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COMPOSITES THEORY AND PRACTICE

Maria Trzaska*, Grzegorz Cieślak, Anna Mazurek

Institute of Precision Mechanics, ul. Duchnicka 3, 01-796 Warsaw, Poland
**Corresponding author. E-mail: maria.trzaska@imp.edu.pl*

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STRUCTURE AND PROPERTIES OF Ni-P/PTFE COMPOSITE COATINGS PRODUCED BY CHEMICAL REDUCTION METHOD

The paper presents the results of investigations on Ni-P/PTFE alloy composite coatingsproduced by the electroless method. Ni-P coatings were deposited from two baths differing in composition and pH (acidic and alkaline). PTFE powders with a nanometric size of particles was used as the dispersion phase. The coatings were characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM), optical microscopy, roughness measurements and Vickers microhardness measurements. Abrasive wear testing of the Ni-P/PTFE coatings was carried out by friction of the ball at a constant pressure on the sample using a Calotest apparatus. Images of the surface morphology, cross section of the coatings and sample surfaces after wear tests are presented. The coatings are compact and have good adhesion to the steel substrate. Incorporation of the PTFE powder particles in the nickel-phosphorus matrix increases the degree of surface development and hardness of the coating material and increases the wear resistance.

Keywords: alloy coatings, composite layers, Ni-P matrix, teflon, electroless process

STRUKTURA I WŁAŚCIWOŚCI KOMPOZYTOWYCH POWŁOK Ni-P/PTFE WYTWARZANYCH METODĄ REDUKCJI CHEMICZNEJ

W pracy przedstawiono wyniki badań stopowych powłok kompozytowych Ni-P/PTFE wytwarzanych metodą bezprądową. Powłoki Ni-P osadzano z dwóch kąpieli różniących się składem oraz pH (kwaśna oraz zasadowa) na stali. Jako fazę dyspersyjną stosowano proszek politetrafluoroetylenu (PTFE). Powłoki były badane za pomocą następujących technik badawczych: skaningowej mikroskopii elektronowej (SEM), dyfrakcji promieniowania rentgenowskiego (XRD), mikroskopii optycznej, pomiarów mikrotwardości metodą Vickersa, pomiarów parametrów chropowatości powierzchni. Badania zużycia ściernego powłok Ni-P/PTFE realizowano za pomocą kulotestera przy stałym nacisku kulki na badaną próbkę. Przedstawiono obrazy morfologii powierzchni, przekrojów poprzecznych powłok i powierzchni po badaniach zużycia ściernego. Wytworzone powłoki charakteryzują się zwartą budową i dobrą przyczepnością do stalowego podłoża. Wbudowanie cząstek proszku PTFE w niklowo-fosforową osnowę wpływa na zwiększenie stopnia rozwinięcia powierzchni, twardości materiału powłok oraz zwiększenie odporności na zużycie ścierne.

Słowa kluczowe: powłoki stopowe, warstwy kompozytowe, osnowa Ni-P, teflon, bezprądowe osadzanie

INTRODUCTION

The surface of metals can be protected by deposited appropriate coatings. Presently, such type of technologies is one of the most important sectors of surface engineering. The process of coating deposition leads to effective protection of metallic materials against harmful environmental impacts (corrosion, erosion) and also gives new properties to the deposited material, including increased resistance to abrasive wear. Ample opportunities in this area are provided by the alloy layers produced by the electrochemical method, which is based on the processes of electrochemical reduction. The use of such coatings is particularly important for elements with complex shapes and for coating non-metallic elements. The functionality of such coatings may be further enhanced by embedding dispersion phase particles in the form of metals, polymers, ceramics, etc. into the alloy matrix. Alloy coatings formed of nickel with phosphorus or boron and deposited by the electroless method play an important role in modern technologies. Alloy coatings based on nickel have a variety of applications, for example, coating metals and non-metal powders [1-3], as well as preparing composite coatings [4-9]. This paper presents the results of studies of composite alloy coatings formed of nickel-phosphorous/teflon (Ni-P/PTFE) produced by the electroless method in baths of different compositions and different pH. The structures and selected properties of the Ni-P alloy coatings and composite nickel-phosphorous/teflon (Ni-P/PTFE) coatings deposited from an alkaline bath (1P-3P) and an acid bath (4P-6P) are presented.

