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WEAR OF ELECTROLESS NICKEL-PHOSPHORUS COMPOSITE COATINGS WITH NANODIAMOND PARTICLES

M. KANDEVA[a,b]*, T. PENYASHKI[c], G. KOSTADINOV[c], ZH. KALITCHIN[d]*, J. KALEICHEVA[e]

[a]*Faculty of Industrial Engineering, Tribology Centre, Technical University – Sofia, 8 Kl. Ohridski Blvd., 1000 Sofia, Bulgaria*
E-mail: *kandevam@gmail.com*
[b]*South Ural State University, 76 Prospekt Lenin, Chelyabinsk, Russia*
[c]*Institute of Soil Science Agrotechnologies and Plant Protection 'N. Pushkarov' – Agricultural Academy, Sofia, Bulgaria*
[d]*SciBulCom 2 Ltd., 7 Nezabravka Str., 1113 Sofia, Bulgaria*
E-mail: *kalitchin@gmail.com*
[e]*Faculty of Industrial Engineering, Technical University – Sofia, 8 Kl. Ohridski Blvd., 1000 Sofia, Bulgaria*

Abstract. The work presents results for the wear characteristics of electroless nickel-phosphorus composite coatings with and without diamond nanoparticles with dimensions of 4, 40, 100, 200 and 250 nm. The coatings were obtained by the technology of EFFTOM NIKEL at the Faculty of Industrial Technology at the Technical University of Sofia. Results obtained were on the effect of heat treatment of coatings and the size of diamond nanoparticles on mass wear, wear rate and wear resistance under abrasive friction conditions when greasing with LITEL U-2 grease. It has been found that the heat treatment of all coatings – with and without diamond nanoparticles – leads to increased abrasion resistance. Embedding diamond nanoparticles into electroless nickel composite coatings increases abrasion resistance but it has not a disproportionate impact. The relationship between the size of nanodiamond particles and abrasion resistance is with a wavy nature with pronounced maxima and minima. The highest wear resistance is obtained with coatings of 100 nm diamond nanoparticles and heat treatment of the coating. By increasing the size of diamond nanoparticles, the hardness of the coatings increases with heat treatment. For coatings without heat treatment, this increase occurs to a diamond nanoparticle size of 100 nm.

Keywords: electroless nickel composite coatings, nanodiamond particles, wear, environmental protection.

* For correspondence.

AIMS AND BACKGROUND

Electroless plating is a chemical deposition process, which can be defined as the deposition of a metal from an aqueous solution of its salt by a controlled chemical reduction that is catalysed by the metal or alloy being deposited[1].

The oxidation of a substance is characterised by the loss of electrons (anodic process), while reduction is distinguished by a gain of electrons (cathodic action)[2].

Electroless plating has been used to obtain coatings of Ni, Co, Pd, Cu, Au, and Ag as well as some alloys containing these metals plus P or B. Electroless Cr deposition has also been claimed[1]. The most common reducing agent used is sodium hypophosphite (NaH_2PO_2). Alternatives are sodium borohydride, dimethylamine borane and hydrazine but they are used much less frequently[2,3]. It is estimated that sodium hypophosphite is used in more than 99% of all electroless nickel plating[4].

The chemical deposition on nickel ions on catalytically active surface by hypophosphite in aqueous solution has been described in Ref. 5.

According to their phosphorus content, electroless nickel coatings can be categorised into four principal groups: low phosphorus (1–3 wt.% P), low-medium phosphorus (3–6 wt.% P), medium phosphorus (6–9 wt.% P) and high phosphorus (9–13 wt.% P) alloys[6].

Chemical composition of the electroless nickel coatings affect its structure and produce often generally distinct physical, mechanical and tribological properties of these coatings[1,3,4–8].

In applications requiring hardness and wear resistance, electroless nickel composite coatings have been even more successful in not only replacing chrome but actually surpassing the performance of hard chrome plating[9].

Depending on the application, any of the coatings might improve wear resistance or lubricity[10]. The method of formation, mechanism of particle incorporation, factors influencing particle incorporation, effect of particle incorporation on the coating structure, hardness, friction, wear and corrosion resistance, high temperature oxidation resistance and applications are discussed in Refs 11 and 12.

