Benigna Szeptycka[1], **Anna Gajewska-Midziałek**[2]
Institute of Precision Mechanics, Duchnicka 3, 01-796 Warsaw, Poland

INVESTIGATIONS OF THE WEAR RESISTANCE OF COMPOSITE COATINGS Ni-SiC

In present work the composite electrochemical coatings were prepared with nickel matrix and particles SiC. The nickel plating bath of low nickel ion concentration (0.75 M) containing brightening organic compound with surfactants and the dispersed particle (SiC) was used for electrodeposition of composite coatings. The content of particles in coatings was examined gravimetrically. The dependence of SiC content in composite coatings Ni-SiC on the concentration and kind of the organic additives is given in Table 1. The roughness of the coatings Ni-SiC was measured using tester TR 100 (Tab. 1). Siemens D500 X-ray diffractometer with CuK_α, radiation was used to determine the dimension of nickel [111] crystallites and microstress (Tab. 1). The microhardness of the deposited layers was measured using a Vickers' method at a load of 0.01 and 0.05 kG. Figures 2 and 3 show the microhardness of composite coating Ni-SiC. The wear experiments of Ni-SiC coatings were made without lubrication using the technique based on measuring system comprising a flat surface and a ball. On the basis of the wear traces and measurement of their diameter, the depth of the wear was calculated, which was the measure of wear resistance. The wear of the composite coatings Ni-SiC in the dependence from of the concentration and kind of the organic additives is given in Figure 4.

The obtained results suggest that the organic compounds used in the experiments had a significant effect on the composition and structure of these coatings. This study has shown that the wear resistance is proportional to the microhardness, the roughness, the size of nickel grain and the particle contents in Ni-SiC coatings.

Key words: **electrodeposition, nickel, composite, silicon carbide, wearability**

BADANIA ODPORNOŚCI NA ZUŻYCIE KOMPOZYTOWYCH POWŁOK Ni-SiC

Osadzano elektrochemicznie kompozytowe powłoki z osnową niklową i cząstkami SiC. Do elektroosadzania kompozytowych powłok używano niskostężeniowej kąpieli zawierającej 0,75 M jonów niklu(II), związek organiczny blaskotwórczy, zwilżacze i jako cząstki dyspersyjne SiC. Zawartość masową cząstek w powłoce oznaczano grawimetrycznie. W tabeli 1 przedstawiono zależność zawartości SiC w powłoce kompozytowej Ni-SiC od stężenia i rodzaju dodatków organicznych. Chropowatość warstw Ni-SiC mierzono profilografem TR 100 (tab. 1). Pomiary wielkości krystalitów [111] osnowy niklowej i mikronaprężeń wykonano na dyfraktometrze Siemens D500 z promieniowaniem CuK_α (tab. 1). Mikrotwardość mierzono metodą Vickersa przy obciążeniu 0,01 i 0,05 kG. Na rysunkach 2 i 3 przedstawiono wyniki pomiarów mikrotwardości kompozytowych powłok Ni-SiC. Badania odporności na zużycie wykonano na kulotesterze. Na podstawie śladów wytarcia i pomiarów ich średnicy obliczano głębokość wytarcia, która była miarą odporności na zużycie. Głębokość wytarcia powłok kompozytowych Ni-SiC w zależności od stężenia i rodzaju dodatków organicznych jest pokazana na rysunku 4.

Stwierdzono, że użyte związki organiczne miały znaczący wpływ na skład i strukturę tych warstw. Odporność na zużycie jest proporcjonalna do mikrotwardości, chropowatości, rozmiarów ziaren niklu i zawartości SiC w powłoce.

Słowa kluczowe: **elektroosadzanie, nikiel, kompozyty, węglik krzemu, odporność na zużycie**

INTRODUCTION

For many engineering applications, wear resistance is one of the most important mechanical properties because wear account for more than 50% loss of all materials in service. Interest in electrodeposited composite coatings has increased rapidly due to their expected new engineering applications, ex. wear or oxidizing resistance. Such coatings are produced by codeposition of fine inert particles in a metal matrix from electrolytic bath. Ceramic materials show optimum wear properties considerably greater than the best -hardened steel alloy [1]. Very good thermal resistance, corrosion resistance and inhibition of destruction's caused by adhesion peelings can be numbered as other advantages for ceramic materials.