RESEARCH METHODOLOGY

Ni-P alloy and Ni-P/PTFE composite coatings were deposited on a carbon steel substrate by the electroless method. The solution compositions and deposition process conditions are summarized in Table 1. As the dispersion phase, PTFE powder with nanometric sized particles was used at contents of 1 and 2 g/dm^3 in the bath. The topography and morphology of the produced coatings were examined by scanning electron microscopy (SEM). The chemical composition of the matrix material of the composite coatings and the thickness of the coatings were examined by an X-ray fluorescence spectrometer (XRF).

TABLE 1. **Solution compositions and deposition process parameters of Ni-P alloy and Ni-P /PTFE composite coatings**
TABELA 1. **Składy roztworów i parametry procesów osadzania powłok stopowych Ni-P i kompozytowych Ni-P/ PTFE**

Coating solution composition		Process parameters	
1P	0 g/dm^3 PTFE	sodium hypophosphite, nickel sulfate, glycine, PTFE	T = 90°C, pH = 8÷9, t = 5 h
2P	1 g/dm^3 PTFE		
3P	2 g/dm^3 PTFE		
4P	0 g/dm^3 PTFE	sodium hypophosphite, sodium acetate, nickel sulfate, lactic acid, PTFE	T = 90°C, pH = 4.5, t = 5 h
5P	1 g/dm^3 PTFE		
6P	2 g/dm^3 PTFE		

The structure of the prepared coatings was analyzed by X-ray diffraction. The thickness of the coatings and their structures were assessed by analyzing metallographic specimens in sections perpendicular to the surface using a VHX5000 Keyence digital microscope. The microhardness on cross sections of the coatings was examined by the Vickers method at a 10 G load (HV 0.01) by a T1202 Wilson-Hardness apparatus.

Studies of surface roughness were performed using a roughness tester, SJ-210P Mitutoyo. Abrasive wear of the coating materials was tested by friction using Calotest used for the study of thin films and surface coatings [10-12]. The Calotest system consisted of a test sample, countersample in the form of a sphere 30 mm in diameter of LH15 bearing steel, a rotary roller, engine and tilting table. Tribological tests were carried out under dry friction at a constant pressure of 0.37 N and a ball rotational speed equal to 720 rpm for 3600 s. The test was carried out in air atmosphere at a temperature of 23 ±3°C. The wear depth of the tested coatings was taken into account as a criterion for the wear resistance.

RESEARCH RESULTS

The Ni-P alloy and Ni-P/PTFE composite coatings were produced by the electroless method on a carbon steel S235JR substrate. Two varieties of Ni-P coatings were prepared with different component configurations and solution concentrations of which they were produced. For the production of Ni-PTFE composite coatings, a polydisperse PTFE powder with nanometric particles sizes was used. Images of the PTFE powder are shown in Figure 1.

The results of X-ray analysis of the Ni-P alloy coatings are shown in Figure 2.