In the present study the effect of nanodiamond particles size and heat treatment on wear characteristics of electroless nickel composite coatings under abrasion and limiting lubrication with LITEL U-2 grease is investigated.

EXPERIMENTAL

The coatings were fabricated by electroless plating process EFTTOM-NICKEL, developed at Technical University of Sofia[13–16]. The nickel coatings include nanodiamond particles sized 4, 40, 100, 200 and 250 nm (5–7 vol.%). Heat treatment was applied to some of the samples to improve its mechanical properties and adhesion in the substrate-coating interface. Applied heat treatment consists of heating at 300°C for 6 h.

The substrate material for all coatings was a carbon steel (GOST 380-94), with chemical composition shown in Table 1.

Hardness of the substrate was 135 $HV_{0.5}$. The roughness of the substrate was examined with mechanical profilometer. Measurement was performed in at least five points on the surface of coatings and the average roughness was $R_a = 0.089$ μm.

Table 1. Chemical composition (wt.%) of the coated material (substrate)

Element	C	Si	Mn	Ni	P	S	Cr	Fe
Percentage	0.4	0.20	0.55	0.30	0.45	0.045	0.30	balance

Twelve different samples with electroless Ni coatings were investigated. They can be divided into two series, i.e. samples without and with heat treatment. In each series there are six samples: (1) coating without diamond nanoparticles; (2) coating with diamond nanoparticles of average size 4 nm; (3) coating with diamond nanoparticles of average size 40 nm; (4) coating with diamond nanoparticles of average size 100 nm; (5) coating with diamond nanoparticles of average size 200 nm, and (6) coating with diamond nanoparticles of average size 250 nm. Volume concentration of diamond nanoparticles was the same in all coatings, i.e. from 5 to 7 vol.%. Designations of tested coatings are shown in Table 2. The ultradispersed diamond powder (diamond nanoparticles) was produced by the detonation synthesis method.

Coatings thickness was measured in 5 points on each coating surface, and the average values are shown in Table 2. Measurements of surface microhardness ($HV_{0.5}$) were carried out using Vickers microhardness tester under the load of 500 g.

Table 2. Designation, thickness and microhardness of tested coatings

Sample	Designation	Description	Thickness (μm)	Micro-hardness $HV_{0.5}$
1	2	3	4	5
1	Ni	electroless Ni coating without nanoparticles and heat treatment	26.2	538
2	Ni^{HT}	electroless Ni coating without nanoparticles and with heat treatment	11.8	560
3	Ni-D4	electroless Ni coating with D nanoparticles of 4 nm size and without heat treatment	23.5	295
4	Ni-$D4^{HT}$	electroless Ni coating with D nanoparticles of 4 nm size and with heat treatment	8.5	545
5	Ni-D40	electroless Ni coating with D nanoparticles of 40 nm size and without heat treatment	15.2	594

to be continued

Continuation of Table 2

1	2	3	4	5
6	Ni-D40^{HT}	electroless Ni coating with D nanoparticles of 40 nm size and with heat treatment	14.5	668
7	Ni-D100	electroless Ni coating with D nanoparticles of 100 nm size and without heat treatment	28.2	688
8	Ni-D100^{HT}	electroless Ni coating with D nanoparticles of 100 nm size and with heat treatment	7.4	745
9	Ni-D200	electroless Ni coating with D nanoparticles of 200 nm size and without heat treatment	27.1	540
10	Ni-D200^{HT}	electroless Ni coating with D nanoparticles of 200 nm size and with heat treatment	9.3	790
11	Ni-D250	electroless Ni coating with D nanoparticles of 250 nm size and without heat treatment	31.2	548
12	Ni-D250^{HT}	electroless Ni coating with D nanoparticles of 250 nm size and with heat treatment	10.8	810

RESULTS AND DISCUSSION

In order to investigate the influences on tribological properties (abrasive wear) of different electroless Ni coatings, two tribological experiments were done with different test equipments. The main goal was to determine the influence of different nanoparticles addition, as well as heat treatment.

