Polish Society of Composite Materials
Polskie Towarzystwo Materiałów Kompozytowych
16: 1 (2016) 37-41
COMPOSITES THEORY AND PRACTICE

Maria Trzaska*, Anna Mazurek

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

Received (*Otrzymano*) *29.02.2016*

PROPERTIES OF NANOCOMPOSITE Ni/DIAMOND COATINGS PRODUCED BY THE ELECTROCRYSTALLIZATION METHOD

This paper presents the results of research on the properties of composite coatings with a nickel matrix and diamond nanopowders as the dispersed phase. The coating deposition processes were carried out in a Watts bath containing organic compounds and dispersion particles in the form of nanodiamond powder. The characteristics of the dispersed diamond phase were determined using a transmission electron microscope and X-ray diffraction identifications. For comparison purposes, the research also covered pure nickel coatings with nanocrystalline structures. The morphology, topography and chemical composition of the produced coatings were analysed using a scanning electron microscope equipped with an EDS detector. The microhardness of the nickel and composite coatings were measured using a Vickers microhardness tester. The tribological properties of the manufactured coatings were studied using a Calotest. The structures of the produced coatings were found to be compact. Compared to the nickel coatings, the Ni/diamond composite coatings were found to have greater microhardness and stronger abrasive wear resistance.

Keywords: **composite nanocoatings, Ni/diamond, diamond powder, electrocrystallization method**

WŁAŚCIWOŚCI NANOKOMPOZYTOWYCH POWŁOK Ni/DIAMENT WYTWARZANYCH METODĄ ELEKTROKRYSTALIZACJI

W pracy przedstawiono wyniki badań powłok kompozytowych z osnową niklową i diamentem jako fazą dyspersyjną. Proces osadzania powłok prowadzono w kąpieli Wattsa z dodatkiem związków organicznych oraz cząstek dyspersyjnych w postaci proszku nanodiamentu. Charakterystykę dyspersyjnej fazy diamentowej określono za pomocą transmisyjnego mikroskopu elektronowego oraz dyfrakcji promieniowania rentgenowskiego. W celach porównawczych badania obejmowały również powłoki niklowe o nanokrystalicznej strukturze. Morfologię, topografię oraz skład chemiczny wytworzonych powłok analizowano za pomocą skaningowego mikroskopu elektronowego wyposażonego w przystawkę analityczną EDS. Mikrotwardość powłok niklowych i kompozytowych wyznaczano za pomocą mikrotwardościomierza Vickersa. Właściwości tribologiczne wytworzonych powłok badano za pomocą kulotestera. Wytworzone powłoki charakteryzowały się zwartą budową. Powłoki kompozytowe Ni/diament w porównaniu do powłok niklowych cechowały się większą mikrotwardością i większą odpornością na zużycie ścierne.

Słowa kluczowe: **powłoki nanokompozytowe, Ni/diament, proszek diamentowy, elektrokrystalizacja**

INTRODUCTION

Electrocrystallization is a surface engineering process applied to obtain new materials with the ability to design their desired properties. The available literature on the subject indicates that very advantageous properties can be obtained by co-depositing non-metallic phase particles together with metal particles in composite coatings when comparing them to basic metallic coatings [1-3]. By appropriate selection of the matrix material and disperse phase, composite coatings with properties adapted to specific needs can be thus produced. The research carried out within the framework of this paper relates to electrochemically produced nanocrystalline composite coatings with a nickel matrix and a disperse phase in the form of diamond nanopowders. The use of diamond powders enables the production of new composite materials with advantageous properties, which are characterized by unique useful properties such as high hardness, high thermal conductivity and chemical resistance to various acids and bases [4-7].