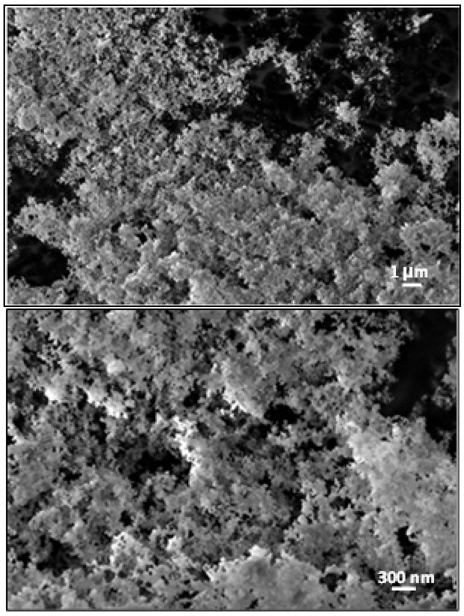

Fig. 1. Images of PTFE powder particles

Rys. 1. Obrazy cząstek proszku PTFE

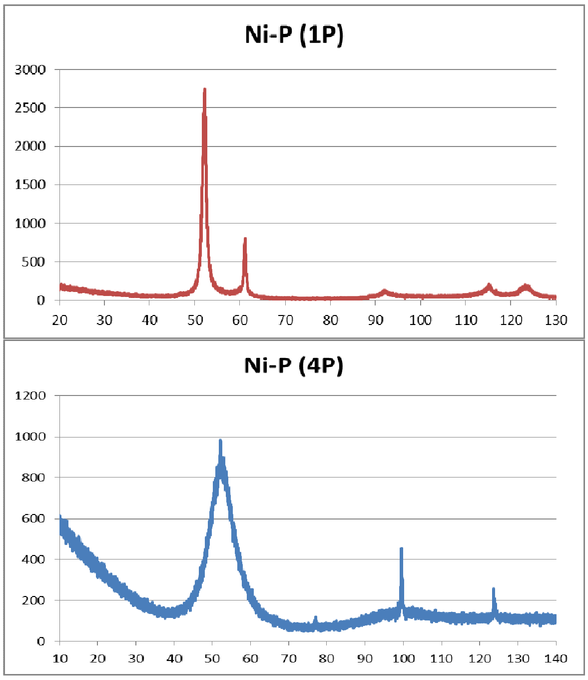

Fig. 2. Diffraction patterns of Ni-P alloy coatings

Rys. 2. Dyfraktogramy powłok stopowych Ni-P

The diffraction patterns of the Ni-P (1P) coating deposited from the alkaline bath exhibit peaks characteristic of nanocrystalline structures. The Ni-P coating (4P) deposited from the acid bath is characterized by an amorphous structure. Images of the nickel-phosphorus alloy and Ni-P/PTFE surfaces produced in baths of different composition and varying contents of PTFE powder are shown in Figures 3-5.

The chemical analysis results of the Ni-P alloy matrix materials, Ni-P coating thickness and efficiency of the deposition processes are summarized in Table 2.

The content of the alloy P component has an influence on the structure and properties of the deposited Ni-P coatings. The PTFE dispersion phase contained in the bath and its incorporation in the deposited coating affects the efficiency of the deposition process of the Ni-P/PTFE coating which determines the thickness of the forming coating. The greatest thickness for the given parameters of the deposition process was exhibited by the Ni-P coating without the addition of PTFE dispersion phase. The amount of PTFE embedded in the nickel-phosphorus matrix was examined by an EDS analyzer. For the coatings deposited from the alkaline bath, the fluorine content in the coating equals 3% by weight, while in the coatings deposited from the acid bath it is 1.5% by weight.