In addition to the wear data, the hardness of each of tested materials was determined, as an ancillary mechanical property, to make appropriate correlations. The use of hardness as a parameter for predicting the wear behaviour of materials must be done with caution since these characteristics very often they are not in correlation[17].

Abrasive wear tests were carried out on Taber Abraser with a modified standard test conditions (only one abrasive roller was used), in the ambient air at room temperature[16].

Obtained results of the mass loss are shown as a function of sliding distance, in the form of the comparative wear curves. Wear rate (*W*) in mg/m is calculated by fitting the wear curves (it is the slope of wear curve), assuming that the steady-state wear was from the beginning of the tests (which is common thing for the abrasive wear). In order of easier comparison of different coatings, a value of relative wear resistance (R) is also introduced in Ref. 2. The relative wear resistance (R) is calculated as a ratio of reference sample wear rate (W_y) and wear rate of the analysed sample (W_x), where x and y denote the designation number of the sample – $R_{x,y} = W_y/W_x$. Relative wear resistance of the reference sample is always R = 1.

Abrasive wear of the coatings was determined at various number of cycles, i.e. at N = 300, 600, and 900, and corresponding mass losses are presented in Table 3. By calculating the linear wear of the coatings (which is not presented), it was confirmed that it was lower than the thickness of all tested coatings.

Table 3. Mass loss of tested coatings

Sample	Coatings	Number of cycles (N)		
		300	600	900
		Sliding distance (m)		
		71.8	143.6	215.4
		Mass loss (mg)		
1	Ni	2.2	10.0	17.8
2	Ni^{HT}	2.6	5.5	8.9
3	Ni-D4	3.5	8.9	13.5
4	Ni-D4^{HT}	0.8	1.2	2.1
5	Ni-D40	4.7	6.3	8.8
6	Ni-D40^{HT}	0.8	1.9	2.8
7	Ni-D100	0.5	1.6	2.7
8	Ni-D100^{HT}	0.4	0.7	1.3
9	Ni-D200	4.4	6.9	11.3
10	Ni-D200^{HT}	4.3	5.1	5.8
11	Ni-D250	2.4	6.2	7.9
12	Ni-D250^{HT}	0.6	1.6	3.3

Tables 4, 5 and 6 provide results for the wear rate, the wear resistance and relative wear resistance of the coatings from the results in Table 3.

Table 4. Wear rate of tested coatings

Sample	Coatings	Number of cycles (N)		
		300	600	900
		Sliding distance (m)		
		71.8	143.6	215.4
		Wear rate (mg/m)		
1	Ni	3.1×10^{-2}	7.0×10^{-2}	8.3×10^{-2}
2	Ni^{HT}	3.6×10^{-2}	3.8×10^{-2}	4.1×10^{-2}
3	Ni-D4	4.9×10^{-2}	6.2×10^{-2}	6.3×10^{-2}
4	Ni-D4^{HT}	1.1×10^{-2}	0.8×10^{-2}	1.0×10^{-2}
5	Ni-D40	6.5×10^{-2}	4.4×10^{-2}	4.1×10^{-2}
6	Ni-D40^{HT}	1.1×10^{-2}	1.3×10^{-2}	1.3×10^{-2}
7	Ni-D100	0.7×10^{-2}	1.1×10^{-2}	1.3×10^{-2}
8	Ni-D100^{HT}	0.5×10^{-2}	0.5×10^{-2}	0.6×10^{-2}
9	Ni-D200	6.1×10^{-2}	4.8×10^{-2}	5.2×10^{-2}
10	Ni-D200^{HT}	6.0×10^{-2}	3.6×10^{-2}	2.7×10^{-2}
11	Ni-D250	3.3×10^{-2}	4.3×10^{-2}	3.7×10^{-2}
12	Ni-D250^{HT}	0.8×10^{-2}	1.1×10^{-2}	1.5×10^{-2}

