Normal Electrochemical Deposition of NiFe Films

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Author's contribution

The sole author designed, analyzed and interpreted and prepared the manuscript.

Article Information

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ABSTRACT

The electrodeposition of Fe-Ni alloys was performed galvanostatically in chloride solutions of pH 1.5–2.5 at 70°C, and the deposition behavior was investigated. The presence of hydroxide iron in the electrolyte was the main cause of anomalous codeposition. Hydroxide iron precipitates were eliminated by using boric acid and filtration. The formation of iron ions, Fe^{3+}, was inhibited by hydrochloric acid. The chloride electrolyte is stability and elimination of anomalies deposition. Changing the current process does not lead to a noticeable change in the content of iron and nickel in the film. The current value is selected when the contents of iron and nickel change due to additives in the electrolyte leads to modify the content of the film. Congruent deposition of FeNi occurs with relevant electrolyte compositions and films.

Keywords: Permalloy; electrochemical deposition; chloride electrolyte; hydroxide iron precipitates.

1. INTRODUCTION

Electroplating of nickel-iron alloys has been known for many years [1-4]. Nickel-iron, especially permalloy, plating has been used for more than 60 years, but there are still several problems related to stability and maintenance that should be resolved. The mechanism of

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nickel-iron alloy plating is of the so-called anomalous type [3], which means that the iron is plated preferentially, even though it is not the more noble metal of the two. The formation of Fe^{3+}-based precipitates slowly destroys the electrolyte. Adding a suitable complexing agent for Fe^{3+} postpones the formation of ferric compounds.

The preferred plating bath for electrodeposition of nickel-iron alloys, particularly $Ni_{81}Fe_{19}$ or permalloy, consisted of simple mixtures of the sulfate or chloride salts of nickel and iron. The typical bath would also contain boric acid as a pH buffer and a few organic additives, such as lauryl sulfate (wetting agent) and saccharin (stress reducer) [5-19].

With the objective of testing several models of the "anomalous codeposition" (ACD) that is encountered in the electrodeposition of nickel-iron alloys, the effects of bath pH and complexing agents on the composition of deposits were examined [20-26].

When two or more metals are electrodeposited simultaneously, the elemental composition of the resulting film does not necessarily reflect the composition of the deposition solution. In particular, for binary alloys of two iron group metals (iron, cobalt, and nickel) and alloys of iron group metals with zinc or cadmium, the less noble metal deposits preferentially for a wide range of deposition conditions. ACD results in a larger concentration of the less noble metal in the film than in the solution. The extent of the anomalous behavior for a particular alloy system has been shown to depend on a variety of experimental parameters, including the metal concentrations in the solution, pH, and presence of additives, deposition potential, deposition current density, and agitation of the solution during deposition.

2. THE MECHANISM OF ANOMALOUS DEPOSITION OF FE-NI ALLOYS

It is important to clarify the mechanism of anomalous deposition of Fe-Ni alloys. The hydroxide suppression model [20] and the mathematical model [21] were proposed to explain the anomalous codeposition of Fe with Ni. The hydroxide suppression model [20] explained that noble Ni deposition was strongly suppressed in the presence of $Fe(OH)_2$ and preferentially formed and adsorbed on the cathode, and the mathematical model [21] was based on the great difference in the dissociation constant between $FeOH^+$ and $NiOH^+$.