Kerr [2] published that the optimum inclusion level for SiC in nickel coating deposited from Watts bath (1.3 mM Ni^{2+}) is approximately 12% vol. of ceramic material, for several engineering applications, and further increase in the SiC level may cause the deposit to become brittle. For optimum wear resistance, the deposit hardness must normally be in the range 370 to 615 HV (3.6 to 6 GPa). Lower values tend to give rise to excessive wear rates while higher values result in brittle coatings. For wear purposes, there is a critical relationship

[1] dr inż., [2] mgr inż.

between the coating hardness and inclusion contents in the coating.

In the paper [3] this was showed that the microhardness of the Ni-SiC composite coatings is significantly increased with the embedding of the SiC particles. On the other hand, roughness measurements of the deposits confirmed that the composite coatings have lower roughness values than the corresponding values of pure nickel coatings prepared under the same conditions, a fact that could be associated with their observed microcrystalline structure.

In the present paper, the relationships between: the microstress and the particle contents in the coatings, the microhardness and grain size of nickel matrix, the microhardness, the roughness, the particle contents and abrasive wear resistance of composite Ni-SiC coatings were investigated and discussed.

The objective of presented investigations was to define the effect of bath composition on coating properties (content of particles, microstress, size of nickel grains, wear resistance, microhardness and roughness).

EXPERIMENTAL

The compositions of the nickel plating baths was: 0.45 M $NiSO_4 \cdot 7H_2O$, 0.3 M $NiCl_2 \cdot 6H_2O$, 0.84 M H_3BO_3. The organic compounds used were starting agent SRN, cationic silyl surfactant (SK), anionic high-fluorine surfactant (WFA), cationic high-fluorine surfactant (WFK1) and alkylsulphosuccinate (ASB). To deposit composite coatings SiC1000 technically pure in concentration 5 g SiC/dm^3 of the bath were used. SEM image of SiC particles presents Figure 1. The weight percentage of the dispersed phase in the coatings was determined gravimetrically.

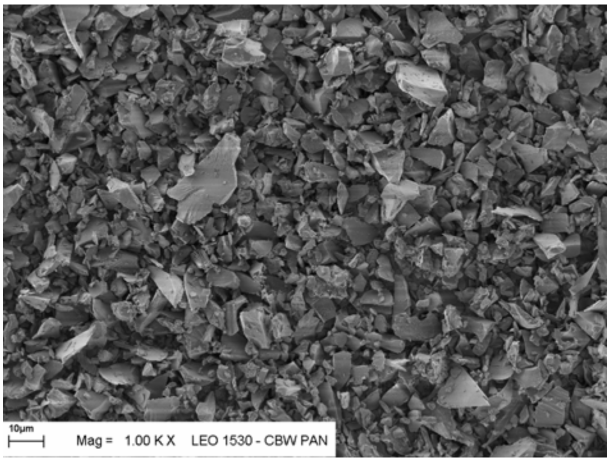

Fig. 1. SEM image of SiC particles

Rys. 1. Obraz SEM cząstek dyspersyjnych SiC

Process has been carried out at current density 4 A/dm^2 at temperature 318 K, pH 4, and duration time 30 min with mechanically stirring.

X-ray diffractometer measurements of deposited layers were made using a Siemens D500 powder diffractometer equipped with a semiconductor Si[Li] detector. CuK_α radiation was used. Average relative deviation of lattice constant (microstress parameter), average grain size were calculated using the Halder and Wagner [4] approach for (111)/(222) planes. The roughness of the nickel foils was evaluated using a TR 100 prod. Elcometr Instruments Ltd. profilograph. The microhardness of the deposited layers was measured using Vickers' method and a Hanneman microhardness tester at a load of 0.01 and 0.05 kG.

In order to determine wear resistance the nickel foils was tested using a technique based on a measuring system comprising a flat surface and ball. The layers were subjected to wear by dry slide friction using a 30 mm diameter ball at an angle of 35°. The pressure of the ball at the point of contact was 0.6 N, the 8000 cycles of friction were applied. The depth of wear as a measure of wear resistance was calculated on the basis of the wear traces and measurements of their diameters.

RESULTS AND DISCUSSION

The additive SRN strongly limited the grain size and SiC content in the coating but wetting agents used in the bath together with this additive not exert greater influence (Tab. 1). As can be seen from the data presented in Table 1, the addition of wetting agents led to a rise in the particle content of Ni-SiC coating deposited in the presence of wetting agent SK.

When, however, the latter coatings were deposited in the presence of wetting agent WFK1, a decrease in the SiC content in the coating was observed already starting from a concentration of 0.014 mM WFK1. As can be seen from these experiments, the choice of the wetting agent for the particles being deposited in the nickel matrix may significantly influence on the composition of the composite layer.

