditions – nominal contact pressure given with the normal load P, average sliding speed V and parameters of the abrasive surface.

The characteristics of the experiment are given in Table 2.

Table 2. Working parameters in the experiment:

Apparent contact area	$A_a = 0,26\ \text{cm}^2$
Nominal contact pressure	$p_a = 9,42\ \text{N/cm}^2$
Average sliding speed	$V = 22,3\ \text{cm/s}$
Abrasive material	CS 10

The parameters of mass and linear wear are studied: speed, wear intensity, absolute and relative wearresistance and their change in time, respectively the friction path.

Wear intensity is determined as mass (or linear) wear for unit friction path, and absolute wearresistance - as the reciprocate value of wear intensity.

The relative wearresistance is the ratio between the absolute wearresistance of the tested coating and the absolute wearresistance of reference sample for equal friction path (number of cycles).

Two reference samples are used in the present work – Nickel coating without nanodiamond particles with heat treatment and without heat treatment.

3.2 Experimental results

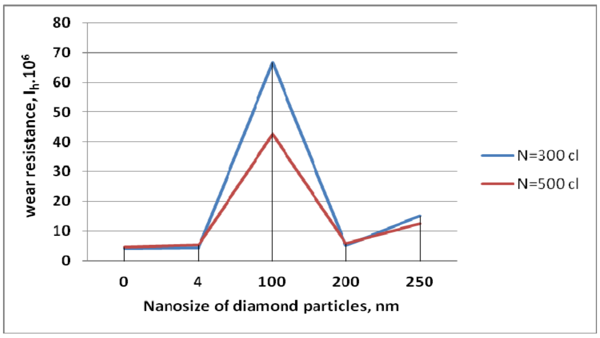

Figure 2. Dependence of wearresistance I_h on nanoparticles size for coatings without heat treatment

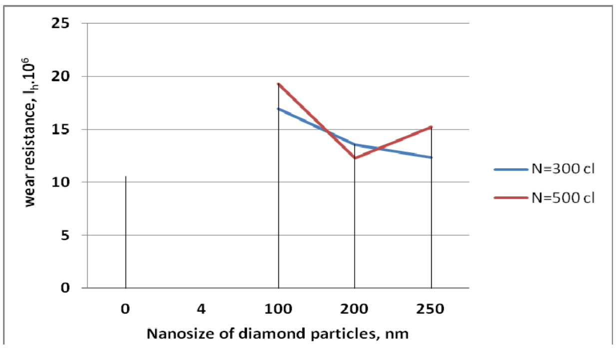

Figure 3. Dependence of wearresistance I_h on nanoparticles size for coatings with heat treatment

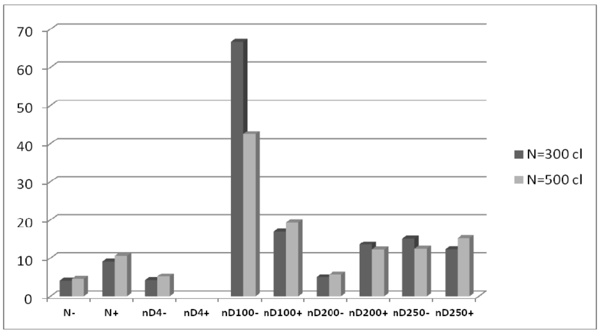

Figure 4. Wearresistance of Ni coatings without and with nanoparticles without and with heat treatment

3.3 Analysis of the experimental results

The presence of nanodiamond particles affects the value and the character of the abrasive wear. This influence becomes more complicated along with the heat treatment of the coating.

For size of nanoparticles 4 nm and 100 nm coatings with heat treatment show higher wear, and for size 200 and 250 nm the opposite effect is observed – wear is lower than that of the case without heat treatment.

The dependence of wear on nanodiamond particles size is of nonlinear character, and the various coatings show different duration of the running-in process.

The boundary number of cycles N*, where the whole coating is worn, is always bigger for coatings without heat treatment.

The highest wearresistance show Ni coatings with nanodiamond particles of the size δ = 100 nm without heat treatment.

4. STARTING CONTACT FRICTION

4.1 Theory

From the point of view of process history, or in time cross-section of the process, tribosystem undergo three friction stages: starting, kinetic and pathological friction. The starting friction, known as static friction in the classical mechanics, is done under conditions of preliminary microdisplacement in the contact zone and the tribosystem performs the transition between static state (at rest) and movement. Kinetic is the friction when the body is moving upon the counterbody.

The kinetic friction matches the stationary and the pathological regimes of contact joint operation.

The pathological friction is characterized by abrupt increase of friction with wear and seizure in contact.