EXPERIMENTAL PART

Nickel and Ni/diamond composite coatings were deposited using electrocrystallization processes realized in a compound solution containing nickel(II) sulfate(VI), nickel(II) chloride, boric acid and organic compound additives. Carbon steel S235JR was used as the substrate material. For the production of composite coatings, diamond nanopowder was used with the various contents in a bath (1 and 2 g/dm^3). In order to prevent sedimentation of the diamond powder during coating deposition, the electrolyte solution was stirred with a mechanical stirrer at a speed of 50 RPM. The electrodeposition process was carried out at 45°C and at a current density of 3 A/dm^2.

The structural characteristics of the dispersed phase and the produced coatings were determined by X-ray diffraction analysis using a Philips PW-1830 X-ray diffractometer and transmission electron microscopy (TEM). The morphology of the surface and chemical composition of the produced coatings was analysed using a Zeiss LEO 435VP scanning electron microscope with an EDS detector. The degree of surface development of the coatings was analysed with a Mitutoyo Surftest SJ-210P surface roughness tester. The tribological properties of the produced coatings were analysed using a Calotest. The testing system was composed of the tested sample and a steel bearing ball with a diameter of 30 mm placed on the rotating roll. Testing was conducted with constant pressure of the ball on the sample (0.37 N) and the rotation speed of the ball was fixed at 720 RPM. The test was realized in air atmosphere [8, 9].

TEST RESULTS

JSC Sinta diamond powder was used to produce the composite coatings. A TEM image of the diamond nanoparticles is shown in Figure 1.

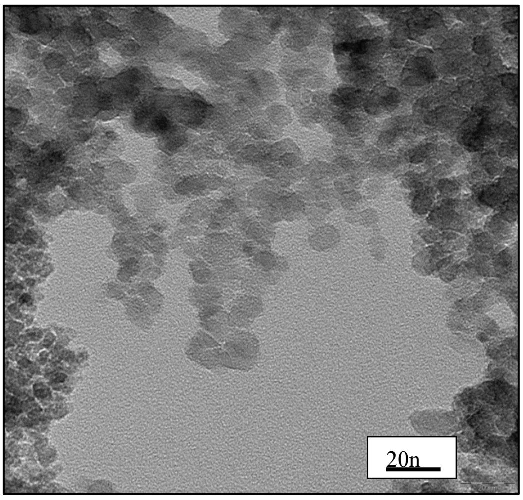

Fig. 1. TEM image of diamond nanoparticles
Rys. 1. Obraz TEM cząstek diamentu

The nanodiamond powder used to produce the composite coatings is characterized by a small variation in nanoparticle sizes within the range of 5 nm. The results of analysis of the X-ray diffraction of the diamond nanoparticles are shown in Figure 2.

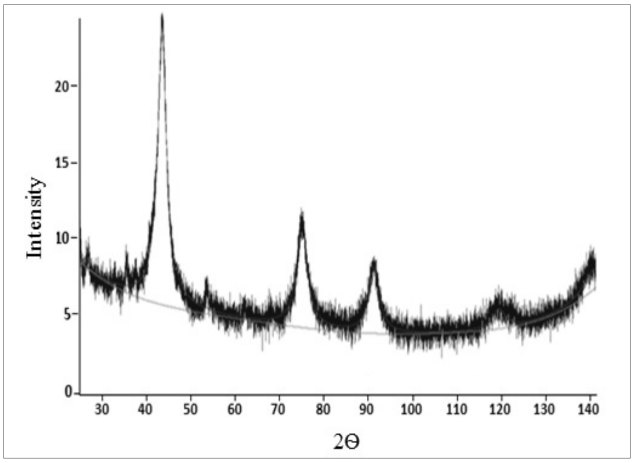

Fig. 2. X-ray diffraction pattern of used diamond nanoparticles
Rys. 2. Dyfraktogram rentgenowski stosowanych cząstek diamentu

The extended reflections on the diffraction pattern shown in Figure 2 confirm the nanocrystalline structure of the diamond powder used. The diffraction pattern of nickel deposited by the electrocrystallization process in the electrolyte solutions is shown in Figure 3.

