

ABRASION WEAR OF ELECTROLESS NICKEL COMPOSITE COATINGS MODIFIED WITH BORON NITRIDE NANOPARTICLES

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Abstract. The present work investigates the morphology, structure and characteristics of the wear of electroless nickel-phosphorous coatings containing cubic boron nitride (cBN₁₀) nanoparticles – 10 nm (5–7 vol.) in LITEL U-2 grease abrasive friction. The coatings were fabricated by electroless plating process EFTTOM-NICKEL, developed at Technical University of Sofia. Comparative studies of nanoparticles containing dispersive reinforced and non-reinforced coatings were performed. The studies were carried out in conditions of heat treatment at 300°C for 6 h and without heat treatment. In both cases surface layers with incorporated nanoparticles cubic boron nitride (cBN₁₀) within electroless nickel-phosphorous coatings were observed by scanning electron microscopy (SEM) and X-ray energy dispersive microanalysis (EDX) methods. It was confirmed that the heat treatment leads to formation a finer (possibly nano) crystalline structure compared to that of non-heat-treated specimens as well as to formation of more nodules. In a comparative study of abrasive grease LITEL-U-2 wear, it was found that separately addition of cBN₁₀ nanoparticles and heat treatment resulted in a significant increase in the wear resistance of modified coatings. The effect of heat treatment is higher (4.42 times) compared with the influence of the addition of nanoparticles (2.4 times) without heat treatment. With the combined influence of nanoparticle addition and heat treatment, the best effect is achieved – 4.9 times the wear resistance increasing. It has been found that in the presence of lubricant (grease LITEL-U-2) there is a non-linear increase in abrasion resistance combined with increased hardness of the modified coatings, unlike the abrasive wear in dry friction where there was no such dependence.

Keywords: tribology, electroless nickel plating, dispersive reinforced coatings, abrasive wear, cubic boron nitride nanoparticles modification.

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^{1,2}. The potential

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of the electroless nickel coatings increases due to the ability of easy production of functional composite coatings with incorporated various hard non-metal (ceramic) particles. Those dispersive reinforced surface layers contain one, two or more kinds of hard oxides, nitrides, carbides, such as Al_2O_3 , Si_2O_3 , TiO_2 , SiC , WC , cBN , etc. particles. The application of electroless nickel composite coatings with particles of different materials, sizes and amounts is very interesting, since its dispersive reinforcing generally leads to the improvement of mechanical and tribological characteristics^{3,4}.

Generally, these particles are evenly and thoroughly distributed throughout the coating, which is firmly bonded to the substrate. These coatings, therefore, have all of the inherent features of electroless nickel as well as the properties of whatever particles are selected.

Theoretically, almost any type of particle could be incorporated, as long as it could withstand the conditions within an electroless nickel bath, and if it were of the appropriate size. Generally speaking, diamond and silicon carbide electroless nickel composite coatings are chosen for wear resistance, while boron nitride and PTFE composite coatings are selected for lubricity. However, depending on the application, any of these coatings might improve wear resistance or lubricity⁵. 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 elsewhere^{6,7}.

The present work investigates the morphology, structure and characteristics of the wear of chemical composite nickel coatings containing cubic boron nitride (cBN) nanoparticles in LITEL U-2 grease abrasive friction test.

EXPERIMENTAL

Materials. The physical and mechanical properties of an electroless nickel coating depend on its composition, which basically is determined by the formulation of the plating bath. A solution with one nickel cations source is used, preferably nickel sulphate, but nickel chloride is also used for limited application⁸. The combination of both salts may also be used as components of effective electroless nickel bath⁹. The coatings were fabricated by based on nickel sulphate electroless plating process EFTTOM-NICKEL, developed at Technical University of Sofia¹⁰. The composite nickel coatings include cubic boron nitride nanoparticles of 10 nm (cBN_{10}) 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 St3kp (GOST 380-94), with chemical composition shown in Table 1.

Hardness of the substrate was 135 HV 0.05. The roughness of the substrate was examined with a mechanical profilometer. Measurement was performed in at least five points on the surface of coatings and the average roughness was $Ra = 0.089 \mu\text{m}$.