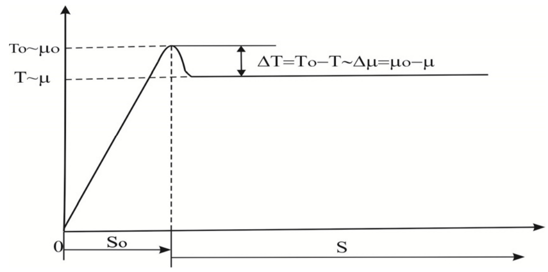

Figure 5. Variation of friction force and friction coefficient with displacement

The difference between starting friction force T_o and sliding friction force T

$$\Delta T = T_o - T \tag{1}$$

gives *the jump in the friction force* during system transition from state at rest and state of movement, and corresponds to the jump in the *friction coefficient*, i.e.

$$\Delta\mu = \mu_o - \mu \tag{2}$$

The work of the starting friction force is given by:

$$A_s\left(\vec{T}_o\right) = T_o S_o = \mu_o P S_o \tag{3}$$

And the work of the kinetic friction force is:

$$A_p\left(\vec{T}\right) = TS = \mu PS \tag{4}$$

Let present the ratio

$$\psi_s = \frac{A_s\left(\vec{T}_o\right) - A_p\left(\vec{T}\right)}{A_s\left(\vec{T}_o\right)}.100 = \frac{\mu_o PS_o - \mu PS}{\mu_o PS_o}.100$$

Then:

$$\psi_s = \frac{\Delta\mu}{\mu_o} = 100,\% \tag{5}$$

The parameter ψ_s is called relative change of the starting friction; it is the ratio between the jump of friction and the starting friction coefficient.

Similarly, for the relative change of the kinetic friction ψ_p is obtained the expression:

$$\psi_p = \frac{A_s\left(\vec{T}_o\right) - A_p\left(\vec{T}\right)}{A_p\left(\vec{T}\right)}.100 = \frac{\mu_o PS_o - \mu PS}{\mu PS}.100 = \frac{\Delta\mu}{\mu}.100$$

or

$$\psi_p = \frac{\Delta\mu}{\mu} = 100,\% \tag{6}$$

4.2 Procedure and experimental results

The parameters of starting friction have been studied using a test rig with functional scheme as shown in Figure 5.

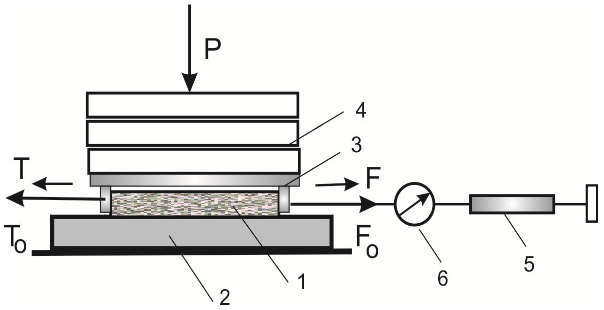

Figure 5. Functional scheme of the test rig for study of starting friction

The experimental arrangement consists of body 1 and counterbody 2, which form a contact. The body 1 is fixed in the holder 3 and is connected through the nonelastic thread with the dynamometer 6 and micrometric screw 5.

Tangential force is loaded on the body 1 near the contact surface through slow turning of the micrometric screw. The normal load P is set by means of the loading bodies 4.

The body 1 is a prismatic sample of size 30 x 50 x 8 mm made of duraluminium (*Al*), and the counterbody 2 represents a round disk of diameter ϕ 100 mm and thickness 3 mm with the deposited coating.

The procedure of measurement the friction force is of following sequence:

- The specimen 2 with coating is fixed in the bed of the base, and the body 1 is mounted in the holder 3, then they are put on the specimen 2.
- The normal load is set by the loading bodies 4.
- The elastic dynamometer 6 is put in the initial reset to zero.
- The micrometric screw 5 is turned very slowly and the pointer of the dynamometer 6 shifts with ease. In the moment of shivering of the pointer backwards, the indication of the dynamometer is read. The maximum value of the indication corresponds to the value of the starting friction force To.
- The screw keeps on turning and the indications of the dynamometer are observed; they match the kinetic friction force T after the jump of friction.
- The dial of the dynamometer is calibrated in force [N].
- During the tests the body 1 is made of the same material but for each test with different coatings a different specimen of this material is used. All specimens of the body have equal size and roughness $Ra = 0,418\ \mu m$.

Table 3 shows the results of starting and kinetic friction, and the figures give some diagrams.