Table 5. Wear resistance of tested coatings

Sample	Coatings	Number of cycles (N)		
		300	600	900
		Sliding distance (m)		
		71.8	143.6	215.4
		Wear resistance (m/mg)		
1	Ni	0.32×10^{2}	0.14×10^{2}	0.12×10^{2}
2	Ni^{HT}	0.28×10^{2}	0.26×10^{2}	0.24×10^{2}
3	Ni-D4	0.20×10^{2}	0.16×10^{2}	0.16×10^{2}
4	Ni-D4^{HT}	0.91×10^{2}	1.3×10^{2}	1.03×10^{2}
5	Ni-D40	0.15×10^{2}	0.23×10^{2}	0.24×10^{2}
6	Ni-D40^{HT}	0.91×10^{2}	0.77×10^{2}	0.77×10^{2}
7	Ni-D100	1.42×10^{2}	0.91×10^{2}	0.80×10^{2}
8	Ni-D100^{HT}	2.0×10^{2}	2.0×10^{2}	1.67×10^{2}
9	Ni-D200	0.16×10^{2}	0.21×10^{2}	0.19×10^{2}
10	Ni-D200^{HT}	0.17×10^{2}	0.28×10^{2}	0.37×10^{2}
11	Ni-D250	0.3×10^{2}	0.23×10^{2}	0.27×10^{2}
12	Ni-D250^{HT}	1.25×10^{2}	0.9×10^{2}	0.67×10^{2}

Table 6. Relative wear resistance and the influences of heat treatment and addition diamond nano particles on the increase of wear resistance of tested coatings

Sample	Coatings	Wear resistance (m/mg) (900 cycles)	Relative wear resistance (R)		
			influence of heat treatment	influence of particles addition	both influence
1	Ni	0.12×10^2	$R_{1,1} = 1$	$R_{1,1} = 1$	$R_{1,1} = 1$
2	Ni^{HT}	0.24×10^2	$R_{2,1} = 2$	$R_{2,2} = 1$	$R_{2,1} = 2$
3	Ni-D4	0.16×10^2	$R_{3,3} = 1$	$R_{3,1} = 1.33$	$R_{3,1} = 1.33$
4	$Ni\text{-}D4^{HT}$	1.03×10^2	$R_{4,3} = 6.44$	$R_{4,2} = 4.29$	$R_{4,1} = 8.58$
5	Ni-D40	0.24×10^2	$R_{5,5} = 1$	$R_{5,1} = 2$	$R_{5,1} = 2$
6	$Ni\text{-}D40^{HT}$	0.77×10^2	$R_{6,5} = 3.2$	$R_{6,2} = 3.21$	$R_{6,1} = 6.42$
7	Ni-D100	0.80×10^2	$R_{7,7} = 1$	$R_{7,1} = 6.67$	$R_{7,1} = 6.67$
8	$Ni\text{-}D100^{HT}$	1.67×10^2	$R_{8,7} = 2.9$	$R_{8,2} = 6.96$	$R_{8,1} = 13.9$
9	Ni-D200	0.19×10^2	$R_{9,9} = 1$	$R_{9,1} = 1.58$	$R_{9,1} = 1.58$
10	$Ni\text{-}D200^{HT}$	0.37×10^2	$R_{10,9} = 1.95$	$R_{10,2} = 1.54$	$R_{10,1} = 3.08$
11	Ni-D250	0.27×10^2	$R_{11,11} = 1$	$R_{11,1} = 2.25$	$R_{11,1} = 2.25$
12	$Ni\text{-}D250^{HT}$	0.67×10^2	$R_{12,11} = 2.48$	$R_{12,2} = 2.79$	$R_{12,1} = 5.58$

The wear curves for coatings without nanoparticles (Ni and NiHT) are shown in Fig. 1 and for coatings with D nanoparticles (Ni-D4, $Ni\text{-}D4^{HT}$, Ni-D40, $Ni\text{-}D40^{HT}$, Ni-D100, $Ni\text{-}D100^{HT}$, Ni-D200, $Ni\text{-}D200^{HT}$, Ni-D250 and $Ni\text{-}D250^{HT}$) in Figs 2–6.