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

Four different samples with electroless Ni coatings were investigated: (1) Ni coating without BN nanoparticles and without heat treatment (Ni); (2) Ni coating without BN nanoparticles and with heat treatment (Ni-BN^{HT}); (3) Ni coating with BN nanoparticles and without heat treatment (Ni-BN), and (4) Ni coating with BN nanoparticles and with heat treatment (Ni-BN^{HT}). The average size of cubic boron nitride (c-BN) nanoparticles was 10 nm, and its volume concentration was from 5 to 7%. Designations of tested coatings are shown in Table 2.

The thickness of the coatings is measured with a Pocket LEPTOSKOP 2021 Fe at 5 points on each coating surface and the mean 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. At least three measurements were made for each sample in order to eliminate possible segregation effects and to obtain a representative value of the material microhardness. Average values are presented in Table 2.

Table 2. Designation, thickness and microhardness of tested coatings

Sample	Designation	Description	Thickness (μm)	Microhardness HV 0.5
1	Ni	electroless Ni coating without nanoparticles and heat treatment	26.2	538
2	N ^{HT}	electroless Ni coating without nanoparticles and with heat treatment	12.8	560
3	Ni-BN ₁₀	electroless Ni coating with nanoparticles of 10 nm size and without heat treatment	22.8	518
4	Ni-BN ₁₀ ^{HT}	electroless Ni coating with BN nanoparticles of 10 nm size and with heat treatment	9.5	590

Morphology and structure of the obtained dispersive reinforced layers containing cubic boron nitride nanoparticles. There are cubic boron nitride nanoparticles (cBN) built into the surface layers, deposited by electroless nickel plating in the composite coating. A subsequent heat treatment of the obtained coating was performed. It was evaluated the effect of heat treatment on the morphology and struc-

ture of the deposited composite coating (Ni-P-cBN₁₀) using the Scanning electron microscopy (SEM) and the X-ray energy dispersive microanalysis (EDX) methods.

The applied so far heat treatment methods¹⁰ of the chemical (electroless) nickel coatings have shown a direct proportionality between the increase of the hardness and the wear resistance associated with an increase in the temperature of the heat treatment.

However, this increase is up to a specified temperature of about 400°C for 1 h retention. When the temperature rises above 400°C a significant decrease of the mechanical parameters is observed. The changes of the coatings properties after the heat treatment are due to structural changes associated with the transformation of the initially chemically applied amorphous nickel coating into a crystalline state.

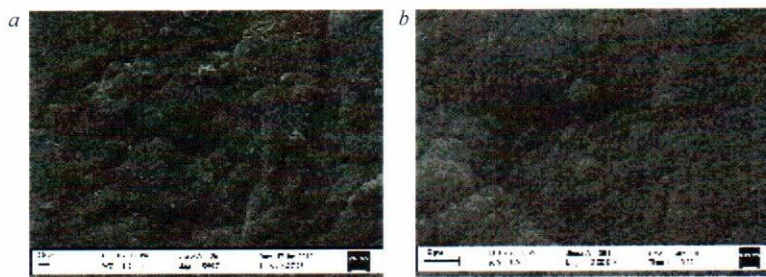


Fig. 1. SEM of the surface of the obtained dispersive reinforced with cubic boron nitride (cBN₁₀) composite nickel coating without heat treatment

The addition of cBN nanoparticles in the chemical nickel plate solution suppress the formation of a microcrystalline structure as early as deposition of the coating and because of this the produced amorphous material has a finer structure (Fig. 1a). Nanosized particles can therefore also be used as a modifier in the chemical deposition process. The figures below show the morphology of the surface of the obtained dispersive reinforced with cubic boron nitride (cBN₁₀) modified nickel coating. The investigations were performed by scanning electron microscopy at various magnifications. The conducted energy dispersive X-ray spectroscopy microanalysis (EDX) on the surface of a composite nickel coating (containing nano-sized cBN particles) without heat treatment, confirms the presence of chemical elements Ni, P, N and B in the deposited layers (Fig. 2b). Deposition of composite nickel coatings is related to the formation of nodules that grew on particles grain surfaces. A zone (Fig. 2a) with the presence of some nodules was selected for mapping analysis. The presence of boron and nitrogen elements indicates the successful incorporation of nanosized cBN nanoparticles into the modified electroless nickel coatings.

