The Effect of the Amount of Ferrous Sulfate in the Electrolyte on the Hardness of the Ni-Fe Alloy Coating Generated by Electrical Deposition Method

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Abstract

Nickel-iron alloy coatings with different amounts of ferrous sulfate in plating bath on steel substrate under turbulence 250 rpm and current density 2.5 A d⁻¹m⁻² were created. the amount of ferrous sulfate added to the plating bath varied from 20 g l⁻¹ to 80 g l⁻¹. EDX test was taken from each sample. the amount of iron that precipitated from each plating solution in the alloy coating was obtained. it was observed that for the values of 20 g l⁻¹, 40 g l⁻¹, 60 g l⁻¹, 80 g l⁻¹ iron sulfates 21.29(wt.%), 43.35(wt.%), 51.19(wt.%), 58.8(wt.%) iron were deposited in the alloy coating, respectively. According to the SEM photos obtained from the samples, it was observed that only the sample contains 20 g l⁻¹ ferrous sulfates in the plating bath without cracks and increasing the amount of ferrous sulfate in the plating bath leads to an increase in internal stresses in the coating, which is the main reason creating cracks. Finally, the hardness of the coatings was obtained, which was the highest hardness for the 20 g l⁻¹ sample.

Keywords: Nickel-Iron Alloy, Ferrous Sulfate, Microhardness, Electroplating, Coating.

1. Introduction

Nickel coatings are widely used in engineering applications due to their unique properties such as acceptable abrasion resistance, excellent corrosion behavior and good thermal stability. Alloy coatings show better properties such as hardness, wear resistance and corrosion behavior compared to single element coatings. As a result, the addition of elements such as Cr, P, CO and Fe improves mechanical, electrochemical, corrosion and magnetic properties. The life span of many materials in corrosive environments is reduced [1].

In recent years, much attention has been paid to the magnetic and mechanical properties of iron-nickel alloy coatings. Permalloy (80 wt.% Ni, 20 wt.% Fe) has many applications in electromagnetic instruments due to its soft magnetic properties. The deposited Permalloy alloy nanocrystals with a grain size of approximately 11 nm showed a microhardness of up to 900 VHN. Permalloy, iron has significant properties for practical applications [2-4].

electrodeposition has a convenient method for alloy deposits. this method does not require complex equipment. The advantages of this method can be mentioned as low temperature, low cost, good thickness control and high deposition ability[1,5,6].

simultaneous electrodeposition of iron and nickel shows the phenomenon of anomalous codeposition. Under certain conditions, the reduction of nickel is limited, although it is nobler than iron.

The composition and properties of iron and nickel alloy coatings strongly depend on the solution and deposition conditions. Several explanations are provided for this theory. Dahms and Croll [7, 8] were the ones who claimed that this phenomenon occurs when the PH is high in the surface, which results in the formation of ferrous hydroxide around the electrode and limits the deposition of nickel. Understanding the anomalous phenomenon is very important in the process of creating a nickel-iron alloy coating by electroplating method.

metal matrix composite coatings obtained by electrodeposition have received much attention in recent years. During the electroplating process, the reinforcing particles suspended in the electroplating bath are placed along with the metal ions in the growing metal matrix. Composite coatings have excellent properties such as high hardness, high corrosion resistance, and good wear resistance compared to alloy coatings [9-13].

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	FeSO ₄ (g l ⁻¹)	NiSO ₄ (g l ⁻¹)	Nicl ₂ (g l ⁻¹)	H ₃ BO ₃ (g l ⁻¹)	Na ₃ C ₆ H ₅ O ₇ (g l ⁻¹)	Saccharin (g l ⁻¹)	SDS (g l ⁻¹)
Sample 1	20	250	45	35	25	3	0/1
Sample 2	40	250	45	35	25	3	0/1
Sample 3	60	250	45	35	25	3	0/1
Sample 4	80	250	45	35	25	3	0/1

Table. 1. Chemical composition of plating bath.

2. Materials and Methods

Initially, cylindrical specimens with a thickness and diameter of 1 cm of steel (ST37) were prepared. Both surfaces of the cylinder were polished by sanding to a smooth surface. After sanding, we connected the samples to a metal wire in a solution of water and acetone in a ratio of one with each other for 15 minutes inside the ultrasonic device in order to remove contaminants placed superficially.

