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Surface Treatment of Powder Material by Metal Coatings - Study of Corrosion Properties

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Abstract

Coatings by one-component Ni and two-component Ni-Co were electrodeposited onto Fe powder particles from a sulphate-based electrolyte. The coatings were deposited under galvanostatic conditions at 25^{o}C. A fluidized bed arrangement was applied. The influence of cathodic current intensity and electrolysis time on the composition of metal coating was studied. The amount of deposited metals increased with the current intensity and with the electrolysis time. Scanning electron microscope and optical microscope were used to characterize the metal coatings.

Voltammetric measurements have been conducted in order to characterize the electrochemical behaviour of Ni and Ni–Co coatings in two corrosion environments (0.5 M H_2SO_4 and 3% NaCl water solution) at laboratory temperature. It was shown that the increase in Ni content enhances the corrosion resistance of the system powder Fe/Ni coating.

Key words : Ni coating, Ni-Co coating, Voltammetry, Polarization resistance

Introduction

Electrochemical coating represents a possible way how to improve various properties of basic material. Electrochemically deposited alloy films have a wide range of applications, from corrosion protection of steel sheets to microelectronic devices.

In metallurgical production, especially in powder metallurgy, such one-, two- or multilayer coatings are required on powder surfaces, most frequently on Fe powders. The electrodeposition of two-component coatings on dispersed electrodes is a much more complicated process than that of a one-component coating.

Electrochemical processes on powders may be realized in a cell with a fluidized bed electrode(1). Alloy electrodeposition on powder materials depends strongly on variable experimental factors. Basic parameters affecting electrodeposition of binary Ni-Cu coating, i.e. the composition of the electrolyte and the current density, were analysed in detail in the previous work(2) from the point of view of their capability of bringing deposition potentials of Ni and Cu closer together. It was observed that a large excess of Ni is required in order to suppress the preferential deposition of the more electropositive Cu. The addition of sodium citrate as complexing agent reduces significantly the currentless deposition of Cu on Fe powder. An increase in current density leads to the preference of the less noble metal, i.e. Ni, in the deposit on the powder. The influence of hydrodynamic parameters of the fluidized bed (particle size, rotation speed and density of the suspension of powder particles in the electrolyte) is manifested through the changes in the surface area available for the electrochemical deposition of coating, as well as through the quality of the contacts between the solid cathode and the powder particles. The composition of two-component Ni-Cu coating deposited on Fe powder was studied by constant current electrolysis. Both the Cu and Ni contents in the deposit decrease with increase in the particle size, and with decrease in the suspension density. The quality of the contacts worsens with increase in particle size and rotation speed, which results in a lower portion of Ni in the deposit due to the preferential Ni deposition on the compact cathode.(3) Considerable differences have been found between the electroless and electrolytically deposited layers of Ni, Cu and binary Ni-Cu on the hollow Fe powder particles.The coatings prepared by electroplating are much stronger, harder and uniformly distributed on the surface.(4) The corrosion behaviour of the three different nickel/copper coatings deposited on hollow iron particles in atmospheres of 3 M NaCl was investigated by using SEM and ToF-SIMS methods. The best corrosion resistance was observed for two layers of Cu/Ni coating obtained by successive electrodeposition of a Cu layer and then a Ni layer.(5)

Nickel, cobalt, and their alloys are important engineering materials in many applications because of their unique properties, such as magnetic and electrocatalytic properties, heat conductivity as well as wear and corrosion resistances. The presence of cobalt in nickel alloy is known to improve the hardness of the solid.(6)

The aqueous corrosion is of electrochemical nature and consists of anodic and cathodic reactions on the surface of the corroding metal. These reactions can be studied by using electrochemical methods in which the electrical potential applied to the electrode is controlled and the resulting current is measured. The semilogarithmic form of the polarization curves is often used to determine the corrosion current and potential. This type of analysis is referred to as Tafel Slope Analysis. According to Faraday´s law, there is a linear relationship between the metal dissolution rate (corrosion rate) and the corrosion current.(7)

The commonly used method to monitor corrosion in industry is the polarization resistance method. The polarization resistance (R_p) can be related to the corrosion current by equation

$$i_{corr} = B / R_p \qquad (1)$$

where B represents a proportionality constant.