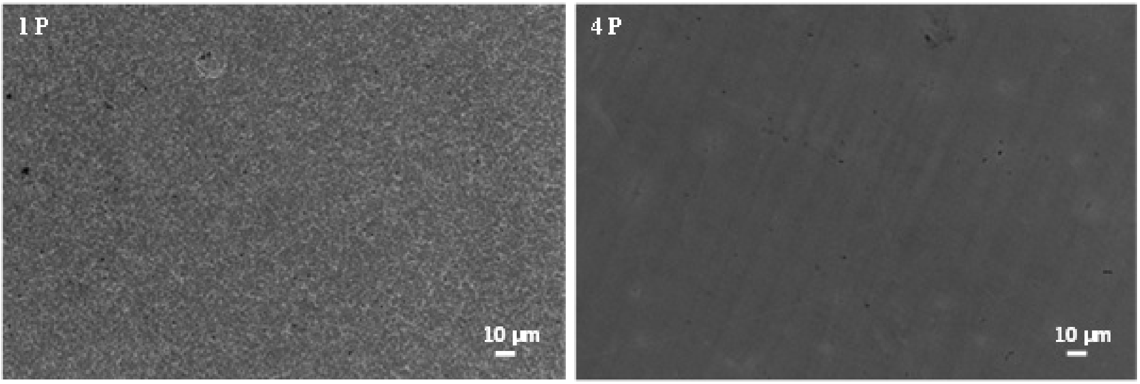

Fig. 3. Topography of Ni-P alloy coating surface

Rys. 3. Topografia powierzchni powłok stopowych Ni-P

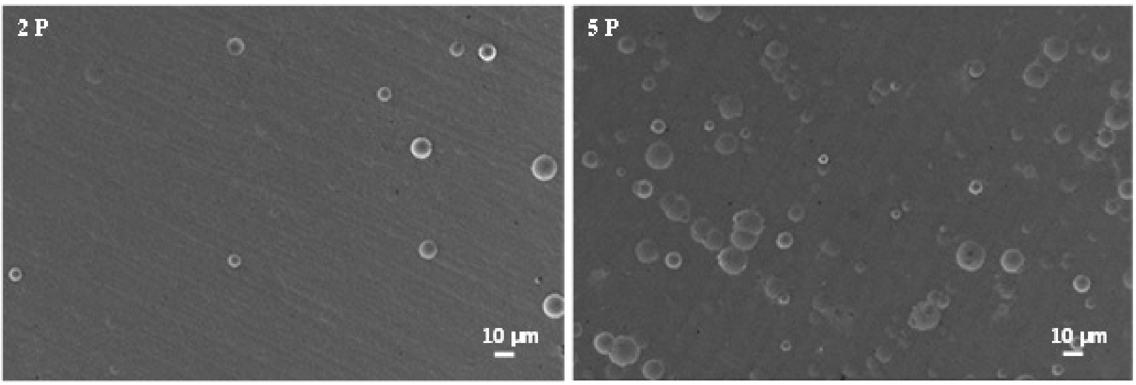

Fig. 4. Topography of Ni-P composite coating surface containing 1 g /dm^3 PTFE in bath

Rys. 4. Topografia powierzchni powłok kompozytowych Ni-P/PTFE o zawartości PTFE 1 g/dm^3 w kąpieli

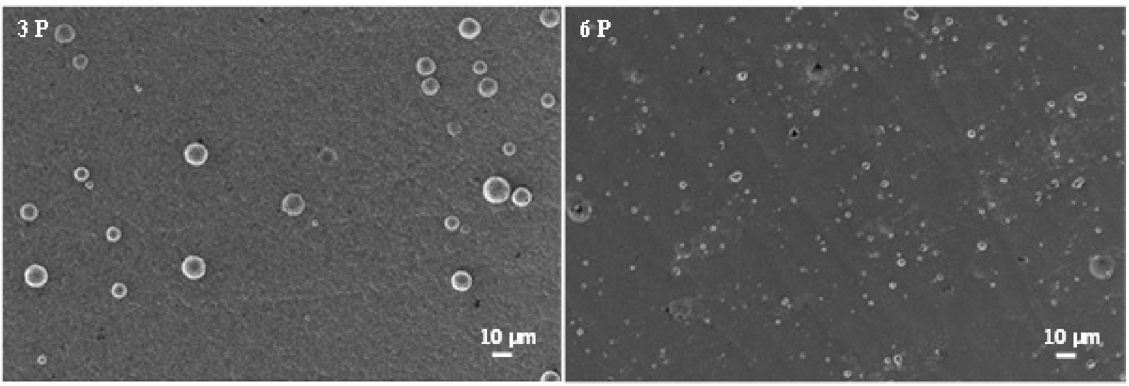

Fig. 5. Topography of Ni-P composite coating surface containing 2 g /dm^3 PTFE in bath

Rys. 5. Topografia powierzchni powłok kompozytowych Ni-P/PTFE o zawartości PTFE 2 g/dm^3 w kąpieli

TABLE 2. **Chemical composition and thickness of Ni-P, Ni-P / PTFE coatings and efficiency of deposition processes**
TABELA 2. **Skład chemiczny i grubość powłok Ni-P, Ni-P/ PTFE oraz wydajność procesów osadzania**