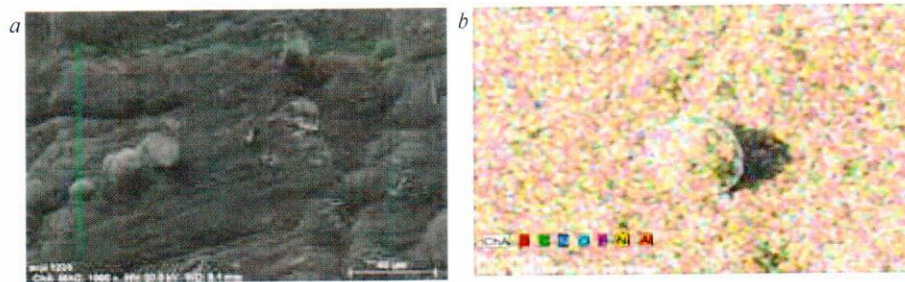


Fig. 2. EDX of nickel-phosphorus coating (dispersive reinforced with nano-sized cBN_{10}) without heat treatment and some nodules

Result of the next heat treatment of the composite coating is saturated crystalline solid solution, from which a new phase of nickel phosphide Ni_3P is separated and an equilibrium mechanical mixture of Ni and Ni_3P phases is formed. This phenomenon is a diffusion process known as dispersion hardening. During the incomplete phase transformation of the amorphous coating, the incomplete realisation of the diffusion process is associated with the obtaining of metastable phases of NiP_2 and Ni_5P_4 . In the presence of nickel-phosphorus-coated cBN particles and subsequent heat treatment, besides nickel phosphide Ni_3P around the particles, Ni_3B nickel boride is probably formed. In such cases, the equilibrium mechanical mixture already contains three phases – Ni, Ni_3P and Ni_3B .

The lowest temperature for heat treatment in which the formation of a crystalline structure of chemically deposited nickel-phosphorous coating is observed is 300°C . But the complete diffusion process and dispersion hardening require longer retention. A heat treatment mode at a temperature of 300°C and a duration of 6 h is selected in order to obtain good mechanical properties associated with the completion of the phase transformation in the nickel-phosphorous composite coating.

Comparison of the morphology of the deposited modified coatings with and without subsequent heat treatment was performed using a scanning electron microscopy techniques. At the same magnification of the surface of the composite nickel coatings, it can be seen that the heat treated specimens have a visually finer (probably nano) crystalline structure (Fig. 3) compared to the non-treated samples (Fig. 1). The recrystallisation processes during the heat treatment have also occurred, probably related to the higher rate of crystal nucleuses formation than their rate of growth, and therefore more nodules have formed (Fig. 3b).

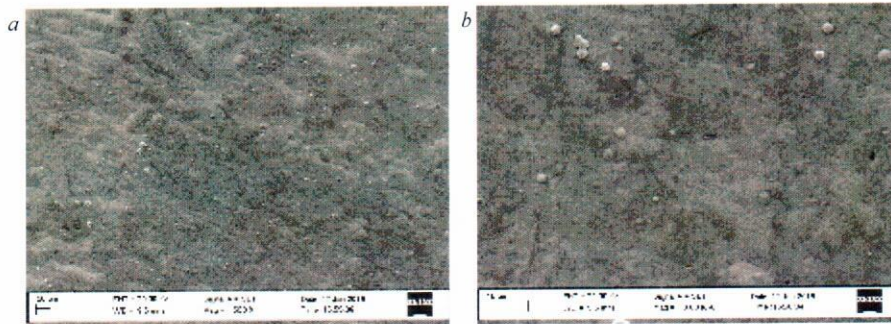


Fig. 3. Scanning electron microscopy on the surface of heat treated nickel-phosphorous coating dispersive reinforced with nano-sized particles cBN_{10} .

The energy dispersive X-ray spectroscopy microanalysis (EDX) carried on the surface of heat treated nickel-phosphorus coating containing nano-sized cBN particles ($300^{\circ}C$ at 6 h) confirms the presence of chemical elements Ni, Fe, P, N and B (Fig. 4).