The polarization resistance can be used as a quantitative parameter to compare the corrosion resistance of metals under various conditions.

Voltammetry of immobilized microparticles (VIM) is suitable for the determination of the corrosion properties of powdered metals (8,9). Micro amounts of metallic powder are fixed on a paraffin-impregnated graphite electrode (PIGE) immersed in electrolyte solutions and polarization curves are recorded.

Materials and Experimental Pocedure

The iron powder with trace elements 0.0035 % C, 0.03 % O_2, 0.033 % Mn, 0.016 % P, 0.015 % Si (type ASC-200) was prepared by pressure water atomization (Mannesmann) process. The powder was screened into five granulometric classes: 0-45, 45-63, 63-100, 100-125, 125-160 µm. The Ni and binary Ni-Co coatings were deposited on the iron powder particles (5 g) of fraction 63 - 100 µm.

The electrolyte, containing 1.2 M $NiSO_4$, 0.3 M NaCl and 0.3 M H_3BO_3 (pH 2) was used as a nickel-plating bath. The two-component coatings were deposited from a bath containing 0.69 M $NiSO_4.7H_2O$, 0.125 M $CoSO_4.7H_2O$ and 0.26 M NaCl (pH 2). Various electrolysis conditions were used: a) current intensity of metal deposition varied from 0.2 A to 1.0 A, electrolysis time was 30 min; b) the current intensity was 0.8 A and the electrolysis time was 20, 40, 60, 80 and 100 minutes, respectively.

The fluidized system was realized by circular stirring of the electrolyte suspension. The rate of stirring was 800 r.p.m. A stainless steel cathode and a Ni sheet anode were used; a diaphragm separated the cathodic (plastic, volume 150 ml) and anodic (glass, volume 400 ml) compartments of the cell (Figure 1).

After each electrolysis the cell was disengaged and the coated powder was removed, filtered, rinsed in distilled water and dried by acetone. After dissolution the content of metals was determined by atomic absorption spectrometry (AAS).

The corrosion resistance of deposited coatings has been examined in the environment of the 0.5 M H_2SO_4 and 3 % NaCl water solutions. Experiments were carried out in a three-electrode electrochemical cell with a platinum plate as counter-electrode, Ag/AgCl/3M KCl as reference electrode and Fe particles fixed on PIGE as the working electrode. Voltammetric measurements were recorded on the potentiostat PGP 201 Voltalab at a scan rate of 5mV/s. All experiments were carried out at laboratory temperature. The microstructure of Fe powder was analysed by SEM and optical microscopy.

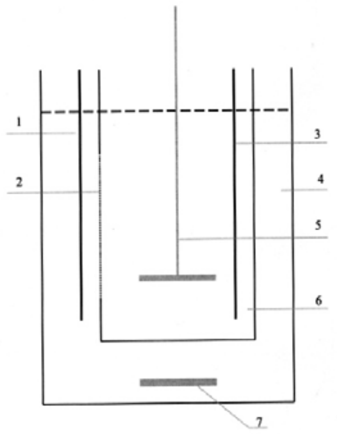


Figure 1. Scheme of experimental cell for electrolysis 1- nickel anode, 2- textile diaphragm, 3- stainless steel cathode, 4 – anodic compartment, 5- glass stirrer, 6- cathodic compartment, 7- magnetic stirrer