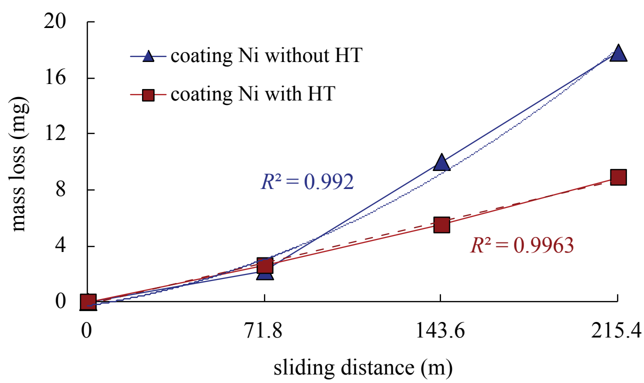

Fig. 1. Mass loss versus sliding distance for coatings without nanoparticles (without and with heat treatment)

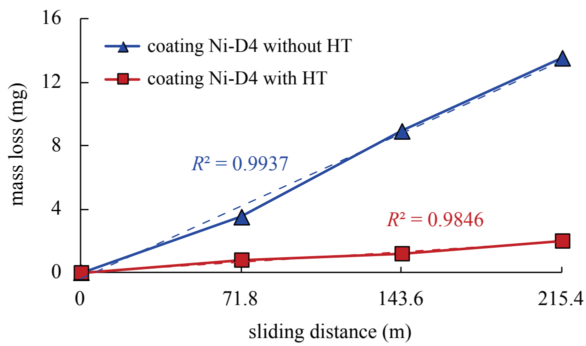

Fig. 2. Mass loss versus sliding distance for coatings with diamond nanoparticles of 4 nm size (without and with heat treatment)

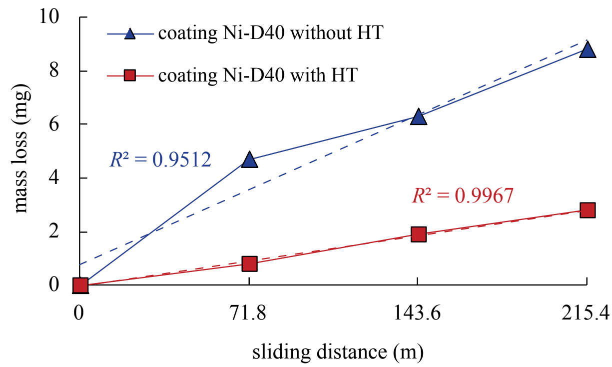

Fig. 3. Mass loss versus sliding distance for coatings with diamond nanoparticles of 40 nm size (without and with heat treatment)

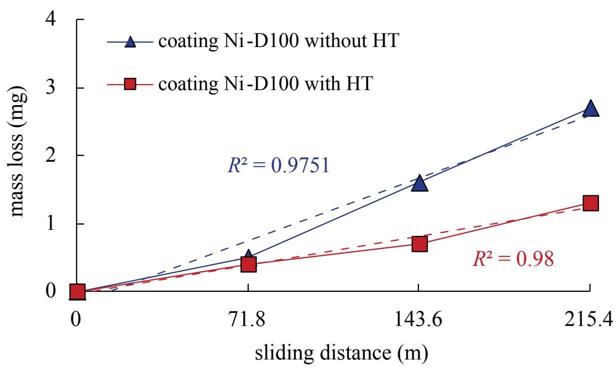

Fig. 4. Mass loss versus sliding distance for coatings with diamond nanoparticles of 100 nm size (without and with heat treatment)

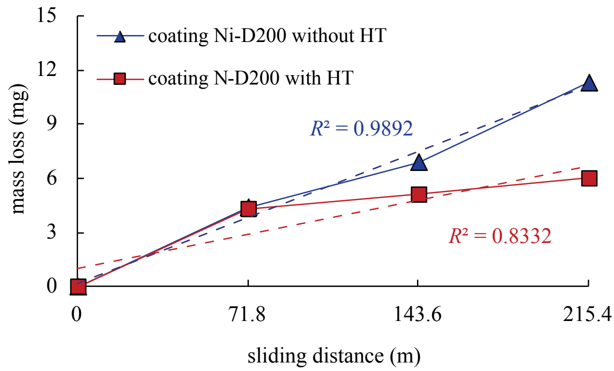

Fig. 5. Mass loss versus sliding distance for coatings with diamond nanoparticles of 200 nm size (without and with heat treatment)