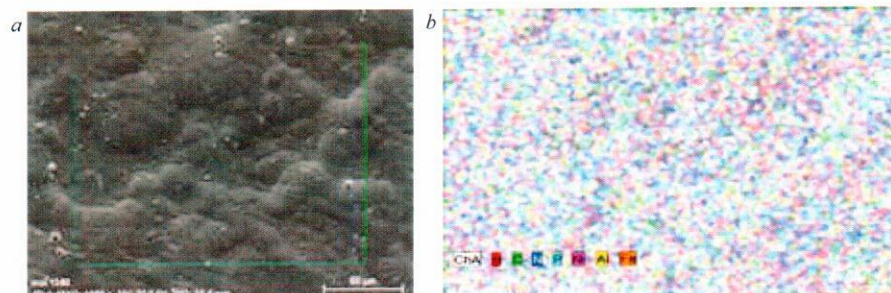


Fig. 4. EDX of heat treated nickel-phosphorous coating modified with nano-sized cBN_{10} particles with an increased concentration of nodules

A zone (Fig. 4a) with an increased concentration of nodules was selected for analysis. In the heat treated nickel-phosphor coating contained cBN particles also is observed the presence of the element Fe (Fig. 4b), unlike the mapping spectra of the non-treated coatings (Fig. 2b). The presence of iron is probably due to the matrix-to-coating diffusion processes.

Abrasive wear test on grease LITEL U-2. Abrasive wear tests were carried out on Taber Abraser (Fig. 5) with a modified standard test conditions (only one abrasive roller was used), in the ambient air at room temperature. The Taber Abraser generates a combination of sliding and rolling motion and is primarily used for tests under mild abrasion condition⁷.

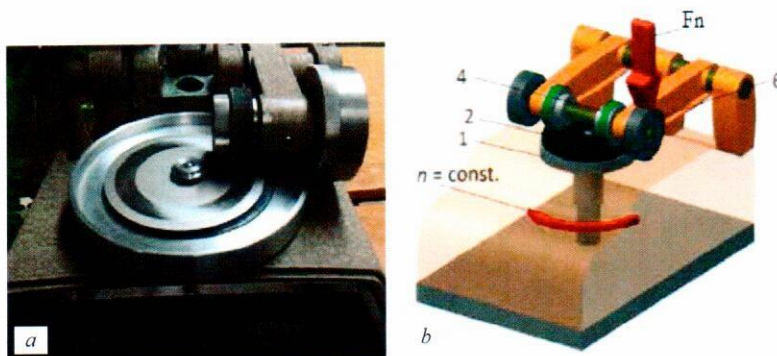


Fig. 5. Taber Abraser testing: image – *a* and 3D model – *b*

A disc sample (1) of 100 mm diameter and 3 mm thickness with coating (2) is fixed on the horizontal turntable platform, driven with constant rotational speed (n) of 60 rpm by the electric motor (3). The disk sample is lubricated with grease LITEL U-2. Abrasive roller (4), a Taber abrading wheel Calibrase CS-10, is mounted on horizontal axis (5) and provides through weights (6) the necessary normal load (F_n). Abrasive roller (wheel) is driven by the rotating test sample. The wheels produce abrasion marks that form a pattern of crossed arcs over a circular ring. The width of the worn area (circular ring) is 12.7 mm, with the inner radius of 31.75 mm and outer radius of 44.45 mm. Therefore, the distance between the rotational axis of disc sample (1) and mass centre of the contact area (K) is 38.1 mm, and the worn area is approximately 30 cm². The sliding action between the coated disc sample and abrasive roller is due to the relative motion between them which is characterised by the roller slip. This occurs because the axle of the roller is shifted from the disc sample centre of rotation with the drifting angle of around 30°. Thus, the average tangential (sliding) velocity of the coated disc sample is 0.239 m/s.

Abrasive wear is calculated as a mass loss, i.e. as a difference between the initial mass of the sample and its mass after given number of abrasion cycles (N), counted by the counter (pos. 7 in Fig. 2). Before and after testing, the coated disc was degreased and cleaned, and its mass is measured by the electronic balance with accuracy of 0.1 mg. Normal load of 250 g (2.45 N) was constant for all tests and coatings. The sliding distance (S) is calculated from the following equation:

$$S = 2\pi r N \quad (1)$$

where $r = 38.1$ mm is the distance between the rotational axis of disc sample and mass centre of the contact area, and N – the number of abrasion cycles.

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). Wear resistance is calculated as a reciprocal value of the wear rate ($I = 1/W$) in m/mg.

In order of easier comparison of different coatings, a value of relative wear resistance (R) is also introduced (equation (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. Relative wear resistance of the reference sample is always $R = 1$.

$$R_{x,y} = W_y / W_x \quad (2)$$

Abrasive wear during lubrication with grease is determined at three cycles (friction path) – $N = 300, 600$ and 900 abrasion cycles. The results for mass loss (mg), wear rate (mg/m) and wear resistance (m/mg) for all samples at the three friction cycles are given in Table 3.