Coating	Thickness [µm]	Standard deviation	Efficiency of deposition process [%]	Chemical composition of matrix material			
				P [% wt.]	Standard deviation	Ni [% wt.]	Standard deviation
1P	41.34	0.19	100.00	3.7	0.08	96.3	0.076
2P	14.27	0.19	34.52				
3P	12.56	0.12	30.38				
4P	46.35	0.39	100.00	10.1	0.15	89.9	0.15
5P	18.04	0.08	38.92				
6P	19.78	0.21	42.68				

The Ni-P and Ni-P/PTFE coating structures in cross sections perpendicular to the surface are shown in Figures 6 and 7. All the produced coatings are compact and have a uniform thickness over the entire coated surface and a good connection with the substrate material.

The study of microhardness and roughness of the produced Ni-P and Ni-P/PTFE coatings are summarized in Table 3. The Ni-P (1P) and Ni-P/PTFE coatings (2P, 3P) deposited from the alkaline bath have a higher hardness and much greater degree of surface development when compared to the Ni-P/PTFE (4P, 5P, 6P) coatings deposited in an acid bath. The incorporation of PTFE particles increases the degree of surface development of the tested coatings. In the case of coatings (1P, 2P, 3P) deposited from an alkaline bath, the incorporation of PTFE particles has no significant effect on the hardness of the coating materials. However, in the case of coatings (4P, 5P, 6P), the incorporation of PTFE particles causes an increase in hardness of the coating materials.

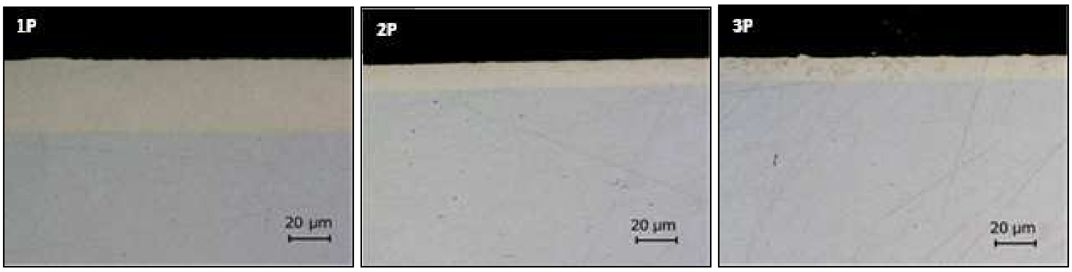

Fig. 6. Structures in cross sections of Ni-P (1P) and Ni-P/PTFE (2P, 3P) coatings deposited from alkaline bath
Rys. 6. Przekroje poprzeczne powłok Ni-P(1P) oraz Ni-P/PTFE (2P, 3P) osadzanych z kąpieli alkalicznej

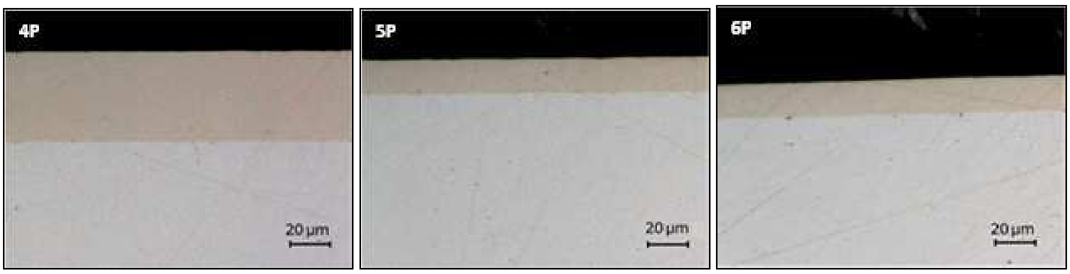

Fig. 7. Structures in cross sections of Ni-P (4P), and Ni-P/PTFE coatings (5P, 6P) deposited from acid bath
Rys. 7. Przekroje poprzeczne powłok Ni-P(4P) oraz Ni-P/PTFE (5P, 6P) osadzanych z kąpieli kwaśnej