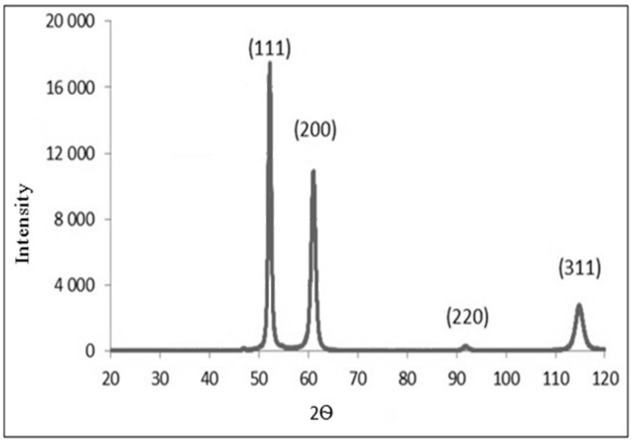

Fig. 3. X-ray diffraction pattern of produced Ni coatings
Rys. 3. Dyfraktogram rentgenowski wytworzonych powłok Ni

The extension of the reflections on the diffraction pattern of the nickel coating demonstrates its nanocrystalline structure. Images of the surface topography of the produced nickel and composite coatings with different concentration of diamond dispersed phase are shown in Figure 4.

The structures of the coatings in their cross sections perpendicular to the sample surface are shown in Figure 5.

The coatings produced in electrocrystallization processes are characterized by good adherence to the substrate material and exhibit compact structures. No coating flakings or cracks were observed. Incorporation of the dispersed phase in the form of diamond nanoparticles in the nickel matrix is evidenced by the results of the chemical composition of the composite coatings shown in Figures 6 and 7.

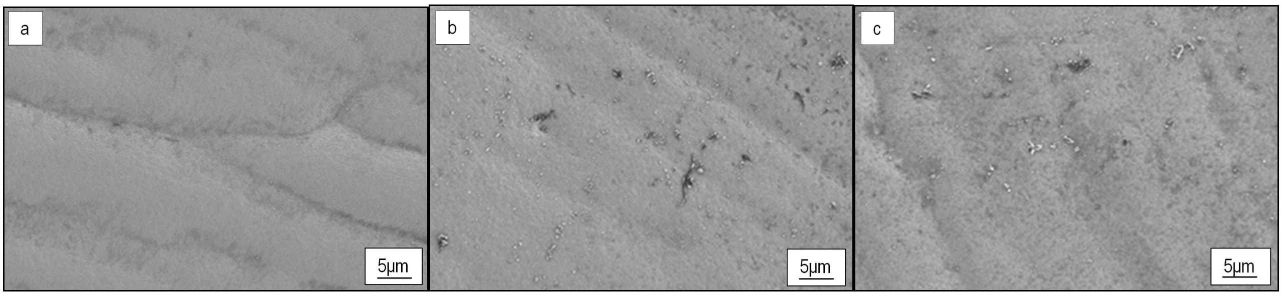

Fig. 4. SEM images of surface topography of produced coatings: a) Ni, b) Ni/diamond (1 g/dm^3), c) Ni/diamond (2 g/dm^3)

Rys. 4. Obrazy SEM powierzchni powłok: a) Ni, b) Ni/diament (1 g/dm^3), c) Ni/diament (2 g/dm^3)