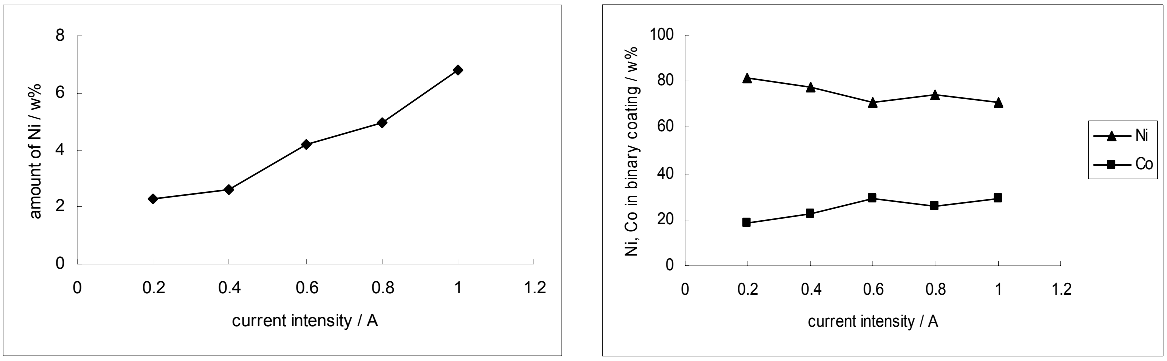
Results and Discussion

The composition of Ni coating and binary Ni-Co coating deposited on Fe powder is plotted versus the current intensity in Figures 2a and 2b. As expected, the amount of deposited nickel increases with the increase of current intensity. In the case of binary Ni-Co coating the increasing current intensity favours the Co deposition and suppresses the Ni deposition, but the Ni content is always higher than Co content. An increase of current intensity causes the cathode potential to become more negative and this condition should increase the proportion of less noble metal in the deposit.(10)

The electrolysis time is an important parameter which strongly influences the electrodeposition of metals. Of course, the total amount of the deposited metal and therefore the final thickness of the coating layer will increase with deposition time. Figures 3a and 3b presents the composition of Ni coating and binary Ni-Co coating versus electrolysis time.

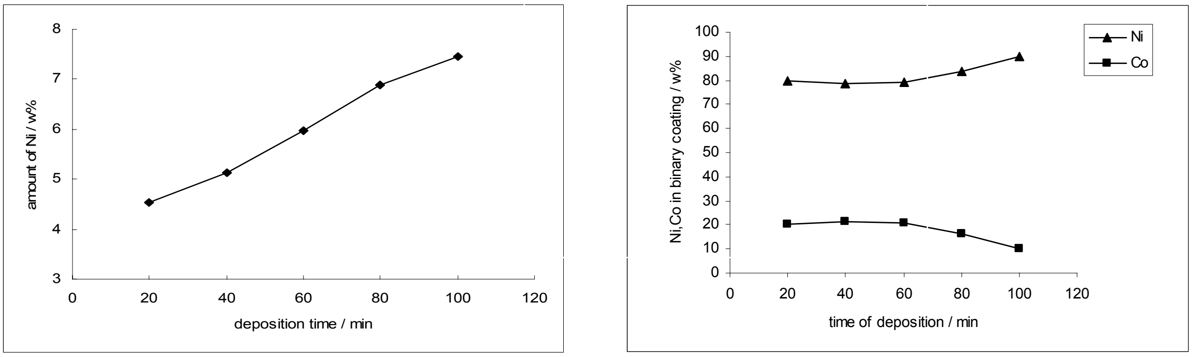
The surface of Fe powder without coating and Fe powder covered with the binary Ni-Co coating is shown in Figures 4 and 5. The micrograph suggests a uniform distribution and compact morphology of the film. The Fe particles are very rough and porous and so they are of very large surface. Consequently the thickness of metal coating is small. The roughness of the surface decreases, a smoothing of the surface increases with higher current intensity and electrolysis time.