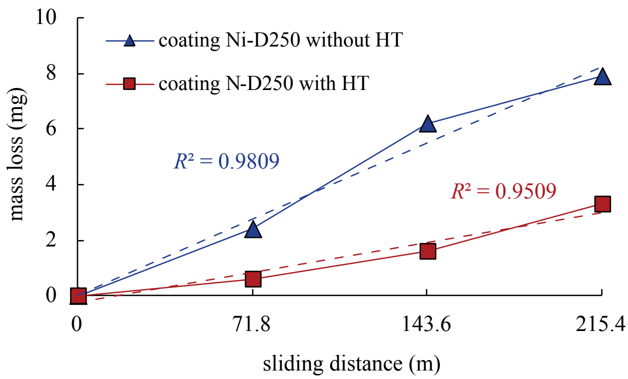

Fig. 6. Mass loss versus sliding distance for coatings with diamond nanoparticles of 250 nm size (without and with heat treatment)

Presence of diamond nanoparticles decreases the abrasive wear. The heat treatment also leads to reduction of wear for both version of Ni coating (with and without D nanoparticles). The lowest wear rate of 0.5×10^{-2} mg/m at 900 cycles of friction is observed for coating Ni-D100^{HT}, i.e. coating with D nanoparticles of 100 nm size, and with heat treatment. The increase of wear resistance for this coating was approximately 13.9 times in comparison to coating Ni (coating without nanoparticles and heat treatment) which shows the highest wear rate of 8.3×10^{-2} mg/m.

Influences of heat treatment and addition of nanoparticles on the increase of the abrasive wear resistance are analysed separately and together. It was done by comparing the wear rates of tested coatings, and it was expressed by using the relative wear resistance (R). Relative wear resistance (R) was calculated according to the equation given in Ref. 2. In analysing the influence of heat treatment, six coatings without heat treatment (samples 1, 3, 5, 7, 9 and 11) were the reference coatings, with the relative wear resistance R = 1, for the same coatings with heat treatment (samples 2, 4, 6, 8, 10 and 12). In analysing the influence of nanoparticles addition two coatings without nanoparticles (samples 1 and 2) were the reference coatings, with the relative wear resistance R = 1, for the corresponding (with or without heat treatment) coatings with nanoparticles. In analysis of both influences one coating without heat treatment and nanoparticles (sample 1) was the reference coating, with the relative wear resistance R = 1, for all other coatings. Calculated relative wear resistances are presented in Table 5.

Figure 7 presents the graph of the effect of the size of nanodiamond particles on the wear rate for coatings without and with heat treatment. The curve has a wavy character. In the range of 0 to 100 nm, the wear rate decreases to a minimum, after which it starts to rise, reaches a maximum of 200 nm and decreases again. This tendency is observed in non-heat and heat treatmented coatings.

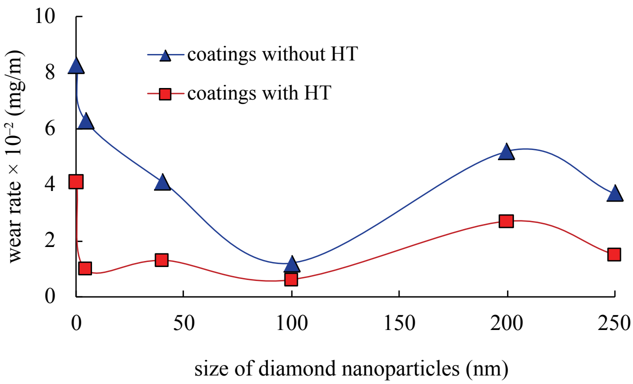

Fig. 7. Wear rate versus size of diamond nanoparticles for tested coatings (without and with heat treatment)

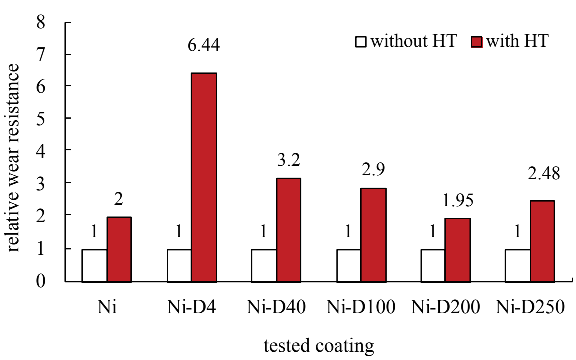

Fig. 8. Influence of heat treatment (HT) on the wear resistance of tested coatings

In order to visually perceive the results shown in Table 5, three diagrams are presented, showing the influence of heat treatment (Fig. 8), influence of nanoparticles addition (Fig. 9), and both influences (Fig. 10) on the increase of abrasive wear resistance of tested coatings.

