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ELECTRODEPOSITION OF COMPOSITE Ni+FeAl GRADIENT COATINGS

An electrolytic co-deposition technique has been successfully used to prepare the composite Ni+FeAl gradient coatings with a continuous compositional gradient. Production of this functional gradient material (FGM) was carried out by simultaneous electrodeposition of nickel with FeAl powder on a copper substrate from a bath in which iron aluminide particles concentration was increased gradually and suspended by mechanical stirring (Fig. 1). Electroplating of the Ni+FeAl FGMs was conducted under constant current conditions at room temperature (Tab. 1).

The microstructure, distribution and percentage volume fraction of FeAl powder in the deposit, were analyzed and determined by metallographic microscope. Surface morphology was carried out using a scanning electron microscope (SEM). Structural investigations were conducted by XRD. The microhardness (μHV) of the deposit was determined with a Vickers diamond testing machine.

The percentage volume fraction of iron aluminide for composite Ni+FeAl gradient coatings plated under proposed conditions indicated that the FeAl particle content in the deposit increased gradually in the direction of the deposit growth from 0 to 39.3 vol.% (Fig. 2). The microstructure of a cross-section (Fig. 3) and SEM observations of the deposit surface morphology (Fig. 4) revealed that FeAl particles were uniformly distributed in the Ni matrix and their content increased gradually throughout the thickness. Microstructural transition with composition of the FeAl FGM including a nickel matrix, a dispersive structure, a network structure, and an alternative dispersive structure, have been discussed.

The phase composition investigations of the Ni+FeAl FGM exhibited a diphase composite structure with a polycrystalline nickel matrix into which the solid crystalline FeAl particles with the B2 structure were embedded (Fig. 5).

The Vickers microhardness as a function of the thickness of the gradient deposit has been also determined (Fig. 6). It increased with a gradual increase in the thickness from 196.9 for pure nickel up to 453.1 μHV for the top layer of the deposit. It was found that embedding of FeAl particles with high intensity and hardness in the Ni matrix, started to harden and strengthen the deposit.

Key words: electrodeposition, FGM, composite coatings, Ni+FeAl

ELEKTROOSADZANIE GRADIENTOWYCH WARSTW KOMPOZYTOWYCH Ni+FeAl

Warstwy kompozytowe Ni+FeAl o ciągłym gradiencie składu chemicznego otrzymywano techniką elektrolitycznego współosadzania. Otrzymywanie takiego funkcjonalnego materiału gradientowego (FMG) realizowano poprzez jednoczesne współosadzanie niklu i proszku FeAl na podłożu miedzi z kąpieli zawiesinowej, w której zawartość cząstek aluminidku żelaza była stopniowo zwiększana. W celu utrzymania proszku FeAl w postaci zawiesiny stosowano mieszanie mechaniczne (rys. 1). Elektroosadzanie FMG Ni+FeAl prowadzono w warunkach galwanostatycznych w temperaturze pokojowej (tab. 1).

Mikrostrukturę, rozkład i procentowy udział objętościowy cząstek FeAl w warstwach analizowano za pomocą mikroskopu metalograficznego. Badania morfologii powierzchni realizowano przy użyciu skaningowego mikroskopu elektronowego (SEM). Badania strukturalne prowadzono metodą rentgenowskiej analizy fazowej. Pomiary mikrotwardości (μHV) warstw wykonano za pomocą mikrotwardościomierza Vickersa.

Procentowy udział objętościowy aluminidku żelaza w kompozytowych warstwach gradientowych Ni+FeAl, otrzymanych w proponowanych warunkach, wskazuje, że zawartość cząstek FeAl w warstwie wzrasta stopniowo w kierunku wzrostu warstwy od 0 do 39,3% obj. (rys. 2). Obserwacje mikrostruktury zgładu poprzecznego (rys. 3) i morfologii powierzchni warstw (rys. 4) wykazują, że cząstki FeAl są jednorodnie rozmieszczone w osnowie Ni i ich zawartość wzrasta stopniowo ze wzrostem grubości warstwy. W pracy dyskutowane są przemiany mikrostrukturalne FMG Ni+FeAl wywołane zmianą składu chemicznego z uwzględnieniem osnowy niklowej, struktury dyspersyjnej, struktury komórkowej i alternatywnej struktury dyspersyjnej.

Badania składu fazowego FMG Ni+FeAl wykazują dwufazową strukturę kompozytu o osnowie polikrystalicznego niklu, w której zabudowane są krystaliczne cząstki stałe FeAl o strukturze B2 (rys. 5).