The polarization resistance is the parameter for determination of the corrosion rate. The relationship between R_p of coated Fe powder and the current intensity applied during electrodeposition process is presented in Figures 6 and 7. In the case of both, one- and two-component coatings R_p increases with the current intensity of metal deposition. During the corrosion test a protective layer of corrosion products is formed on the surface of the sample and consequently the corrosion resistance increases. According to equation (1) the protective layer reduces the rate of the corrosion process. The R_p increase is more marked in the case of the binary Ni-Co coating.



a) b)

Figure 2. The effect of current intensity on the content of
a) Ni in the coating layer deposited on Fe powder (as fraction of weight of coated particles)
b) Ni, Co in the binary coating deposited on Fe powder (Ni+Co = 100%)
Experimental conditions: deposition time 30 min, rotation speed 800 r.p.m.



a) b)

Figure 3. The effect of deposition time on the content of
a) Ni in the coating layer deposited on Fe powder (as fraction of weight of coated particles)
b) Ni, Co in the binary coating deposited on Fe powder (Ni+Co = 100%)
Experimental conditions: electrolysis current 0.8 A, rotation speed 800 r.p.m.

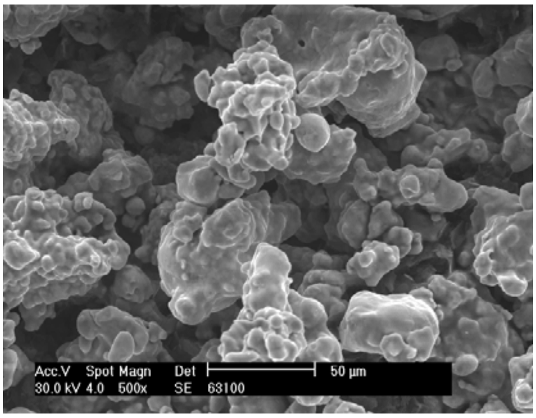


Figure 4. Detail of Fe powder particles without metal coating, SEM

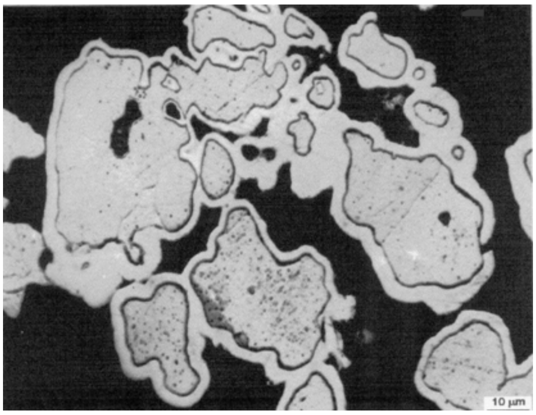


Figure 5. Optical micrograph of Ni-Co coating on Fe powder; 500x deposition time 30 min, current intensity 1A, rotation speed 800 r.p.m.

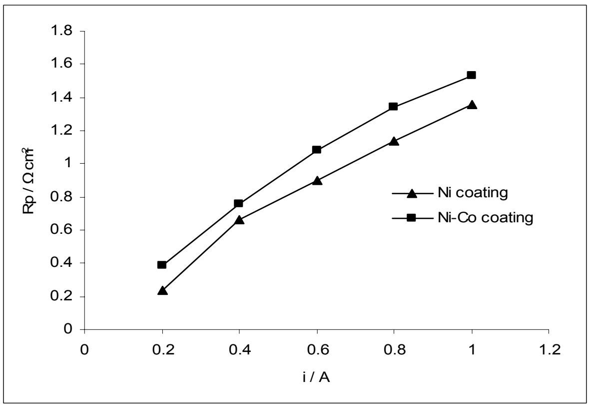


Figure 6. The polarization resistance depending on the current intensity of Ni and Ni-Co deposition on Fe powder in 0.5 M H_2SO_4 Electroplating : deposition time 30 min, rotation speed 800 r.p.m. Electrochemical measurement: scan rate 5 mV/s

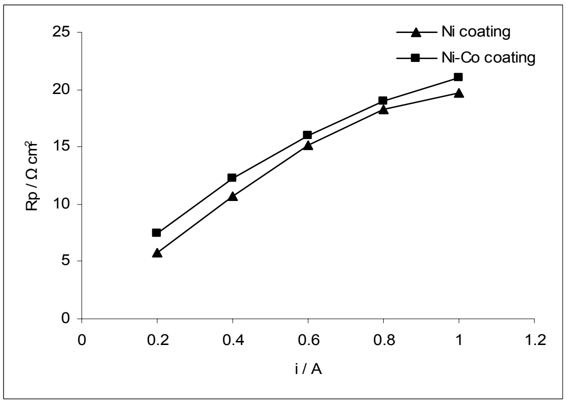


Figure 7. The polarization resistance depending on the current intensity of Ni and Ni-Co deposition on Fe powder in 3 % NaCl Electroplating : deposition time 30 min, rotation speed 800 r.p.m. Electrochemical measurement: scan rate 5 mV/s