The electrodeposition of Fe-Ni alloys was performed [23] galvanostatically in sulfate solutions of pH 1–3 at 40°C. The deposition behavior of the Fe-Ni alloy showed a typical feature ACD, in which electrochemically less noble Fe deposits preferentially under most plating conditions. ACD behavior in Fe-Ni alloy deposition was evidently dependent on the pH buffer capacity of the solutions. This can be explained in terms of the preferential adsorption of FeOH on the deposition sites of more noble Ni due to the much smaller dissociation constant of $FeOH^+$ than of $NiOH^+$ in the multi-step reduction process of hydrated iron-group metal ions.
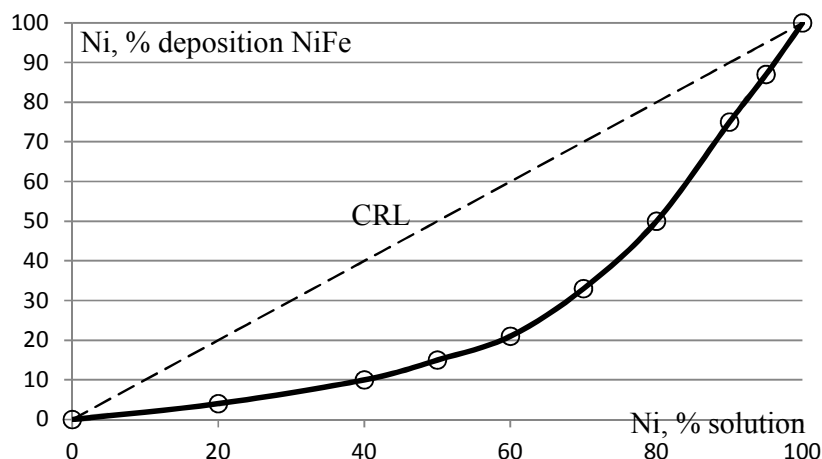

Fig. 1. Effect of solution composition on the alloy composition of Fe-Ni alloys electrodeposited from sulfate solutions at 100 mA/cm^2

Fig. 1 shows the relationship between the alloy content of Ni, which is more noble than other constituent in the deposited Fe-Ni, and the composition of the solutions. The CRL in the figure expresses the composition reference line that shows when the mass percentage composition of the alloy just equals the metal percentage of the solution. When the Ni content of the alloys is higher than the CRL, a normal type alloy deposition occurs, thus indicating the preferential deposition of the more noble Ni. In contrast, as shown in Fig. 1, the Ni content of the alloys was lower than the CRL over the entire composition range of the solutions. This result indicates that anomalous codeposition occurring during the Fe-Ni deposition.

In Fe-Ni alloy plating solutions, the solubility product of $Fe(OH)_2$ is 2.2×10^{-15}, which is almost identical to that of $Ni(OH)_2$. The critical pH values calculated from these solubility constants are 7.0 and 6.8 for the precipitation of $Fe(OH)_2$ and $Ni(OH)_2$, respectively. Therefore, both metal hydroxides are considered to precipitate almost simultaneously when the pH of the cathode layer increases during alloy deposition. Consequently, the $Fe(OH)_2$ suppression mechanism [20] seems to be invalid for Fe-Ni alloy deposition.

Another mechanism of Fe-Ni alloy deposition proposed [21]. They assumed that the deposition of iron-group metals proceeds with the previous formation of metal hydroxide ions, as expressed by the following reactions.

$$Me^{2+} + OH^- = MOH^+ \qquad (1)$$

$$MOH^+ + 2e^- = M + OH^- \qquad (2)$$
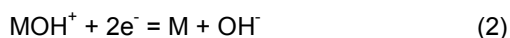
In the above reactions, M expresses iron-group metals. Since the dissociation constants of $FeOH^+$ and $NiOH^+$ are 5.78×10^{-8} and 4.50×10^{-5}, respectively, the hydroxide ion concentration is almost a thousand times smaller in $FeOH^+$ than in $NiOH^+$ in the cathode layer. As a result, Fe deposits preferentially under most plating conditions. The reason why the Ni deposition rate was suppressed in Fe-Ni alloy solutions cannot be explained. Ni deposition was suppressed only in codeposition with Fe, indicating that the mechanism proposed by [21] also cannot be accepted.

The increase in pH in the cathode layer was confirmed by directly measuring the pH. The result showed that the concentration of the metal hydroxide ion was 0.21 M/L for Fe and 10^{-3} mol/L

for Ni. This indicates that $NiOH^+$ is scarcely formed but $FeOH^+$ is formed in the cathode layer when the current density is increased to permit ACD.