Table 3. Mass loss, wear rate and wear-resistance 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-BN ₁₀	6.2	12.6	16.2
4	Ni-BN10 ^{HT}	2.1	3.1	3.6
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-BN ₁₀	8.6×10^{-2}	8.7×10^{-2}	7.5×10^{-2}
4	Ni-BN ₁₀ ^{HT}	2.9×10^{-2}	2.2×10^{-2}	1.7×10^{-2}
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-BN ₁₀	0.12×10^2	0.11×10^2	0.13×10^2
4	Ni-BN ₁₀ ^{HT}	0.35×10^2	0.46×10^2	0.59×10^2

A picture of initial non-wear texture and working texture after 900 abrasion cycles of nickel coating with nano-sized cBN₁₀ particles after heat treatment is presented in Fig. 6.

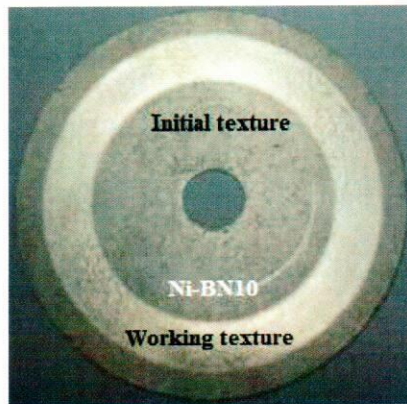


Fig. 6. Optical image of the initial and working texture on the tested coated discs (coating Ni-cBN₁₀)

The wear loss curves of particulate free Ni-P coatings without heat treatment and after heat treatment are graphically constructed in Fig. 7 using the results of Table 3. The wear loss results of Ni-P-cBN₁₀ coatings (with nanoparticles) without heat treatment and after heat treatment are graphically constructed in Fig. 8 using the results of the same table.

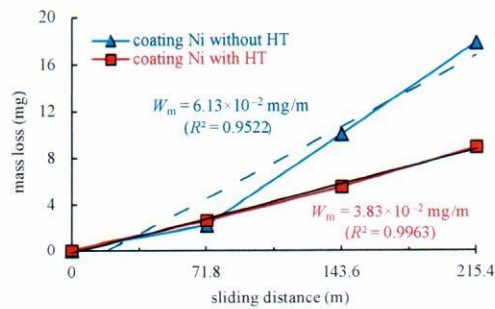


Fig. 7. Mass loss versus sliding distance for coatings without nanoparticles (without and with heat treatment)

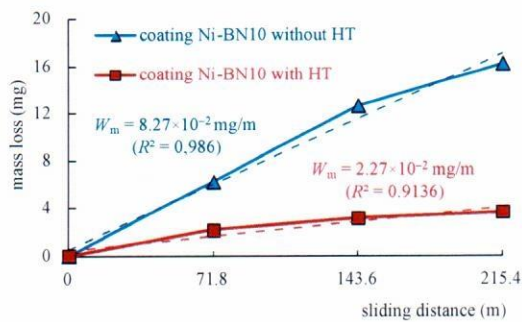


Fig. 8. Mass loss versus sliding distance for coatings containing cBN₁₀ nanoparticles (without and with heat treatment)

The curves of wear rates of particulate free Ni-P coatings without heat treatment and after heat treatment are shown in Fig. 9. The curves of wear rates of Ni-P-cBN₁₀ coatings (containing nanoparticles) without heat treatment and after heat treatment are shown in Fig. 10.

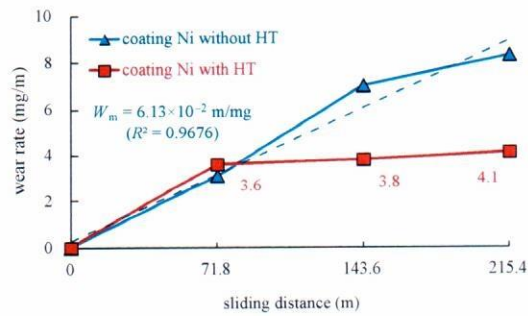


Fig. 9. Wear rate versus sliding distance for coatings without nanoparticles (without and with heat treatment)

