

INFLUENCE OF TECHNOLOGICAL PARAMETERS ABOUT ELECTRODEPOSITION OF NICKEL-IRON ALLOY

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Abstract: The paper presents the results achieved on synthesis and characterization of electrodeposition Ni-Fe alloy obtained for different technological parameters. The investigated characteristics were the efficiency current, the average thickness and the structural properties of the obtained deposition. An inorganic bath with the molar ratio of Ni^{2+}/Fe^{2+} about 4:6 has been used to deposition the samples on cooper substrates. The SEM technique was used to analyze the surface morphology and roughness; and EDS analysis was carried out to determine the composition of the coverage of the alloy. In addition, the micro-hardness and deposit thickness were measured. In all the cases the structure is found to be affected by technological parameters such as current density, temperature, pH consequently having an influence on alloys properties.

Key words: nickel, iron, alloy, codeposition

1. INTRODUCTION

Ni-Fe alloy electrodeposits were an attractive option for industries due to their superior properties (Doo et. al. 1999). The physical properties of the alloys are affected by the composition, the morphology and the structure of the deposit (Leith et. al. 1999, Myung 2001, Su et. al. 2009), properties that are influenced by the technological parameters of the electrodeposition.

It must be highlighted that Ni-Fe alloy electrodeposition has also received a great attention because of the anomalous deposition rate of nickel in presence of iron ions in the electrolyte (Doo et. al. 1999). Finally, it must be pointed out the influence of the experimental conditions onto the hydrogen co-reduction and consequently onto the efficiency current (Leith et. al. 1999, Su et. al. 2009).

The present research aims to study the technological parameters influences, such as current density, temperature and pH, on the electrodeposition of nickel-iron alloy.

2. EXPERIMENTAL

The bath composition was as: $119.983 \text{ g}\cdot\text{L}^{-1} \text{ NiSO}_4\cdot 7\text{H}_2\text{O}$, $3.993 \text{ g}\cdot\text{L}^{-1} \text{ NiCl}_2\cdot 6\text{H}_2\text{O}$, $178.142 \text{ g}\cdot\text{L}^{-1} \text{ FeSO}_4\cdot 7\text{H}_2\text{O}$, $5.015 \text{ g}\cdot\text{L}^{-1} \text{ FeSO}_4\cdot 4\text{H}_2\text{O}$, $12.5 \text{ g}\cdot\text{L}^{-1} \text{ H}_3\text{BO}_3$ (Solmaz & Kardaş 2009). The pH electrolyte was adjusted with 0.1 M H_2SO_4 or 0.1 M NaOH solutions. The home made electrochemical cell, which follows dimensions of 13.9 cm (length), 12.5 cm (width), 10 cm (height), corresponding to a volume of 1.75 L and equipped with a cathode and a mild steel anode, was employed in this study.

The above described cells were connected in galvanostatic regime to power supply. The electrolysis time was fixed in order to use in 900 C of electricity. The temperature of electrolyte solution was kept constant at the desired value using Hotplate Stirrer SB 302 Stuard. The electrodeposition was carried with constant stirring of the bath solution with a magnetic stirrer. The tested conditions are given in Table 1:

Exp. No.	Current density [$\text{A}\cdot\text{dm}^{-2}$]	Temperature [$^{\circ}\text{C}$]	pH
1.	1.75	25	2.5
2.	3.51	25	2.5
3.	1.75	35	2.5
4.	3.51	35	2.5
5.	1.75	25	3.5
6.	3.51	25	3.5
7.	1.75	35	3.5
8.	3.51	35	3.5

Tab. 1. Electrodepositing conditions tested

The plating current efficiency (CE) for the deposition of each alloy was calculated according to the following equation:

$$CE = \frac{(m \cdot Fe\%) / Eq_1 + (m \cdot Ni\%) / Eq_2}{I \cdot t} \quad (1)$$

where: m is the deposit weight [g], Fe% and Ni% [wt.%] are the percents of iron and nickel in deposits, respectively, Eq_1 and Eq_2 are the electrochemical equivalent [$\text{g}\cdot\text{C}^{-1}$] of Fe^{2+} and Ni^{2+} , respectively, I is the deposition current [A], and t is the deposition time [s]. The morphology of the deposits was observed with a scanning electron microscope (SEM). The attached energy dispersive spectroscopy (EDS) was used to determine the approximate composition of the alloy. The roughness [μm], measurement was performed by analyzing images acquired by SEM. The thickness of metallic layer was measured by means of analyzer, based on the non-destructive physical method. The micro-hardness, [HV], of the metallic cover was measured using the micro-hardness tester. The loading weight was of 490.3 mN and the load duration of 15 s respectively. All experiments and tests were carried out at the Faculty of Food Engineering from "Stefan cel Mare" University of Suceava, Romania.