After degreasing with acetone solution, the samples were rinsed with distilled water Deoxidize for 30 seconds in a 10% solution of hydrochloric acid and distilled water in a ratio of one. After Deoxidize operation, the samples were rinsed with distilled water and placed in the plating solution. The plating solution used is shown in Table. 1. Iron sulfate is the supplier of iron ions and nickel sulfate is also the supplier of nickel ions in the plating solution. Nickel chloride with ion production cl⁻¹ amplifies the flow inside the solution and the acid Boric controls the pH of the solution, which should be between 2.5 and 3.5. Sodium citrate reduces the phenomenon of anomalous. Saccharin has a De-tensioning role in the deposition and SDS acts as a surface activator and prevents the formation of bubbles on the sample surface during the process. For plating of model (3003D-MO_SUPPLY rectifier POWER DC_MEGATE) used. After being in solution, the sample is connected to the negative pole of the DC rectifier and its positive pole to the nickel anode. By applying current, hydrolysis reactions and hydrogen reduction occur in the solution and the Ni²⁺ and Fe²⁺ atoms move towards the cathode. All samples are subjected to a flow of 2.5 A d⁻¹m⁻² for a duration of 1:45.in The first 5 minutes the current was halved and after that the current was set to 2.5 A d⁻¹m⁻².

3. Result and Discussion

Fig. 1. shows the images obtained with an electron microscope at 250X magnification. Fig. 1.a for 20 g I^{-1} ferrous sulfate in solution, Fig. 1.b for 40 g I^{-1} ferrous sulfate, Fig. 1.c It corresponds to 60 g I^{-1} of ferrous sulfate and Fig. 1.d corresponds to 80 g I^{-1} of ferrous sulfate. As can be seen from Fig. 1., the crack was barely visible in Fig. 1.b and Fig. 1.c and Fig. 1.d, and only Fig. 1.a, which corresponds to 20 g I^{-1} of ferrous sulfate, was free of any cracks. By increasing the amount of iron in the alloy coating formed due to the increase of ferrous sulfate to the plating bath according to the shape, it increases the internal stresses and leads to cracking.



Fig. 1. SEM images of the morphology of alloy coatings.

Fig. 2. shows the weight percent iron and nickel elements in alloy coatings.

The results show that with increasing the amount of ferrous sulfate in the electrolyte, the amount of iron deposition in the coating increases and the deposition of iron in the electrolyte containing 20 g l^{-1} of ferrous sulfate is 21.29 (wt.%), which according to studies by other researchers, this amount iron in the coating can be close to the optimal amount to achieve the desired properties. Table. 2. given the hardness of the samples in Vickers. Hardness of samples by hardness machine. Evolution (evium-vertex) was obtained. Sample 1 is related to the hardness of pure nickel and samples 2 to 4 are related to the samples of alloy coatings formed in the electrolyte containing 20 to 80 g l^{-1} of iron sulfate.



Fig. 2. change of Weight percentage Fe and Ni in alloy coatings via Fe_2O_3 in the plating bath.

As can be seen from the figure, in the 20 g l^{-1} sample, the hardness is at its highest, and in fact decreases with increasing the amount of ferrous sulfate in the bath, or in other words, increasing the amount of iron in the coating.

Table. 2. Microhardness obtained from the samples.

Sample	1	2	3	4	5
FeSO4(wt.%) In the plating bath(g l ⁻¹)	0	20	40	60	80
Microhardness(HV)	220	585	410	330	300

4. Conclusion

1-The 20 g l⁻¹ sample of ferrous sulfate had a higher gloss than other samples.

2-It was observed that for the values of 20 g l⁻¹, 40 g l⁻¹, 60 g l⁻¹, 80 g l⁻¹ iron sulfates 21.29(wt.%), 43.35(wt.%), 51.19(wt.%), 58.8(wt.%) iron were deposited in the alloy coating.

3-According to the SEM images, only in the 20 g l⁻¹ sample of ferrous sulfate, the coating was crack-free and the rest of the samples had cracks.

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