TABLE 1. **The SiC content, the roughness, microstress and the size of grains of the composite coatings Ni-SiC**

TABELA 1. **Zawartość cząstek SiC, chropowatość mikronaprężenia i rozmiary ziaren osnowy niklowej w warstwach kompozytowych Ni-SiC**

Composition of the bath	SiC content % vol.	R_a μm	$\Delta a/a$	D_{111} Å
Without additives	2.71	0.39 ±0.05	0.0002	393
SRN	2.44	0.19 ±0.03	0.0033	225
0.056 mM ASB	2.44	0.25 ±0.03	0.0029	205
0.1 mM ASB	2.71	0.23 ±0.03	0.0028	211
0.016 mM SK	2.71	0.23 ±0.03	0.0033	208

0.033 mM SK	3.24	0.24 ±0.03	0.0031	194
0.014 mM WFK1	2.01	0.29 ±0.03	0.0031	205
0.028 mM WFK1	2.09	0.29 ±0.03	0.0028	190

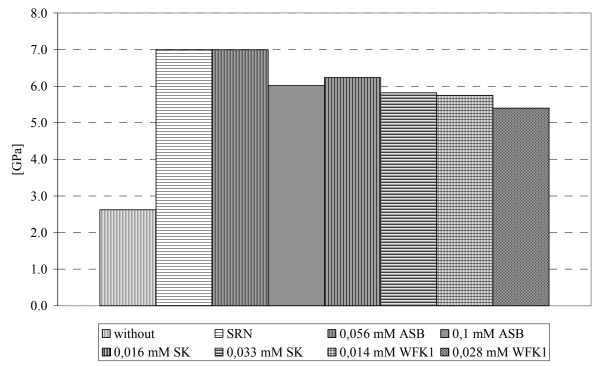

Fig. 2. Microhardness of Ni-SiC composite coatings. Load 0.01 kG

Rys. 2. Mikrotwardość powłok kompozytowych Ni-SiC. Obciążenie 0,01 kG

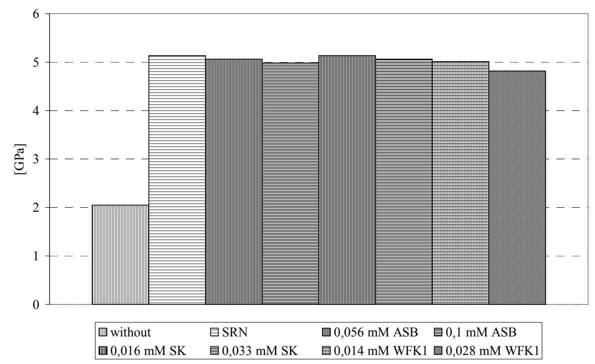

Fig. 3. Microhardness of Ni-SiC composite coatings. Load 0.05 kG

Rys. 3. Mikrotwardość kompozytowych powłok Ni-SiC. Obciążenie 0,05 kG

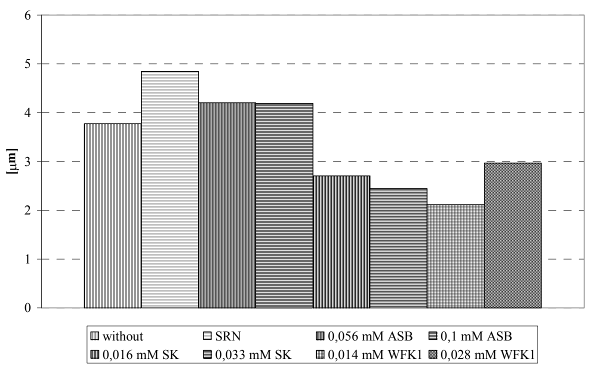

Fig. 4. Depth of abrasion of Ni-SiC composite coatings

Rys. 4. Głębokość wytarcia kompozytowych powłok Ni-SiC

With the aid of the Statistica software package, regression was calculated for the series Ni-SiC coatings.

Using microstress ($\Delta a/a$) as the dependent variable and content of particles in coating (C_v) as independent variable was obtained the following regression equation

$$\Delta a/a \cdot 10^3 = 1.03 \cdot C_v$$

with $r = 0.93$ (correlation coefficient); $p < 0.05$ (level of significance).

The change in the $\Delta a/a$ with SiC contents in the composite coating is shown in Figure 5. Microhardness increase and roughness decreased considerably with decreasing grain size. The change in microhardness with grain size of nickel coatings ($d^{-1/2}$) is shown in Figure 6 in the form of a Hall-Petch plot [5, 6].