The analysis of the results shows that the heat treatment principally increases abrasive wear resistance (Fig. 8). From Fig. 10 it is seen that the influence the heat treatment on abrasive wear resistance is strongest in coatings (Ni-D4), i.e. with dimensions of nanodiamond particles 4 nanometers. Relative wear resistance is 6.44. This result corresponds to the result for the lowest wear level of 1.0×10^{-2} (Table 4 and Fig. 6) of Ni-D4^{HT} coatings. The least is the influence of the nanodiamond particles size on the abrasion resistance is of coatings (Ni-D200), i.e. with dimensions of nanodiamond particles 200 nm. The relative wear resistance is 1.95, which is close to the relative wear resistance of the nickel coatings without nanodiamond particles (Fig. 8). The low abrasion resistance of Ni-D200 coatings on grease friction is probably associated with an increase in the brittleness of this coating when heat treated and the presence of lubricant. Such an abrasion resistance reduction effect was observed by the authors in Ni-D100 coatings, i. e. with nanodiamond particles measuring 100 nanometers in abrasive friction without lubricant[15].

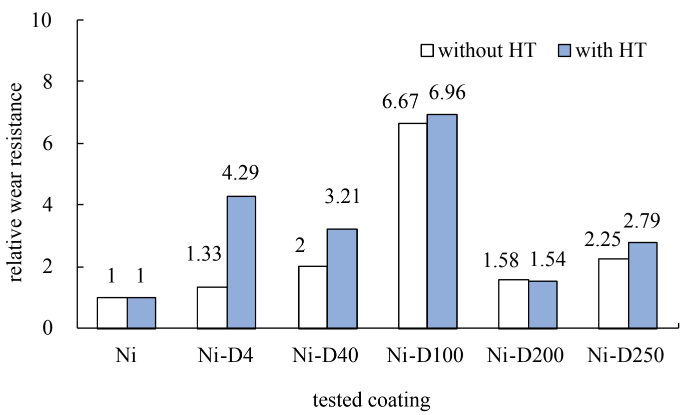

Fig. 9. Influence of nanoparticles addition on the wear resistance of tested coatings

From Fig. 9, it can be noticed that the influence of nanoparticle addition on abrasive wear resistance was higher than the heat treatment influence. As the diamond nanoparticles increase, the change in relative wear resistance is increasing but is not linear. Maximum and minimum values are observed. Maximum relative resistance have diamond nanoparticle coatings of 100 nm that are heat- or not heat treated and have very close values, R = 6.67 and 6.96, respectively. The smallest relative resistance is observed at coatings with diamond nanoparticles of

200 nm, which are heat- or not heat treated with very close values, R = 1.58 and 1.54, respectively (Fig. 9).

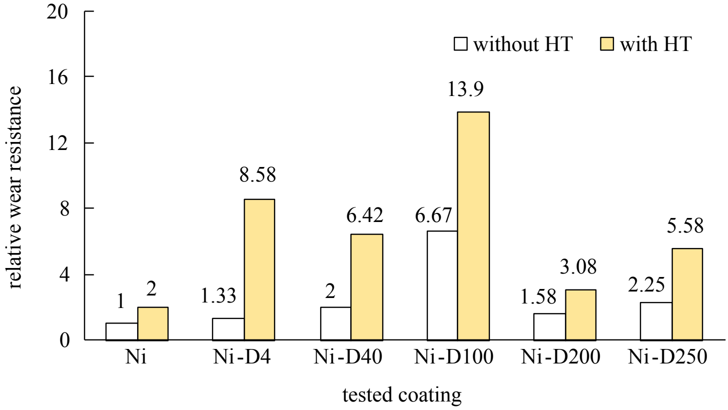

Fig. 10. Influence of heat treatment (HT) and nanoparticles addition on the abrasive wear resistance of tested coatings

The combined effects of adding diamond nanoparticles and heat treatment (Fig. 10) give the best effect on increasing abrasion resistance to friction with grease.