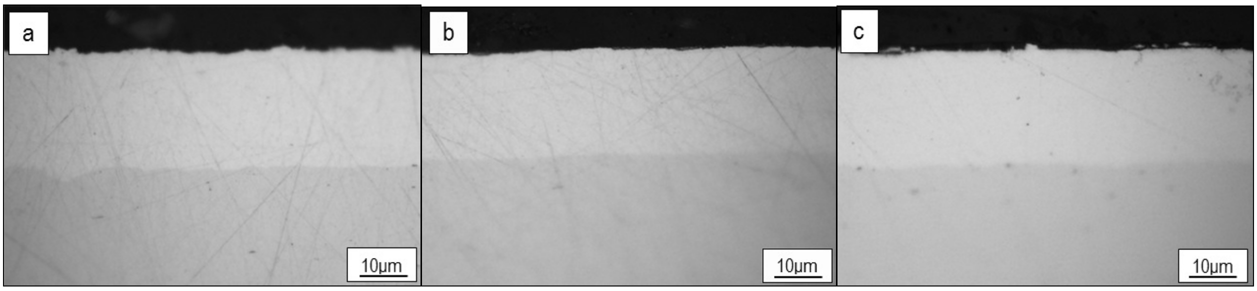

Fig. 5. Cross-sections of produced coatings: a) Ni, b) Ni/diamond (1 g/dm^3), c) Ni/diamond (2 g/dm^3)

Rys. 5. Przekroje poprzeczne wytworzonych powłok: a) Ni, b) Ni/diament (1 g/dm^3), c) Ni/diament (2 g/dm^3)

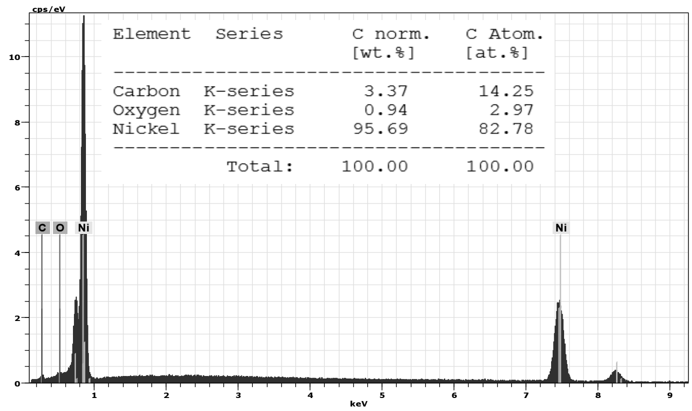

Fig. 6. Chemical composition of Ni/diamond (1 g/dm^3) coating

Rys. 6. Skład chemiczny powłoki Ni/diament (1 g/dm^3)

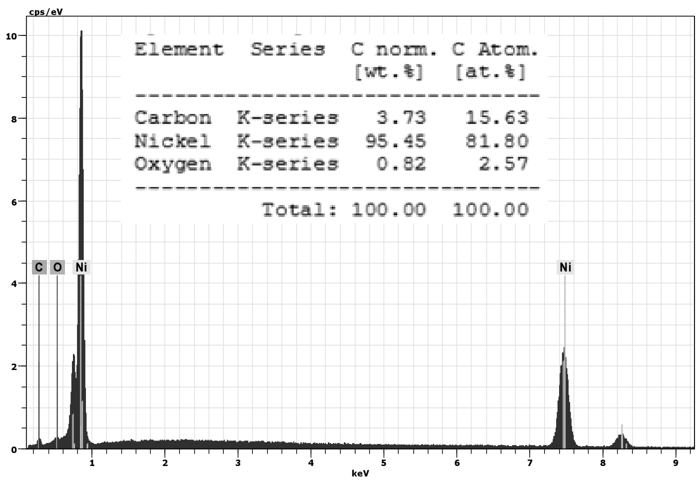

Fig. 7. Chemical composition of Ni/diamond (2 g/dm^3) coating

Rys. 7. Skład chemiczny powłoki Ni/diament (2 g/dm^3)

With an increase in diamond content in the bath, the quantity of embedded particles in the coating also increased. The results of the analysis of the surface roughness of the produced coatings are shown in Table 1.

TABLE 1. **Roughness of substrate and produced coatings**
TABELA 1. **Chropowatość materiału podłoża oraz wytworzonych powłok**

Material	Roughness *Ra* [μm]
substrate	0.118
Ni	0.126
Ni/diamond (1 g/dm^3)	0.132
Ni/diamond (2 g/dm^3)	0.137

The surface roughness measurements indicate that the addition of diamond powder over the range of concentrations in the electrolyte had no significant impact on the surface roughness of the produced coatings. The results of the measurement of the microhardness of the substrate material and the coatings are shown in Table 2.