Określono także mikrotwardość Vickersa jako funkcję grubości warstw gradientowych (rys. 6). Mikrotwardość rośnie ze stopniowym wzrostem grubości warstwy od 196,9 dla czystego niklu do 453,1 μHV dla górnej warstwy FMG. Stwierdzono, że zabudowanie w osnowie Ni cząstek FeAl o wysokiej gęstości i twardości powoduje utwardzenie i umocnienie warstwy.

Słowa kluczowe: elektroosadzanie, FMG, warstwy kompozytowe, Ni+FeAl

INTRODUCTION

Graded structures may be found in ordinary engineering materials from former days; however, if one has begun to tailor the intentional gradient of composition and/or microstructure in a material in order to achieve the desired functions and properties, the materials are called as Functional Gradient Materials (FGMs) [1-7]. The FGMs are now accepted worldwide, and it has opened new fields for material design and effective ap-

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plications for industrial materials; they have found great use as medical, electronic, nuclear energy materials, etc. [2, 5-7].

Up to now many efforts have been made to obtain FGMs using chemical or physical vapor deposition (CVD or PVD), self-propagating high temperature synthesis (SHS), plasma spraying (PS), powder metallurgy (PM), and composite electroplating (CE) [3, 6, 7]. The latter technique is one of the most advanced production routes of FGMs. The major advantages of the CE method over other techniques are the simplicity to control, possibility to process complicated parts, and finally, the low initial capital investment [7-12]. Moreover, composite coatings produced by dispersion electroplating combine the properties of dispersed solid particles and a metal or alloy, so they mostly have a higher wear, high-temperature oxidation, electrochemical activity and corrosion resistance as compared to the pure metal [4, 6-10]. Recently, there is also a trend to using solid powder of primary particle diameter below 100 nm as the composite component [11, 12]. Such a kind of nanocomposite coating may found application in micro-technology and in thin film coatings.

The present study was undertaken to obtain composite Ni+FeAl gradient coating with a progressive change in structure and properties. Our main goal was to report the CE technique of this FGM material production, as well as physical and chemical characterization of as-deposited coatings.

EXPERIMENTAL DETAILS

To obtain composite Ni+FeAl FGM the following Ni plating bath was prepared (g dm^{-3}): 600 nickel sulfate, 10 nickel chloride, 30 boric acid, to which the increasing content of Fe50Al50 (at.%) powder (diameter of particle below 20 μm) was added gradually and maintained in suspension by mechanical stirrer in accordance with the deposition conditions listed in Table 1. The pH of the bath was 3.5.

TABLE 1. **Conditions of electrodeposition process for the Ni+FeAl FGM**

TABELA 1. **Warunki procesu elektroosadzania FMG Ni+FeAl**

No.	FeAl concentration in the bath g dm^{-3}	Plating time min	Stirring speed rpm
1	0	25	0
2	5	60	500
3	20	60	800
4	50	90	800
5	80	90	1000
6	120	120	1200

The constant current electrodeposition was carried out on a copper substrate (1 cm^2) at j = 100 mA cm^{-2}, at room temperature. The substrate pretreatment has been described elsewhere [8-11]. The Ni+FeAl FGM thickness was estimated from the cross-section by microscope to be 220 μm. The experimental set-up has been displayed in Figure 1.

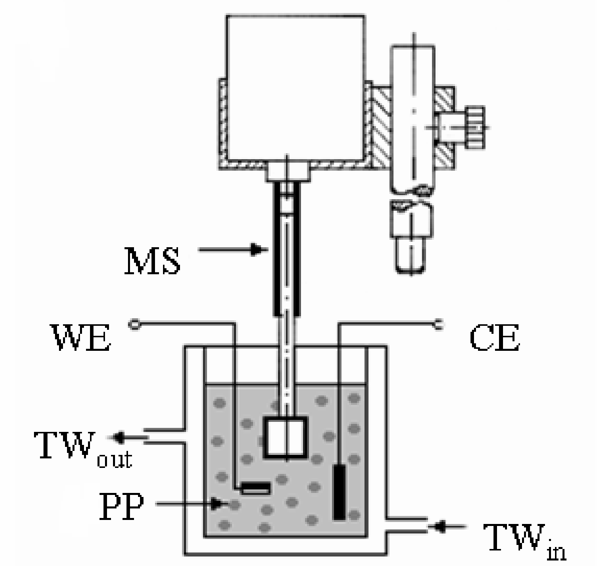

Fig. 1. Schematic diagram of the experimental set-up: electrodeposition cell with a platinum counter electrode (CE), the working electrode (WE) of the coating under study, agitation of the electrolyte containing powder particles (PP) using mechanical stirring (MS), and a constant temperature by thermostated water (TW)