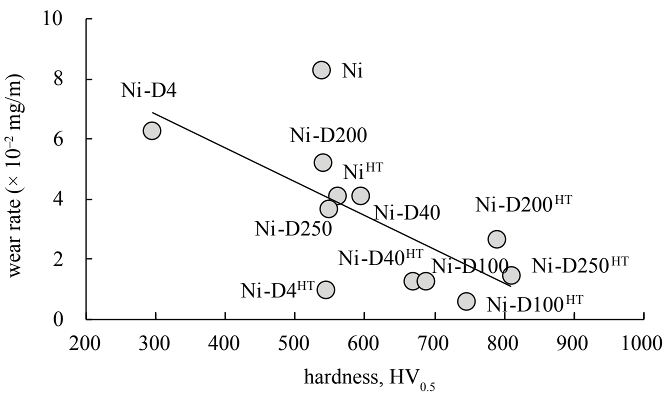

Fig. 11. Wear rate versus hardness of tested coatings

The relationship between obtained abrasive wear values and hardness (Table 2) of tested coatings is shown in Fig. 11. The wear rate generally decreases as hardness increase, as it could be expected, but it is obvious that relationship between the abrasive wear and hardness values is questionable.

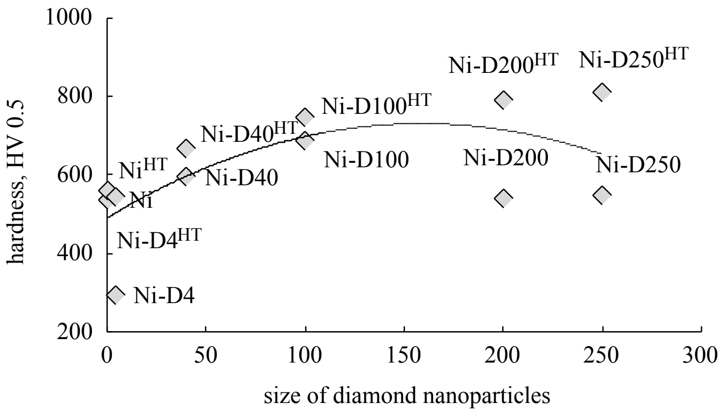

Fig. 12. Hardness versus size of diamond nanoparticles of tested coatings

Figure 12 shows the influence of the size of diamond nanoparticles on the hardness of nickel coatings without and with heat treatment. As the diamond nanoparticle size increases, the hardness of the heat treatment coatings increases and this increase is non-linear. For non-heat treated coatings, the increase in hardness continues to a nanoparticle size of 100 nm, then with increasing nanoparticle size, hardness decreases.

CONCLUSIONS

The main results of the tribological study of the electroless nickel coatings with diamond nanoparticles in terms of abrasion friction lubrication with grease LITEL U-2 are reduced to:

– The incorporation of 4, 40, 100, 200, and 250 nm diamond nanoparticles in the electroless nickel coatings with and without heat treatment increases their abrasion wear resistance.

– As a rule, when increasing the size of diamond nanoparticles, abrasion wear resistance increases, but the relationship between nanoparticle size and wear resistance is not directly proportional. It has a non-linear character with pronounced maxima and minima.

– Maximum abrasion wear resistance is observed at electroless nickel coatings with 100 nm diamond nanoparticles and after heat treatment of the coatings. The wear resistance is 13.9 times greater than electroless nickel coatings without nanoparticles.

– Increasing the size of the diamond nanoparticles in electroless nickel coatings with heat treatment results in an increase of the hardness of the coatings. For coatings without heat treatment the increase in hardness is observed to a diamond nanoparticle size of 100 nm. With an increase in nanoparticle size of more than 100 nm, hardness gradually decreases.

– The influence of heat treatment at 300°C for 6 h on in electroless nickel coatings with and without diamond nanoparticles results in an increase in their abrasion wear resistance.

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