TABLE 3. **Microhardness and roughness of Ni-P and Ni-P/PTFE coating surfaces**
TABELA 3. **Mikrotwardość i chropowatość powierzchni powłok Ni-P oraz Ni-P/PTFE**

Material	HV [0.01 G]	Roughness			
		Ra [µm]	Standard deviation	Rz [µm]	Standard deviation
Substrate	143	0.056	0.01	0.425	0.09
1P	869	0.157	0.01	1.064	0.05
2P	871	0.139	0.01	1.262	0.06
3P	884	0.413	0.01	4.264	0.09
4P	549	0.061	0.01	0.395	0.08
5P	600	0.078	0.01	0.583	0.05
6P	637	0.113	0.02	1.315	0.08

The wear tests of the coating materials by friction were carried out with a constant ball pressure on the sample. The change in the specific pressure during the tribological trial of the tested coatings are shown in Figure 8.

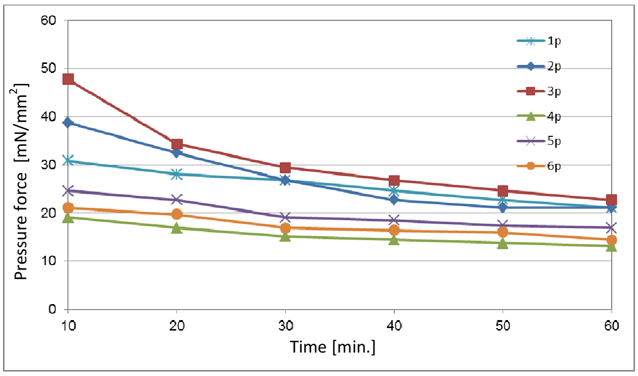

Fig. 8. Unit pressure during tribological test of investigated coatings

Rys. 8. Zmiana nacisku jednostkowego podczas próby tribologicznej badanych powłok

The tribological wear track depth expressed as a function of the test time is shown in Figure 9.

The abrasive wear of the test coatings proceeded with a different intensity and for all the coatings it increased monotonically with the tribological test time. Analysis of the wear graphs show that coatings (1P, 2P, 3P) deposited from the alkaline bath are characterized by greater wear resistance comparing with coatings (4P, 5P, 6P) deposited from the acid bath. The incorporation of PTFE particles has an effect of improving the tribological properties; the coatings with the embedded dispersion phase wear out slower than the coatings without the embedded PTFE particles.

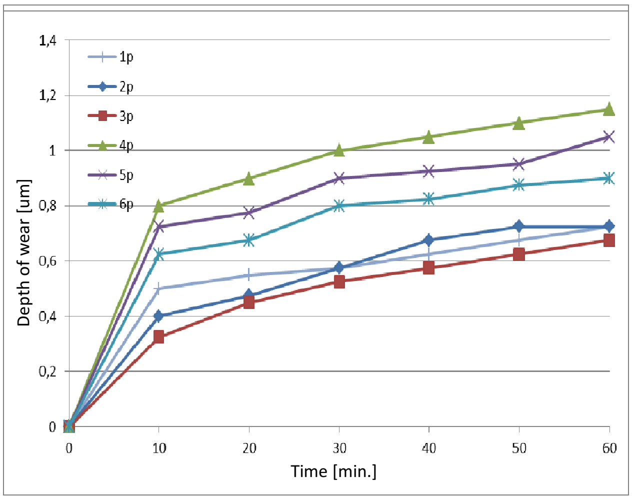

Fig. 9. Abrasive wear of investigated coating materials

Rys. 9. Zużycie ścierne materiałów badanych powłok

Images of the Ni-P and Ni-P/PTFE coating surfaces after abrasive wear tests are shown in Figures 10 and 11. Analysis of the wear signs showed that as a result of the abrasion test, there is a loss in mass of the coating material. In the wear areas , there was no delamination of the coatings from the steel substrate nor discontinuities in the coatings.