Rys. 1. Schemat układu doświadczalnego: naczyńko do elektroosadzania z przeciwelektrodą platynową (CE), elektrodą pracującą (WE) w postaci badanej warstwy, mieszaniem elektrolitu zawierającego cząstki proszku (PP) za pomocą mieszadła mechanicznego (MS) oraz stałą temperaturą utrzymywaną za pomocą termostatu (TW)

The microstructure, distribution and percentage volume fraction of FeAl powder in the deposit, were analyzed and determined by metallographic microscope (Neophot 2) and image analyzer (Image-Pro Plus ver. 3). Surface morphology was carried out using a scanning electron microscope (Hitachi S-4200, Japan). Structural investigations were conducted by XRD using a Philips diffractometer and $Cu_{K\alpha}$ radiation. The microhardness (μHV) of the deposit was determined with a Vickers microhardness tester. The hardness tests were performed under an indentation load of 0.785 N for 15 s.

RESULTS AND DISCUSSION

The results of percentage volume fraction of iron aluminide for composite Ni+FeAl coatings plated under proposed conditions (Tab. 1) show that the FeAl particle content in the Ni+FeAl FGM changes gradually from 0 (deposit No.1) to 39.3 vol.% (deposit No. 6) (Fig. 2). Therefore, Ni+FeAl gradient coatings could be produced by continuous increase in the concentration of FeAl particles in the bath and at a constant value of current density.

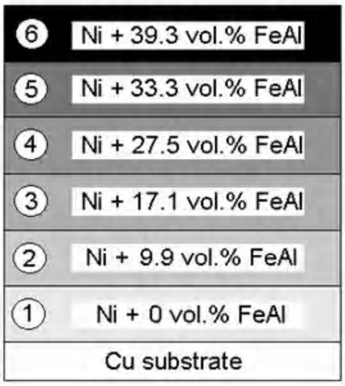

Fig. 2. Percentage volume fraction of FeAl (vol. %) for composite coatings deposited under various plating conditions displayed in Table 1

Rys. 2. Procentowy udział objętościowy FeAl (% obj.) w warstwach kompozytowych otrzymywanych w warunkach osadzania przedstawionych w tabeli 1

As shown in Figure 3, the cross-sectional studies of the Ni+FeAl FGM indicate microstructural transition with composition (see also Fig. 2). The microstructure is characterized by the gradual replacement of the matrix from nickel (Fig. 3a) to intermetallic phase, with an increase in the content of FeAl (Figs 3b-d).

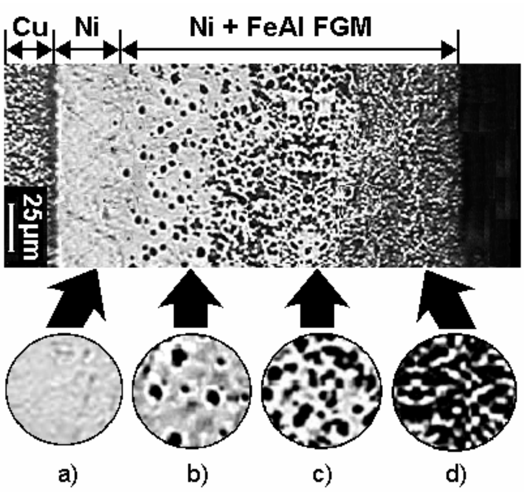

Fig. 3. Cross-sectional image of Ni+FeAl FGM with a visable microstructural transitition: a) a nickel matrix, b) a dispersive structure, c) a network structure, d) an alternative dispersive structure; see Fig. 2

Rys. 3. Obraz zgładu poprzecznego FMG Ni+FeAl z widoczną przemianą mikrostrukturalną: a) osnowa niklowa, b) struktura dyspersyjna, c) struktura komórkowa, d) alternatywna struktura dyspersyjna; patrz rys. 2

In the nickel-rich side, FeAl particles are homogeneously dispersed in the Ni matrix (Fig. 3b). With the increase of FeAl phase, conglomerates of intermetallic phase particles are formed and the consequence of its further growth results in the formation of network structure of FeAl phase (Fig. 3c).