The Fe single solution showed a distinct shoulder at the pH range between 6.3 and 7.0 before reaching the critical pH for $Fe(OH)_2$ precipitation. This indicated the formation of $FeOH^+$, as predicted by the theoretical calculation described above. Therefore, in the anomalous type Fe-Ni alloy deposition, the $FeOH^+$ must be formed in preference to $NiOH^+$. In the Fe-Ni alloy deposition, $FeOH^+$ acts as an inhibitor that decreases the deposition rate of the nobler Ni.

This mechanism of the anomalous type Fe-Ni alloy deposition has a very narrow range of the pH of the electrolyte for possible implementation.

The concept of repressing co-precipitation can be observed in all works.

Romankiw [24] presents the role of a hydroxide present in the electrolyte.

Thommes et al. [25] found an apparent influence on the deposition process in sulfate electrolyte trivalent iron ions, but the authors had difficulties describing this effect. They were limited to examining models of transfer due to convection or diffusion with a rotating cathode and models [21,26].

3. THE DEPOSITION PROCESS OF CHLORIDE ELECTROLYTE THE ALLOY NI$_{81}$FE$_{19}$

Due to the instability of sulfate electrolytes and the presence of sulfur in NiFe films, using chloride electrolytes seems more promising for the electrodeposition of Ni-Fe alloys from chloride baths.

Nanocrystalline Ni-Fe deposits with iron contents of 7, 12, and 31% were obtained [16] by changing the Ni^{2+}/Fe^{2+} mass ratio to 20,7; 13,8; 9,8 in the chloride electrolyte at 56°C, pH=2.5.

Tikhonov [19] examined the deposition process of a chloride electrolyte with a ratio of atoms of 4.26 in the alloy Ni$_{81}$Fe$_{19}$ at 70°C, pH=3.5. When the current density is changes, the iron content in the film varies from 16.1 to 26%. When the current density is 12.2 mA/cm^2 and the content of iron in the film is 19.4%, corresponding to the composition of the electrolyte,

the electrochemical deposition can be called congruent, unlike the anomalous codeposition.

In this study, we investigated the dependence of the properties of the chloride electrolyte on the cooking techniques of the solutions.

4. TECHNIQUE OF PREPARATION OF CHLORIDE ELECTROLYTE

The preparation of chloride electrolytes was investigated using the spectrophotometer B-1100. A series of experiments was carried out with consistent dissolution of electrolyte components in water and a spectrophotometric control.

The attachments $FeCl_2$ $4H_2O$ - 11 +11 +22 = 44 g/l (C_{Fe} = 232 mM) made at the derived water solutions and the spectral transmittance coefficients solutions were sequentially introduced. The dependence of the optical transmittance at wavelengths 315-1050 nm is presented in Fig. 2, as measured in a 10-mm ditch.

The solution is yellow-brown. It primarily looks yellow with a brown sheen of floating particles in the fluid. A suspension of not coagulating precipitates is present in the primary fluid. Light absorption peaks are observed at wavelengths of 345 nm and 945 nm.

After the boric acid is added, the spectrogram changes away from the peaks of absorption (Fig. 3). The solution has a weak acidity, at pH= 4 ÷ 3. After settling for 7 days, the solution color changes to yellow-green, and reddish-brown sediment drops out. The solution is filtered, and the composition of the sediment is analyzed.

The interaction of a sludge of hydroxide iron (III) with a solution of hydrochloric acid leads to the characteristic response for $Fe(OH)_3$, the dissolution of sediment and the formation of a yellow solution of iron (III) chloride. After flushing, the filtrate water contains atoms Cl-15.69% and Fe-81.27%. The relative iron content in the sludge is great. The precipitate is iron hydroxide $Fe(OH)_3$.