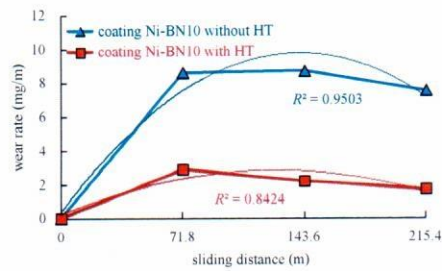


Fig. 10. Wear rate versus sliding distance for coatings containing cBN₁₀ nanoparticles (without and with heat treatment)

The results clearly show that the presence of nanoparticles cBN₁₀ results in a reduction in abrasive mass loss and gives stability to wear rate and wear resistance curves. Heat treatment also reduces abrasive mass loss. The lowest wear rate $W = 2.9 \times 10^{-2}$ mg/m in stationary mode ($N = 900$ cycles) has Ni-P-cBN₁₀ coatings (containing nanoparticles) after heat treatment (Ni-BN₁₀^{HT}). The increase in abrasion wear resistance of this coating is about 4.9 times over the nanoparticles free Ni-P coating and without heat treatment (Table 3). The wear process is steady to the studied friction path range (Fig. 9).

The influence of heat treatment and the addition of nanoparticles cBN₁₀ on abrasive wear resistance by lubrication with LITEL-U-2 grease is analysed separately and together. This is done by comparing the tested coatings by the 'relative wear resistance' (R), which is calculated by equation (2).

In order to analyse the effect of the heat treatment two of the coatings without heat treatment (samples Nos 1 and 3) are considered to be of reference with a

relative wear resistance $R = 1$. The relative wear resistance of the heat treatment specimens (samples Nos 2 and 4) shall be calculated against them.

When analysing the influence of nanoparticles, two of the particulate free Ni-P coatings with and after heat treatment (specimens Nos 1 and 2) are considered to be of reference with a relative wear resistance $R = 1$. The relative wear resistance of the heat treatment specimens (samples Nos 3 and 4) is calculated against them. When analysing the combined effect of heat treatment and the addition of nanoparticles for reference is accepted the nanoparticle free Ni-P coating without heat treatment (specimen No 1) which is considered with a relative wear resistance $R = 1$. The relative wear resistance of all coatings is determined with respect to it.

Calculated values of relative wear resistance are presented in Table 4.

Table 4. Relative wear resistance and the influences of heat treatment and addition of boron nitride nanoparticles on the increase of abrasive wear resistance of tested coatings

Sample	Coatings	Wear resistance (900 cycles) (m/mg)	Relative wear resistance (R)		
			influence of heat treatment	influence of particles addi- tion	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	$R_{2,1} = 2.02$	$R_{2,2} = 1$	$R_{2,1} = 2.03$
3	Ni-BN ¹⁰	0.13×10^2	$R_{3,3} = 1$	$R_{3,1} = 1.1$	$R_{3,1} = 1.1$
4	Ni-BN ₁₀ ^{HT}	0.59×10^2	$R_{4,3} = 4.42$	$R_{4,2} = 2.4$	$R_{4,1} = 4.9$

The diagrams below show the influence of the heat treatment process (Fig. 11), the influence of the nanoparticles addition (Fig. 12) and the double influence – both of the nanoparticles addition and the heat treatment process (Fig. 13) on the abrasion resistance of the tested coatings in order to visualise the results shown in Table 4.

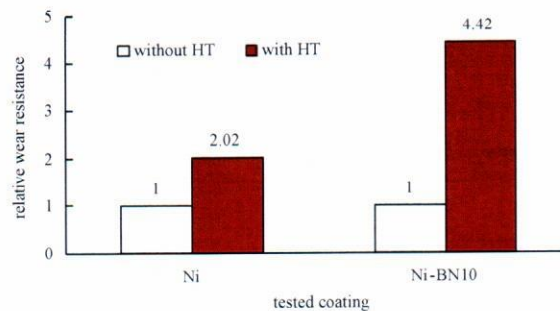


Fig. 11. Influence of heat treatment (HT) on the wear resistance of tested coatings

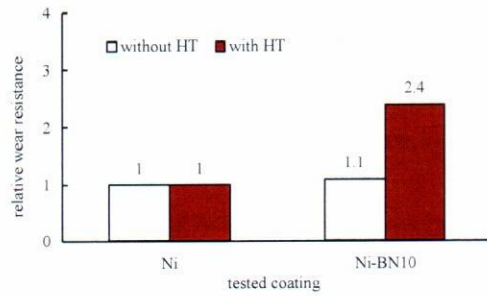


Fig. 12. Influence of nanoparticles addition on the wear resistance of tested coatings