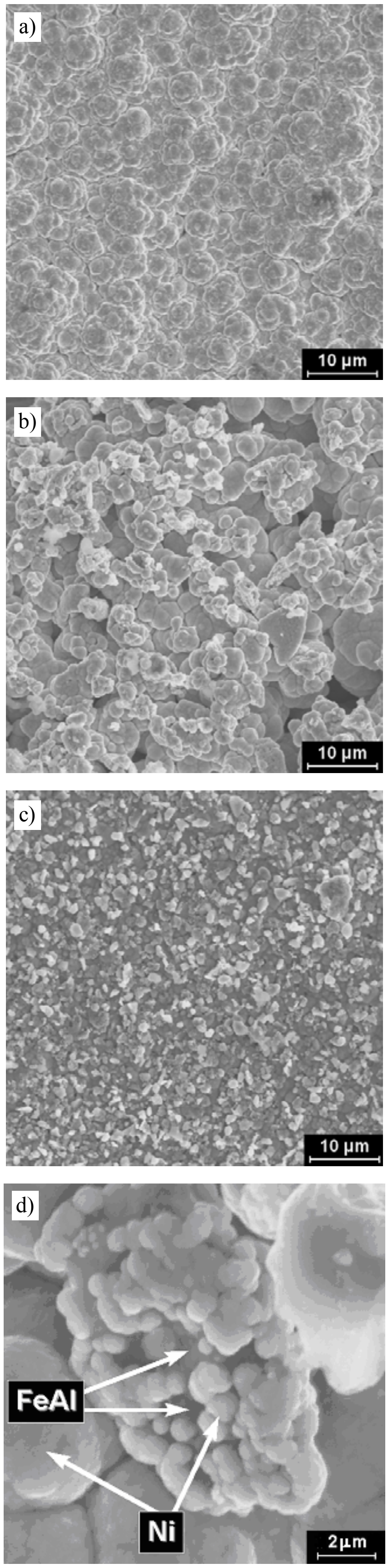

Fig. 4. Surface morphology of the as-plated coatings: a) Ni (No. 1), b) Ni + 17.1 vol.% FeAl (No. 3), c) and d) Ni + 39.3 vol.% FeAl (No. 6); details in Tab. 1 and Fig. 2

Rys. 4. Morfologia powierzchni osadzanych warstw: a) Ni (Nr 1), b) Ni + + 17,1% obj. FeAl (Nr 3) c), d) Ni + 39,3% obj. FeAl (Nr 6), szczegóły w tab. 1 i na rys. 2

Then, the network of nickel phase is gradually diminished and turns into isolated phases dispersed in the iron aluminide matrix in the FeAl-rich side; an alternative dispersive structure is formed (Fig. 3d). Since the network structure may be determined by the formation of percolation conglomerates of FeAl or Ni phase, there is a composition range in which both phases are in a network structure [5].

The Ni+FeAl FGM show very good adhesion to the copper. There are no internal stresses and other defects in the coating microstructure resulting in cracks, voids or defoliation from the substrate. Figure 4 contains some of the SEM photomicrographs for as-plated: Ni (No. 1), Ni + 17.1 vol.% FeAl (No. 3) and Ni + 39.3 vol.% FeAl (No. 6) deposits (see Tab. 1, Fig. 2 for details). All Ni+FeAl deposits, independent of the FeAl content in the bath, are characterized by homogeneous distribution of FeAl particles in the nickel matrix, which will have a considerable effect on the hardness of the Ni+FeAl FGM (Figs. 4a-c). A small tendency to an agglomeration of FeAl particles embedded into the nickel at lower concentration of FeAl particles in the bath, could be observed (Fig. 4b). In Figure 4d, the characteristic grain of embedded particle of FeAl powder in the Ni matrix, is shown. One can observe the small nickel grains onto the surface of the microsize FeAl particle, what may suggest the adsorption model of the composite coating formation [8-11]. The SEM results confirm the composite structure of the deposit.

Figure 5 presents XRD pattern of the Ni+FeAl FGM examined directly after electrodeposition. The corresponding elements are indicated at the top of each diffraction line.