Changing the spectral transmittance coefficients for the sequential introduction of a solution of nickel chloride hydrate attachments $NiCl_2$ $6H_2O$ to 224 g/l gives the following spectral dependence of light transmission, as presented in Fig. 4. The spectral transmittance coefficients are characterized by peaks of light absorption at wavelengths of 395, 725 and 985 nm. When the concentration of nickel chloride is increased, the solution darkens, but the spectral characteristics are practically unaltered. The solution with the additive of boric acid H_3BO_3 40 g/l has a yellow-green color. After settling for 7 days, it does not change color and has no precipitate.
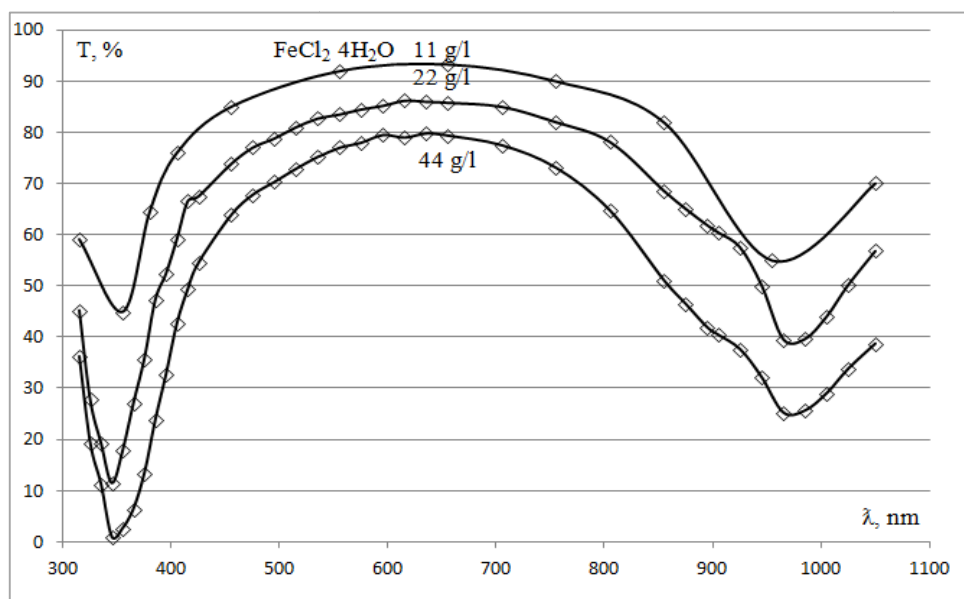

Fig. 2. Optical transmittance T at wavelengths 315-1050 nm in aqueous solution FeCl₂ 4H₂0

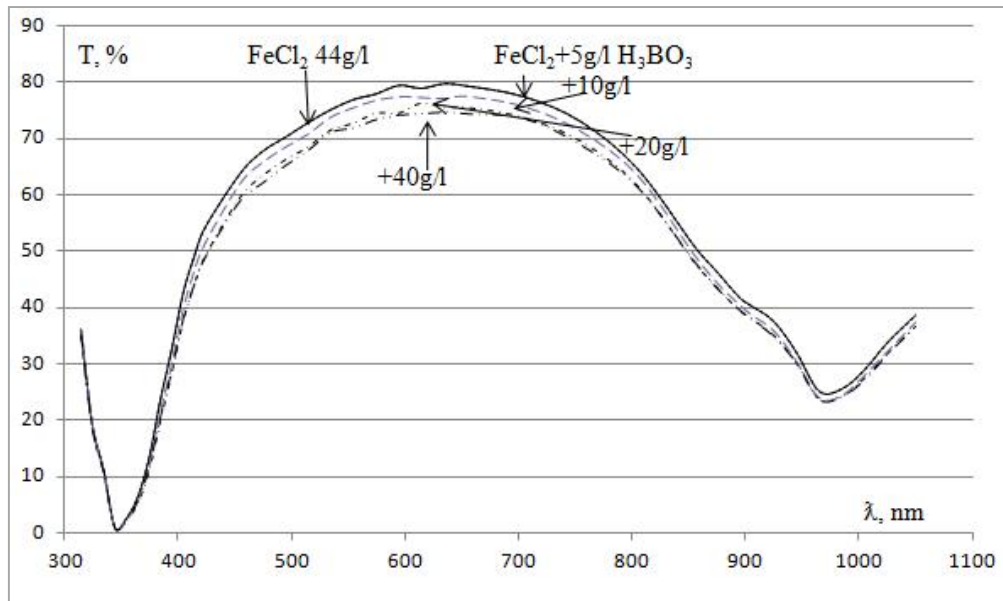**Fig. 3. Optical transmittance T at wavelengths 315-1050 nm in aqueous solution FeCl$_2$ 4H$_2$0 + H$_3$BO$_3$**