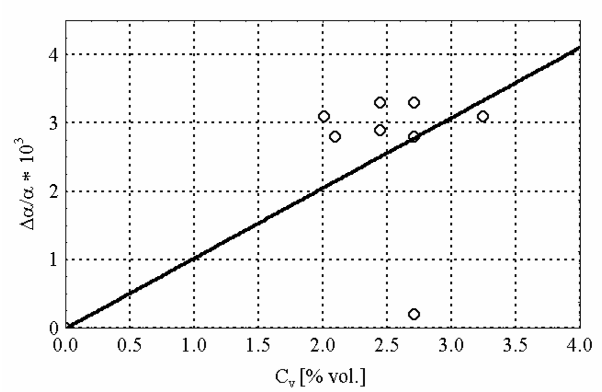

Fig. 5. Dependence of microstress on SiC contents in the coating Ni-SiC

Rys. 5. Zależność mikronaprężeń od zawartości cząstek SiC w warstwie Ni-SiC

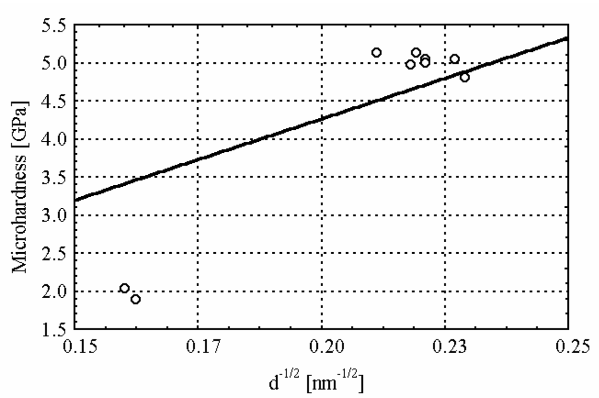

Fig. 6. Dependence of microhardness on nickel grain size D_{111}

Rys. 6. Zależność mikrotwardości od wielkości ziaren niklu D_{111}

Using $d^{-1/2}$ as the independent variable and microhardness (HV0.05) as the dependent variable was obtained the following regression equation

$$HV0.05 = 21.36 \cdot d^{-1/2}$$

with $r = 0.99$; $p < 0.05$.

Wear results are given in Figure 4. The wear test showed that the coating deposited from bath containing additives SRN and 0.014 mM WFK1 had the best wearability. Generally, roughness, microhardness and particle contents in the coatings considerably affect wear resistance.

Depth of abrasion (h) as the dependent variable, and the particles content in the coating (C_v), the microhardness (HV0.01), the size of nickel grain and the roughness (R_a) as the independent variables were used to calculation of the regression equation.

The following regression equation was obtained:

$$h = -0.56 \cdot C_v - 11.2 \cdot R_a + 0.52 \cdot \mathrm{HV0.01} + 0.21 \cdot D_{111}$$

indicating that satisfactory dependence exists between the depth of abrasion and these independent variables. The equation is characterised by the correlation coefficient $r = 0.99$, the standard error of estimation, 0.77.

Depth of abrasion will be decreasing as roughness, microhardness and the size of nickel grain decreases and the SiC content increases. These results are difference from this as were give in the paper [2] probably because that other abrasion tests were used in the both experiments.

The results give in this work suggest that used of the organic compounds may cause such change the structure and the particle content that electrodeposited coatings will has the good wear resistance.

CONCLUSIONS

This study has shown that the wear resistance is proportional to the microhardness, the roughness, the size of nickel grain and the particle contents in Ni-SiC coatings. It has also been demonstrated that the depth of abrasive of the Ni-SiC coatings can be considerably improved by using the wetting agents.

The cationic wetting agent used for deposition of the Ni-SiC composite coatings may significantly influence on the composition and structure of these coatings as that they will has the better abrasive wear resistance.

REFERENCES

[1] Halling J., Thin Solid Films 1983, 108, 103.
[2] Kerr C., Barker D., Walsh F., Archer J., Trans. Inst. Metal Finish. 2000, 78, 5, 171-178.
[3] Gyftou P., Pavlatou E.A., Spyrellis N., Hatzilyberis K.S., Metal Finish. 2000, 78, 6, 223-226.
[4] Halder, Wagner, Acta Crystallogr. 1966, 20, 312.
[5] Hall R.O., Proc. Phys. Soc. Lond. B, 1951, 64, 747.
[6] Petch N.J., Iron Steel Inst. 1953, 174, 25.

Recenzent
Antoni Budniok