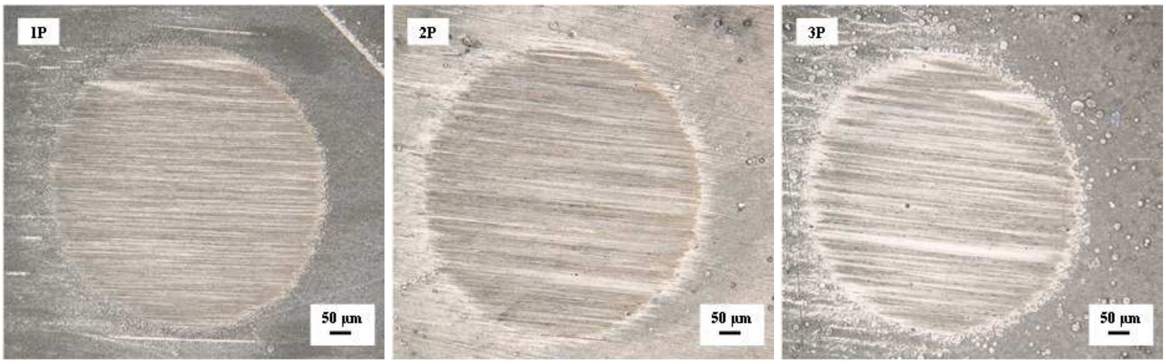

Fig. 10. Surface of Ni-P (1P) and Ni-P / PTFE (2P, 3P) coatings after tribological tests

Rys. 10. Powierzchnia powłok Ni-P (1P) i Ni-P/PTFE (2P, 3P) po badaniach tribologicznych

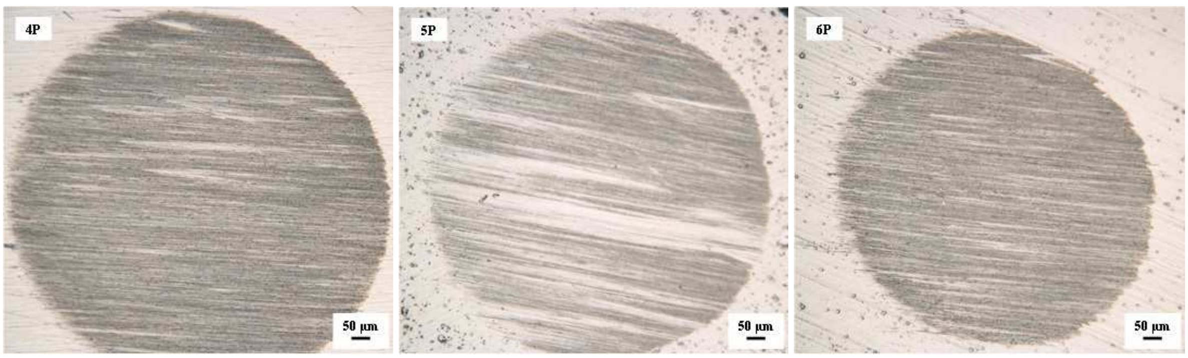

Fig. 11. Surface of Ni-P (4P) and Ni-P / PTFE (5P, 6P) coatings after tribological tests

Rys. 11. Powierzchnia powłok Ni-P (4P) i Ni-P/PTFE (5P, 6P) po badaniach tribologicznych

CONCLUSIONS

Ni-P alloy coatings can be deposited from baths of various compositions at different process parameters. The bath composition and deposition process parameters affect the chemical composition, structure and material properties of the deposited Ni-P alloy coatings. Polydisperse PTFE powder of nanometric dimensions can be used as a disperse phase built into a nickel--phosphorus matrix. The deposition processes of Ni-P/PTFE coatings have a lower yield of the process compared to the Ni-P coating deposition process. The coatings with embedded PTFE powder particles exhibit greater hardness and higher wear resistance compared to the Ni-P coatings without embedded PTFE particles.

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