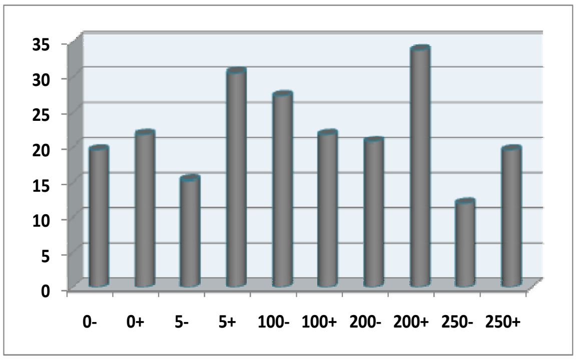

Figure 6. Diagram of starting friction force To

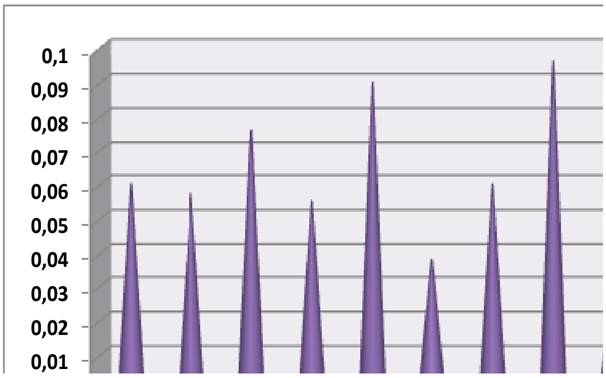

Figure 7. Diagram of the jump of friction force Δμ

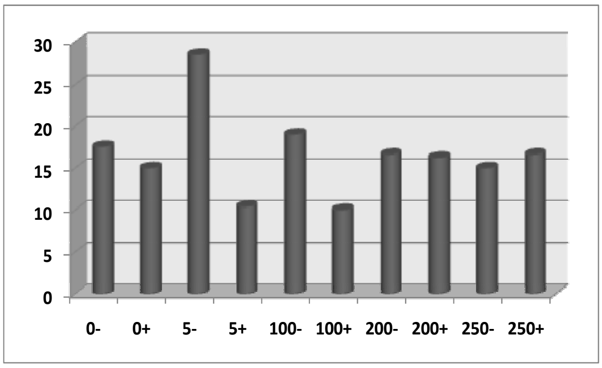

Figure 8. Diagram of the relative change of starting friction Ψs

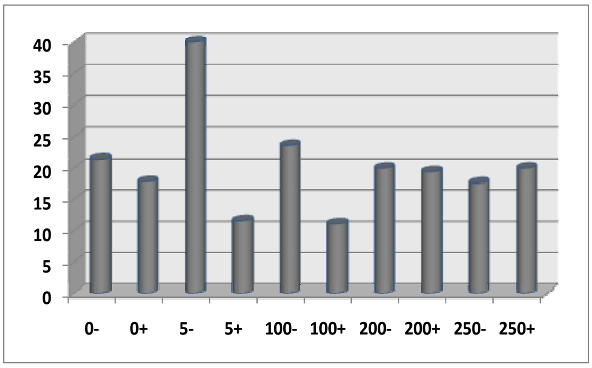

Figure 9. Diagram of the relative change of kinetic friction Ψp

Table 3. Experimental data of friction parameters

№	Series	To, [N]	T, [N]	μo	μ	Δμ	Ψs	Ψp
1	0- ($Ni^{-}-Al$)	19,62	16,35	0,34	0,28	0,06	17,6	21,4
2	0+ ($Ni^{+}-Al$)	21,8	18,53	0,38	0,32	0,057	15	17,8
3	5- ($Ni*nD5^{-}-Al$)	15,26	10,90	0,266	0,19	0,076	28,6	40
4	5+ ($Ni*nD5^{+}-Al$)	30,52	27,25	0,53	0,475	0,055	10,4	11,6
5	100- ($Ni*nD100^{-}-Al$)	27,25	21,80	0,47	0,38	0,09	19,1	23,6
6	100+ ($Ni*nD100^{+}-Al$)	21,8	19,62	0,38	0,342	0,038	10	11,1
7	200- ($Ni*nD200^{-}-Al$)	20,71	17,44	0,36	0,30	0,06	16,7	20
8	200+ ($Ni*nD200^{+}-Al$)	33,79	28,34	0,59	0,494	0,096	16,3	19,4
9	250- ($Ni*nD250^{-}-Al$)	11,99	9,81	0,20	0,17	0,03	15	17,6
10	250+ ($Ni*nD250^{+}-Al$)	19,62	16,35	0,342	0,285	0,057	16,7	20

4.3 Analysis of the experimental results

A jump in the friction force is observed for all tested tribosystems however at different values of starting friction force and kinetic friction force.

The jump is of different duration for the different coatings.

The influence of the size of nanodiamond particles upon friction parameters is not unambiguous.

The relationship between the starting friction force and the size of nanodiamond particles is strongly nonlinear. This is most clearly expressed for coatings with heat treatment. At coatings without heat treatment this relationship has clear maximum for particles size $\delta = 100$ nm, however the value of the maximum is lower than the two maximums in the curve of the coatings without heat treatment.

Genesis and variations in the friction forces depend directly on the formation and evolution of the contact spots, the latest depending on many various factors too.

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