3. RESULTS AND DISCUSSION

Figure 1 presents the influence of the technological parameters tested (current density, temperature, pH) on the composition of the electrodeposits and on the efficiency current (curve --▲--). It can be observed that at a temperature of 25 $^{\circ}\text{C}$ both at pH = 2.5 (exp. no. 1-2), and at pH= 3.5 (exp. no. 5-6), the Fe content is reduced and the Ni content increases respectively with increase current densities, while at the temperature of 35 $^{\circ}\text{C}$ for both pH ranges (for instance, no. 3-4 and 7-8, respectively) compositions are approximately equal regardless of current density.

This increase of % Ni with current density is known as anomalous codeposition (preferential deposition of less noble Fe) and is due the depletion of more readily depositable iron at cathode (Thangaraj & Hegde, 2007). Because in the present study, the Ni^{2+}/Fe^{2+} ratio was high, the % of Ni in the deposit tends to increase with current density.

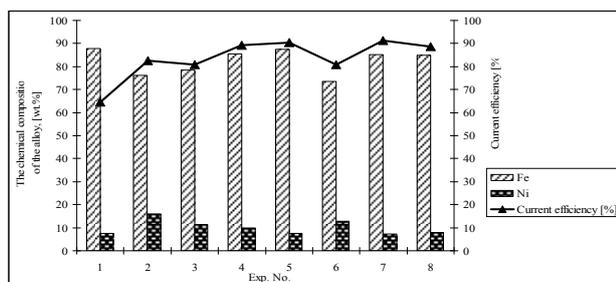


Fig. 1. The effect of technological parameter on chemical composition of the alloy and the current efficiency of electrodeposition

The Ni% in the deposit increases with temperature, (exp. no. 1-3 and 5-7) at a current density of $1.75 \text{ [A}\cdot\text{dm}^{-2}]$. This is due to the fact that an increase in temperature favours the deposition at that metal which was preferentially deposited, because it speeded up the diffusion and thus relieved the depletion of metal at the cathode. As the system follows anomalous codeposition, the Fe is being deposited preferentially, an elevation of temperature has increased the content of the iron as depicted in exp. no. 2-4 and 6-8 respectively) at current density of $3.51 \text{ [A}\cdot\text{dm}^{-2}]$. It is also observed that percent of iron (Fe [wt. %]) in the deposits slightly increases with the increasing pH of the bath.

As expected, the current efficiency increases with the pH increment, the pH being connected with hydrogen evolution. At $\text{pH} = 2.5$, the enhancement of the current density leads to higher current efficiency, but at a pH value of 3.5, the current efficiency decreases with increasing current densities.

From the SEM images of the NiFe binary coatings (figure 2) it can be seen that different microstructures are obtained for the different technological parameter of electrolysis.

In industrial applications, the layer thickness is a critical parameter, since this parameter may determine whether the deposition meets the requirements or not. The hardness measurements are useful in the evaluation of the deposits and for predicting their long time usefulness. Frequently, changes in alloy hardness reflect variations in the structure or composition of the alloy deposit. Roughness is an important parameter used in particular to characterize of NiFe electroactive coatings as electrocatalysts for hydrogen evolution in an acidic (Navarro-Flores et. al. 2005) or alkaline (Solmaz & Kardaş 2009) medium.

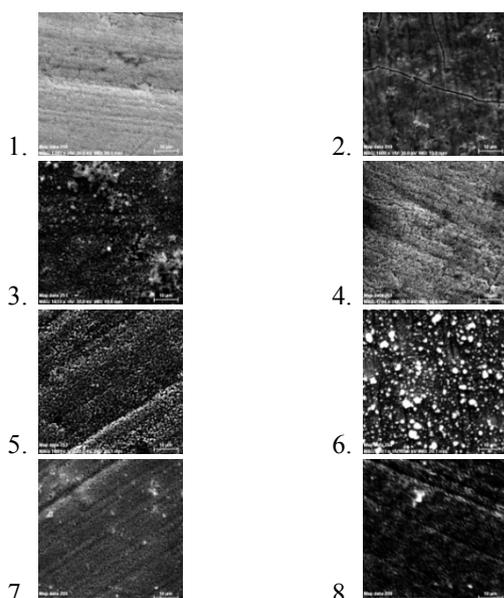


Fig 2. SEM micrographics showing the surface morphology of electrodeposited NiFe alloy at conditions in Table. Note: MAG: $\approx 1800\times$, HV:30.0 KV, WD: $\approx 20.0 \text{ mm}$