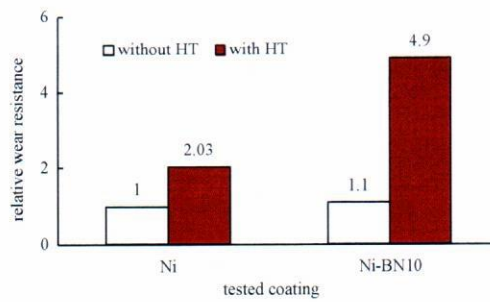


Fig. 13. Influence of heat treatment (HT) and nanoparticles addition on the abrasive wear resistance of tested coatings

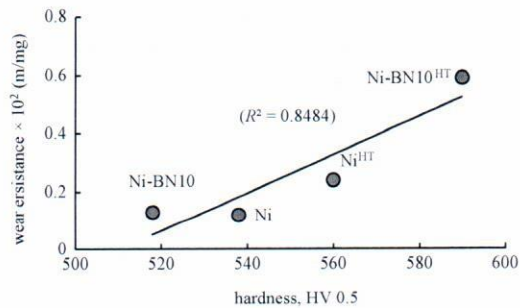


Fig. 14. Wear resistance vs. hardness of tested coatings

The analysis of the results shows that heat treatment increases the wear resistance much more than the addition of nanoparticles. Heat treatment coatings show 2 times higher wear resistance compared to the coatings without heat treatment. The heat treatment of coatings containing nanoparticles results in an increase in wear resistance of 4.42 times (Fig. 11). The presence of cBN₁₀ nanoparticles also results in an increase in wear resistance but less than the heat treatment process. The increase in the wear resistance of coatings containing nanoparticles is 2.4 times higher compared to the same without nanoparticles.

The combined influence of nanoparticle addition and heat treatment process achieves the best effect on the increase in wear resistance (Fig. 13). Nevertheless, both influences separately achieve a significant increase in wear resistance.

Figure 14 represents graphically the relationship between the abrasion resistance and the hardness of the tested coatings (Table 2). Previous studies by the authors of the same coatings in other friction modes (no lubricant, different load values and number of friction cycles) strongly suggest that there is no direct relationship between the abrasion resistance and the hardness of the coatings⁷. In the present study, using lubricant (grease LITEL-U-2), there is a tendency to increase the wear resistance with increasing hardness of the coatings, but this relationship is not a linear.

CONCLUSIONS

1. The preparation of composite dispersive reinforced surface layers with incorporated nano particles of cubic boron nitride (cBN_{10}) are registered using the scanning electron microscopy (SEM) and the energy dispersive X-ray microanalysis (EDX). The nanoparticles within obtained dispersive reinforced coatings are deposited by electroless nickel plating.

2. It was confirmed the effect of low temperature heat treatment (300°C at 6 h) of the obtained containing cubic boron nitride (cBN_{10}) nanoparticles surface coatings deposited by electroless nickel plating.

3. It was confirmed that the heat treatment of deposited electroless nickel coatings have finer (probably nano) crystalline structure compared to the non-treated samples. The recrystallization ongoing processes during the heat treatment were related to more nodules formation.

4. In the comparative analysis of obtained dispersive reinforced composite coatings it was found that both of them – nanoparticles incorporation and heat treatment process achieve increasing wear resistance. The presence of cBN_{10} nanoparticles also results in an increase in wear resistance but less than the heat treatment process. The heat treatment of nickel-phosphorous coatings with nanoparticles result is greater (4.42 times higher) compared to increase in the wear resistance of coatings containing nanoparticles without heat treatment (2.4 times higher).

5. It has been found that the combined (joint) effect of nanoparticle incorporation and heat treatment achieves the best effect. Those coatings have increased 4.9 times the wear resistance of coatings without nanoparticles and without heat treatment. This is due to the newly formed structure containing Ni_3B , whereby the equilibrium mechanical mixture already contains three phases – Ni, Ni_3P and Ni_3B .

6. In the presence of lubricant (grease LITEL-U-2), a nonlinear increase of the abrasion resistance with increasing of the hardness of the coatings is established, unlike the abrasive wear in dry friction where there was no such dependence.

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