TABLE 2. **Microhardness of substrate and produced coatings**
TABELA 2. **Mikrotwardość materiału podłoża oraz wytworzonych powłok**

Material	Microhardness *HV* (0.025)
substrate	170
Ni	457
Ni/diamond (1 g/dm^3)	470
Ni/diamond (2 g/dm^3)	495

The composite coatings show greater microhardnesses compared to the nickel coatings. The increase in diamond concentration incorporated in the nickel matrix improves the microhardness of the composite coatings.

The Calotest was used to measure the abrasive wear of the produced coatings. Based on the diameter of the abrasion traces, the depth of abrasion was calculated, which was applied as a measure of abrasive wear resistance. Images of the abrasion traces on the coating surface after tribological tests are shown in Figure 8. Figure 9 shows the change in unit pressure during the tribology test, whereas the abrasive wear of the coating material is shown in Figure 10.

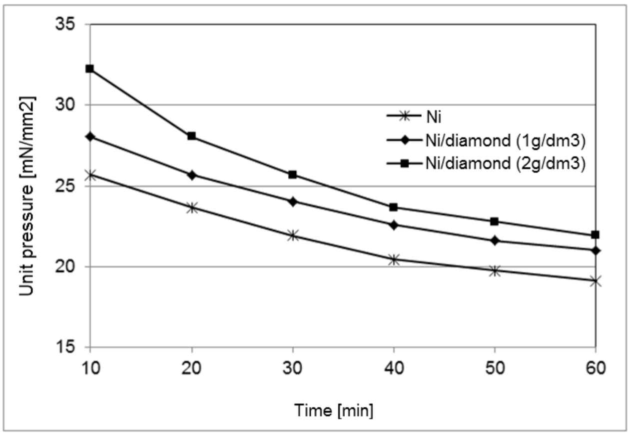

Fig. 9. Evolution of unit pressure during tribological test of investigated coatings

Rys. 9. Zmiana nacisku jednostkowego podczas próby tribologicznej badanych powłok

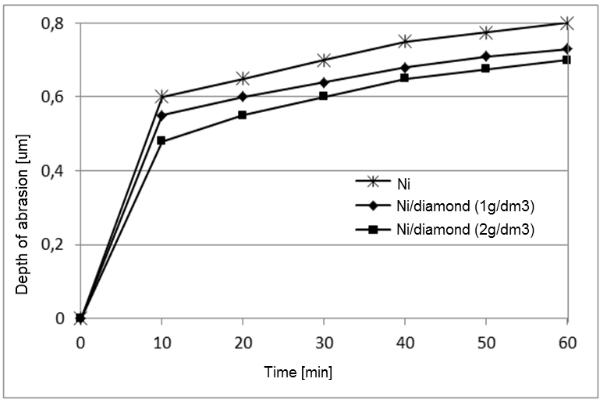

Fig. 10. Abrasive wear of investigated coatings

Rys. 10. Zużycie ścierne materiałów badanych powłok

The results of the tribological tests showed that the Ni/diamond nanocomposite coatings are more resistant to abrasive wear than the nanocrystalline Ni coatings. The composite coatings produced in a bath containing 2 g/dm^3 of diamond powder show the highest resistance to abrasive wear.