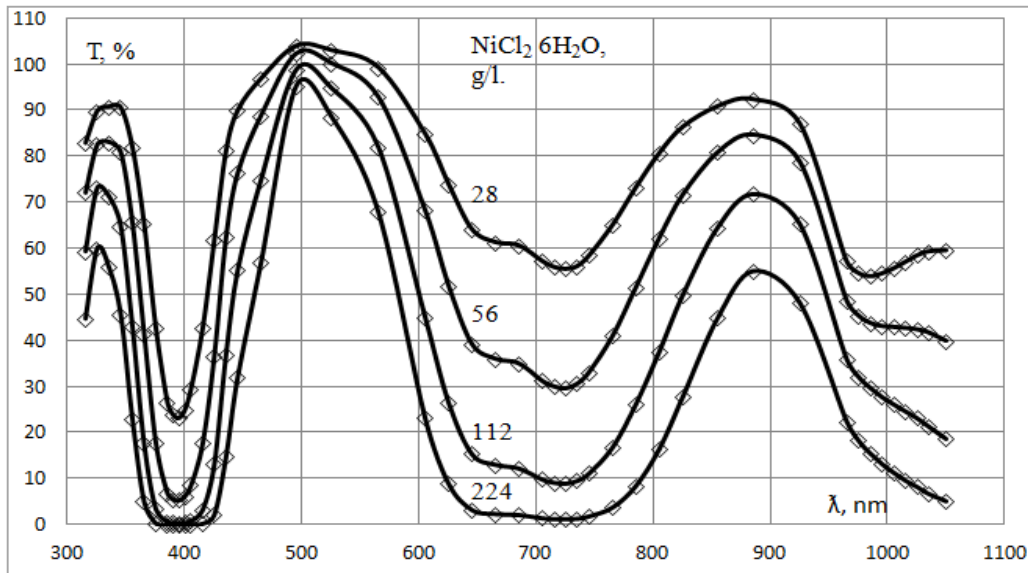**Fig. 4. Optical transmittance T at wavelengths 315-1050 nm in aqueous solution NiCl$_2$ 6H$_2$0**

The chloride electrolyte from solutions with iron, nickel and boric acid is shown in a spectrofotogramma in Fig. 5. The spectral transmittance coefficients when mixing solutions of ferric chloride and nickel are characterized by peaks of light absorption at wavelengths of 395, 725 and 985 nm, which correspond to the nickel, and the solution has the largest concentration of nickel, 4.26 times more than iron.