The polarization resistance dependence on the electrolysis time of the metal coating deposition indicates the same trend (Figures 8 and 9).

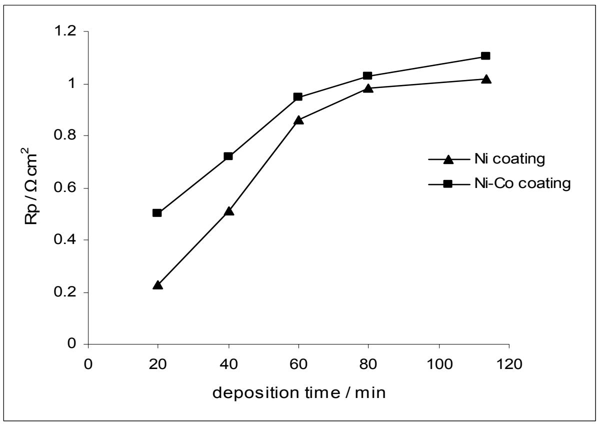


Figure 8. Dependence of R_p on the electrolysis time of Ni and Ni-Co deposition on Fe powder in 0.5 M H_2SO_4 Electroplating : current intensity 0.8 A, rotation speed 800 r.p.m. Electrochemical measurements: scan rate 5 mV/s

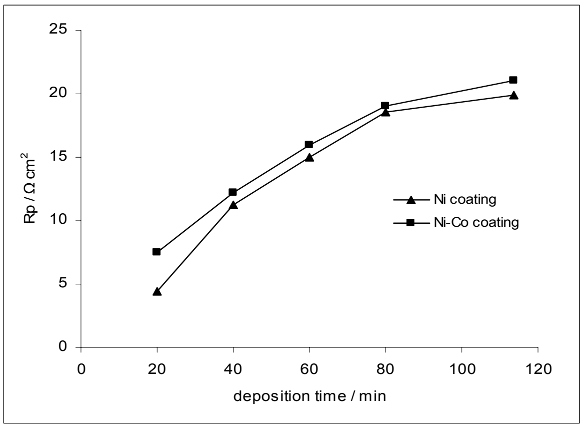


Figure 9. Dependence of R_p on the electrolysis time of Ni and Ni-Co deposition on Fe powder in 3 % NaCl Electroplating: current intensity 0.8 A, rotation speed 800 r.p.m. Electrochemical measurements : scan rate 5 mV/s

The values of the polarization resistance are higher in the water solution of 3 % NaCl. The difference between R_p of one-, and two-component coating is insignificant for the environment of Cl^{-I} ions.

Conclusions

Metal coatings improved the surface morphology and corrosion resistance of basic powder materials. An increasing trend was observed in the amount of deposited Ni with increase of current intensity and prolonged time of electrodeposition. In the case of binary Ni-Co alloy coating the current intensity and electrolysis time increase supports Co deposition and suppresses Ni deposition, but the amount of nickel is in general higher than the amount of cobalt. The coating layer was thin and followed the surface roughness.

The corrosion resistance of system powder Fe/Ni coating and powder Fe/Ni-Co alloy coating is in general higher than for uncoated Fe powder. From the comparison of the corrosion characteristics it follows that the higher value of corrosion resistance was observed in the case of two-component Ni-Co coating in both corrosion environments.

Voltammetry of immobilized microparticles is a suitable method for evaluation of the corrosion process, especially of powdered metals. The corrosion ressistance was determined by simple polarization measurements and a Tafel Slope Analysis.

Acknowledgements

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