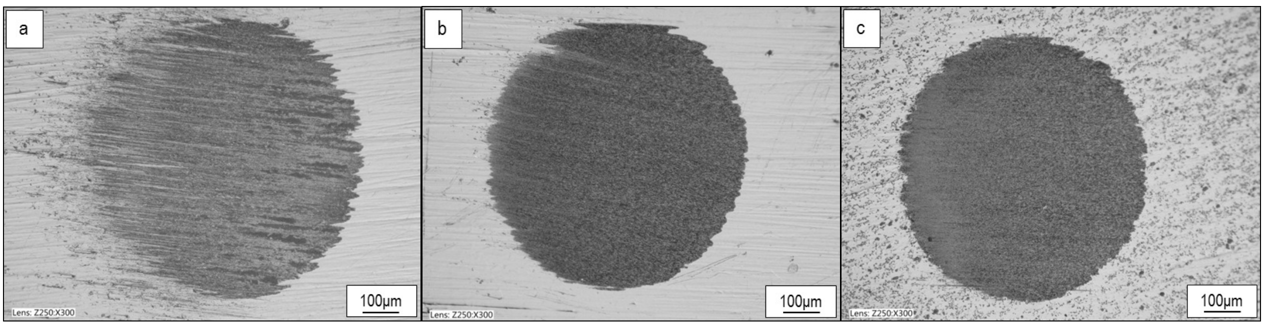

Fig. 8. Images of abrasion traces on coating surface after tribological tests on: a) Ni, b) Ni/diamond (1g/dm^3), c) Ni/diamond (2 g/dm^3)

Rys. 8. Obrazy powierzchni śladu wytarcia powłok po badaniach trybologicznych: a) Ni, b) Ni/diament (1 g/dm^3), c) Ni/diament (2 g/dm^3)

CONCLUSIONS

Nanocrystalline coatings, both nickel and Ni/diamond, produced by electrocrystallization processes are characterized by compact structures, uniform thicknesses, and good adherence to the substrate material. The incorporation of diamond nanoparticles in the nickel matrix improves the microhardness of the coating as well as its resistance to abrasive wear. An increase in diamond concentration in the composite material results in an increase in its hardness and resistance to abrasive wear.

REFERENCES

[1] Trzaska M., Cieślak G., The structure and properties of nanocrystalline Ni/Al_2O_3 layers produced by electrocrystallization, Composites: Theory and Practice 2014, 4, 203-207.

[2] Cieślak G., Mazurek A., Trzaska M., Warstwy kompozytowe Ni/grafen wytwarzane metodą redukcji elektrochemicznej, Inżynieria Powierzchni 2015, 3, 44-47.

[3] Suzuki T., Konno T., Improvement in tool life of electroplated diamond tools by Ni-based carbon nanotube composite coatings, Precision Engineering 2014, 38, 659-665.

[4] Petrov I., Detkov P., Drovosekov A., Ivanov M.V., Tyler T., Shenderova O., Voznecova N.P., Toporov Y.P., Schulz D., Nickel galvanic coatings co-deposited with fractions of detonation nanodiamond, Diamond & Related Materials 2006, 15, 2035-2038.

[5] Burkat G.K., Fujimura T., Dolmatov V.Yu., Orlova E.A., Veretennikova M.V., Preparation of composite electrochemical nickel-diamond and iron-diamond coatings in the presence of detonation synthesis nanodiamonds, Diamond & Related Materials 2005, 14, 1761-1764.

[6] Wang L., Gao Y., Xue Q., Liu H., Xu T., Effects of nanodiamond particles on the structure and tribological property of Ni-matrix nanocomposite coatings, Materials Science and Engineering A 2005, 390, 313-318.

[7] Zhou H., Du N., Zhu L., Shang J., Qian Z., Shen X., Characteristics investigation of Ni-diamond composite electrodeposition, Electrochimica Acta 2015, 151, 157-167.

[8] Betiuk M., Burdyński K., Michalski J., Wach P., Ujawnienie struktury cienkich warstw i powłok w badaniach metalograficznych za pomocą kulotestera, Inżynieria Powierzchni 2007, 3, 70-74.

[9] Trzaska M., Cieślak G., Zużycie tribologiczne nanokrystalicznych powłok wielowarstwowych Ni/Cu, Inżynieria Materiałowa 2015, 2, 87-90.