The addition of saccharin 1.6 g/l reduces the transmission wavelengths of 500 and 900 nm. It also increases the acidity, pH = 3.17. The addition of hydrochloric acid 2 g/l reduces the absorption of light at wavelengths of 540 and 860 nm, and the electrolyte becomes noticeably lighter. It also increases the acidity, pH = 1.73. The electrolyte is stabilized.
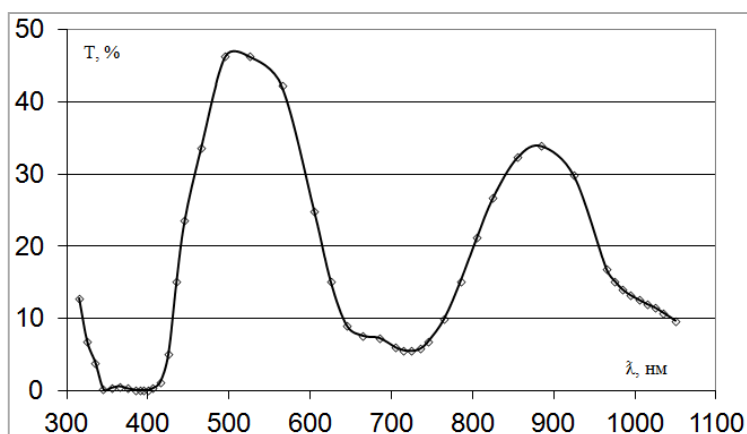
Fig. 5. Optical transmittance T at wavelengths 315-1050 nm in aqueous solution NiCl$_2$ 6H$_2$0 + FeCl$_2$ 4H$_2$0 + H$_3$BO$_3$

5. CHEMICAL PROCESSES IN A SOLUTION OF FERRIC CHLORIDE

The chemical processes in a solution of ferric chloride dissolved in water [27] in the sludge can precipitate iron (II) hydroxide Fe(OH)$_2$. Hydrolysis of ferric chloride (II) occurs in an acidic solution.

$$FeCl_2 + H_2O \leftrightarrow Fe(OH)Cl + HCl$$

$$Fe(OH)Cl + H_2O \leftrightarrow Fe(OH)_2\downarrow + HCl$$
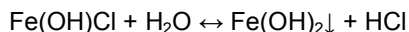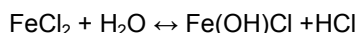
Iron (II) hydroxide has a grayish-green color, does not dissolve in water, and darkens quickly due to oxidation. Similar to all other compounds of divalent iron, it has restorative properties and, in the presence of O$_2$ and H$_2$O, will slowly oxidize dissolved oxygen to hydroxide iron (III) when standing in air.

$$4Fe(OH)_2 + O_2 + 2H_2O = 4Fe(OH)_3 \qquad (1)$$
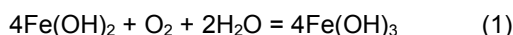
Iron (III) hydroxide with the formula Fe$_2$O$_3$ nH$_2$O has a reddish-brown color and is not soluble in water. The formation of iron hydroxide in the solution is complemented by its presence in the original solution. The content of trivalent iron was assumed to be that indicated on the chemical passport. The main content with a molar mass of 198.83 was 99% of the solution, the impurities of trivalent iron were 0.2%, and the remaining impurities were individually each less than 0.01%. The percentage of converted ferric iron in relation to the content of bivalent iron in ferric chloride hydrate is 0.7%.

The interaction of the brown sludge of hydroxide iron (III) with a solution of hydrochloric acid leads to the dissolution of the precipitate and the formation of a yellow solution of iron (III) chloride.

$$Fe(OH)_3 + 3HCl = FeCl_3 + 3H_2O \qquad (2)$$

The instability of bivalent iron and converted ferric iron is the source of the difficulty in obtaining a given composition of permalloy films. In a number of standard electrode potentials of metals, Fe^{2+} has a value of -0.44 V, and Ni^{2+} has a value of -0.25 V. In accordance with these potentials, iron should precipitate worse than nickel. However, the electrode potential Fe^{3+} is set to -0.036 V. Fe^{3+} + 3e$^-$ → Fe(solid) − 0.036 V [28]. With this electrode potential, converted ferric iron precipitates much faster than nickel does and creates uncertainty in obtaining the whole film from an electrolyte with a specified composition of bivalent iron.