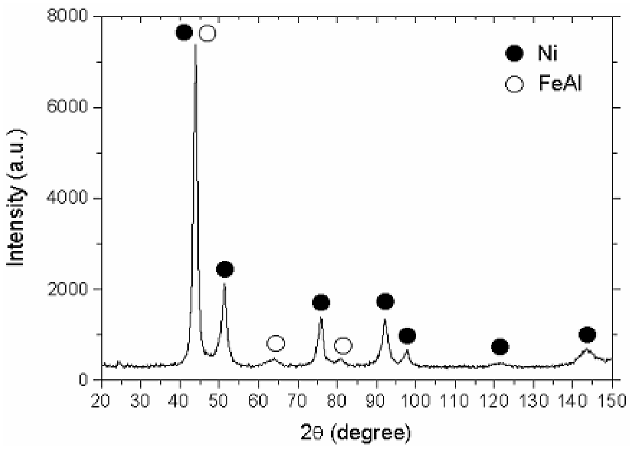

Fig. 5. XRD pattern of the Ni+FeAl FGM

Rys. 5. Dyfraktogram warstwy FMG Ni+FeAl

The phase composition investigations of the composite Ni+FeAl FGM reveal the presence of two phases. The XRD pattern confirms the presence of a composite matrix in a form of the polycrystalline nickel and the FeAl intermetallic phase with the B2 structure. These results confirm that crystalline solid particles of iron aluminde could be embedded into the Ni matrix in the electroplating process.

The Vickers microhardness as a function of the thickness of the Ni+FeAl FGM is shown in Figure 6. It can be seen that the microhardness increases with a gradual increase in the thickness from 196.9 for pure nickel up to 453.1 μHV for the top layer of the deposit. A comparative value of microhardness for the copper substrate equals 133.4 μHV. These μHV changes result from the presence of FeAl particles in the Ni matrix, which obstructs the shift of dislocations in nickel. It is evident that inclusion of FeAl particles with high intensity and hardness in the Ni matrix, tends to harden and strengthen the deposit, and results in the increase of the deposit microhardness.

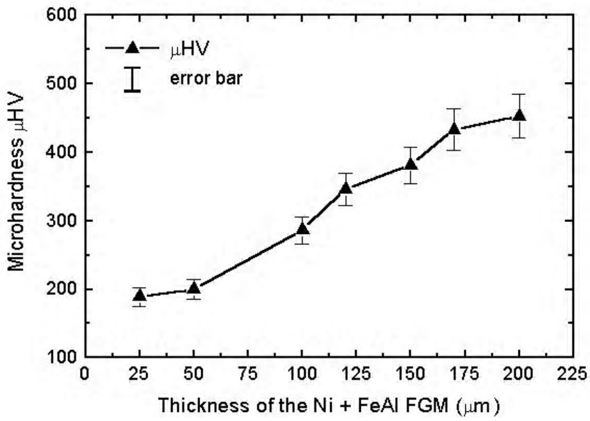

Fig. 6. Vickers microhardness (μHV) as a function of thickness of the Ni+FeAl FGM obtained under conditions listed in Table 1 (see Figs 2 and 3)

Rys. 6. Mikrotwardość Vickersa (μHV) jako funkcja grubości FMG Ni+FeAl otrzymanego w warunkach przedstawionych w tabeli 1 (patrz rys. rys. 2 i 3)

A consequence of the continuity in the electrodeposition process giving the coordination of every point with the ambience in the gradient coatings is that the produced Ni+FeAl FGMs are characterized by a gradual variation of microhardness throughout the thickness. It might be expected that in contrast to the bounded interface of a Cu substrate Ni+FeAl composite coating, the gradient coating with gradually changes in composition from pure nickel to intermetallic phase, will increase the cohesion and the bond strength of the coating substrate system in electroplating and application.

CONCLUSIONS

- The composite Ni+FeAl FGMs with a continuous compositional gradient of the FeAl from 0 to 39.3 vol.% can be produced by gradual increasing the concentration of iron aluminide powder in the bath under proposed electrodeposition conditions.

- The cross-sectional studies of the Ni+FeAl FGM indicate microstructural transition with composition. The microstructure is characterized by the gradual replacement of the matrix from nickel to intermetallic phase, with an increase in the content of FeAl in turn: a nickel matrix ⇔ a dispersive structure ⇔ a network structure ⇔ an alternative dispersive structure.
- It was found that the obtained Ni+FeAl FGMs exhibit a composite structure of polycrystalline nickel matrix into which the crystalline FeAl particles with the B2 structure are embedded.
- The Ni+FeAl FGMs thus produced are characterized by a continuous variation of microhardness throughout the thickness, and are potentially applicable to the improvement the bond strength of composite coatings with the substrate.

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Recenzent
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