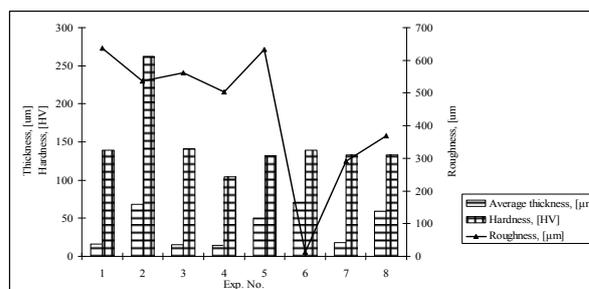


Fig. 3. The influence of technological parameter on thickness, hardness and roughness of NiFe coatings

The influence of the technological parameters tested (current density, temperature, pH) on the thickness, hardness and roughness of the obtained depositions are given in figure 3.

The chemical composition and roughness values for electrodepositions obtained in the experimental conditions 1 and 5 are similar to those presented in literature (Solmaz & Kardaş 2009).

4. CONCLUSIONS

Because Ni-Fe alloy electrodeposition shows an anomalous behavior, it needs testing the technological parameters electrolyses in order to obtain deposits with a given composition, morphology, structure and properties.

The increase of % Ni in the deposit at high current density is attributed to high ratio of $\text{Ni}^{2+}/\text{Fe}^{2+}$ in the bath.

The effect of temperature on the plating process showed that the codeposition of metals is diffusion controlled.

5. REFERENCES

- Doo S.K., Sohn H.J. & Kang T., (1999), Mathematical Modeling of Electrodeposition of Permalloy Thin Film I. Direct Current Plating, *Metals and Materials*, 5, 451-457, Available from: <http://www.springerlink.com> , Accessed: 2010-03-05
- Leith S., Ramli S. & Schwartz D.T. (1999), Characterization of $\text{Ni}_x\text{Fe}_{1-x}$ ($0.1 \leq x \leq 0.95$) electrodeposition from a Family of Sulfamate-Chloride Electrolytes, *Journal of The Electrochemical Society*, 146 (4), 1431-1435, Available from: <http://ias.ac.in/pramana/v66> , Accessed: 2010-03-05
- Myung N. (2001), A Study on the Electrodeposition of NiFe Alloy Thin Films Using Chronocoulometry and Electrochemical Quartz Crystal Microgravimetry, *Bull. Korean Chem. Soc.*, 22, 9, 994-998, Available from: <http://www.newjournal.kcsnet.or.kr.>, Accessed: 2010-03-05
- Navarro-Flores E., Chong Z. & Omanivic S. (2005). Characterization of Ni, NiMo, NiW and NiFe electroactive coatings as electrocatalysts for hydrogen evolution in an acidic medium, *Journal of Molecular Catalysis A: Chemical*, 226, 179-197, Available from: <http://www.sciencedirect.com>, Accessed: 2010-01-07
- Solmaz R. & Kardaş G. (2009). Electrochemical deposition and characterization of NiFe coatings as electrocatalytic materials for alkaline water electrolysis, *Electrochimica Acta* 54, 3726-3734, Available from: <http://www.sciencedirect.com>, Accessed: 2010-01-07
- Su C.-W., Wang E.-L., Zhang Y.-B. & He F.-J. (2009), $\text{Ni}_x\text{Fe}_{1-x}$ ($0.1 \leq x \leq 0.75$) alloy foils prepared from a fluoroborate bath using electrochemical deposition, *Journal of Alloy and Compounds*, 474, 190-194, Available from: www.elsevier.com , Accessed: 2010-02-06
- Thangaraj V. & Hegde C.A. (2007), Electrodeposition and compositional behaviour of Zn-Ni alloy, *Indian Journal of Chemical Technology*, 14, 246-252, Available from: www.elsevier.com/locate/jallcom , Accessed: 2010-02-06