The interaction of iron (III) chloride and boric acid may be due to the reaction of trivalent iron and trivalent boron.

$$FeCl_3 + B(OH)_3 = BCl_3 + Fe(OH)_3. \qquad (3)$$
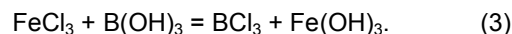
Boron chloride, with the formula BCl$_3$, is a colorless gas that evaporates. The sediment Fe(OH)$_3$ falls out of the solution. Boric acid cleanses the electrolyte from the trivalent iron for a high pH aqueous solution.

Obtaining reproducible results of the electrochemical deposition of permalloy requires respect for the principle of electrolyte purification from admixtures of trivalent iron. From a FeCl$_2$ solution with boric acid forms complexes and highlights of a reddish-brown precipitate,

which are removed by filtration. Data on the composition of the filtrate were obtained using an X-ray Philips XL 40. The composition of the sediment, Cl-15.69% and Fe-81.27% iron, is considerably greater than the chloride of iron component, i.e., iron hydroxides.

6. COMPOSITION OF PERMALLOY FILMS DURING ELECTROCHEMICAL DEPOSITION FROM CHLORIDE ELECTROLYTE OF FILTERED SOLUTION OF FERRIC CHLORIDE AND BORIC ACID

The electrochemical deposition of the chloride electrolyte and the content of nickel and iron atoms in relation to a 4.26 corresponding alloy $Ni_{81}Fe_{19}$ at a temperature of 70°C gave the composition dependence of permalloy films in the current range 270-410 mA, as shown in Fig. 6.

Changing the current process does not lead to a noticeable change in the content of iron and nickel in the film.

The current value is selected when the contents of iron and nickel change due to additives in the electrolyte leads to modify the content of the film.

The nickel content in FeNi films is higher than in the electrolyte, as seen in Fig. 7. An equal deposition of nickel as in the electrolyte is indicated by a dashed stroke. Therefore, nickel is

precipitated better than iron and anomalous codeposition is not observed.

Increasing the concentrations of ferric chloride hydrate to 4.5% compensates for the accelerated deposition of nickel and achieves the corresponding permalloy film composition $Ni_{81}Fe_{19}$.

7. DISCUSSION OF RESULTS

When our electrolyte components concentration has a pH = 1.7, the braking mechanism of deposition of nickel iron hydroxide [23] cannot lead to anomalous deposition.

The changing spectrometric data of the electrolyte sulfate over time showed [14] that the Fe peak rapidly increased and dominated the spectrum within 25 days. The growth peak of Fe is interpreted as the result of the oxidation of Fe^{2+} ions to Fe^{3+}. Over 20% of the originally existing Fe^{2+} ions disappeared due to deposition or oxidation, and the ion content of Fe^{3+} has increased. The sulfate electrolyte is unstable.

For two months, a chloride electrolyte, a filtered solution of ferric chloride and boric acid with an addition of hydrochloric acid to obtain a pH = 1.7, underwent 24 deposition processes with a total thickness of all films of 167 µm. The electrolyte was exhausted and did not change their spectral characteristics. The chloride electrolyte, with the removal of trivalent iron and laced with hydrochloric acid, was stable and provided a reproducible process.
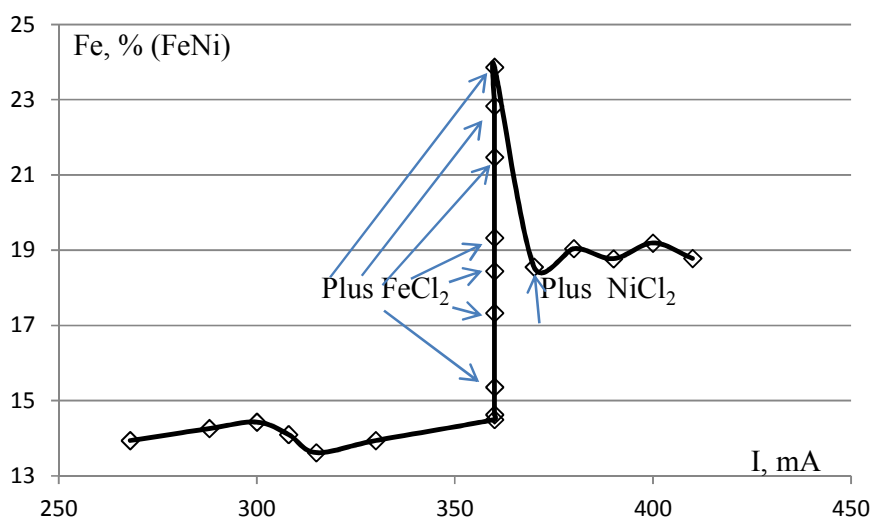
Fig. 6. Electrochemical deposition of FeNi from chloride electrolyte, filtered Fe(OH)₃
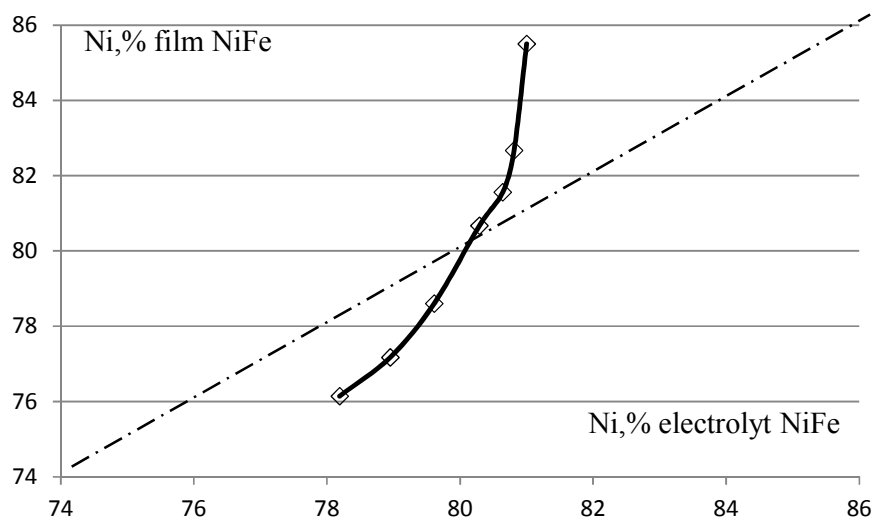
Fig. 7. The dependence of the composition of the NiFe film on the electrolyte composition

In [20 -26,29-31], the effect that hydroxides have on the mechanism of anomalous permalloy deposition process is seen as a result of electrochemical transformations in the electrolyte during the deposition and formation of iron hydroxide, which inhibits the deposition of nickel. This work differs in that the effect of hydroxides, which are available in the prepared solution, are considered in the chemical processes that occur in a solution of ferric chloride. The instability of bivalent iron and converted ferric is the source of the difficulty in obtaining a given composition of permalloy films because converted ferric iron is deposited at a higher speed than bivalent iron and, in an uncertain manner, increases the iron content in the film.

After filtering the solution of ferric chloride boric acid there is no $Fe(OH)_3$. The removal of hydroxide precipitates from the electrolyte using boric acid and filtration eliminates the anomalous permalloy deposition process. Additive hydrochloric acid decreases the pH and electrolyte disturbances, eliminates the possibility of education hydroxides precipitates and gives stable composition permalloy films.

8. CONCLUSION

It seems almost incredible that within 60 years, researchers have observed the electrodeposition of Fe-Ni alloy and the preferential deposition of iron in relation to nickel in all formulations of the electrolyte and the dependence of the whole sediment from current density. The concept of repression of co-precipitation is seen in all works

[20-26,29-31] because the common explanation only details hydroxides. None of the researchers linked the anomalous character of electrodeposition of iron ions to the main feature - the existence of two valences in Fe.

This paper shows that the deposition process has a considerable influence from the chloride electrolyte preparation methodology and that the interaction of main and auxiliary chemicals should be considered. Electrolyte purification of precipitates of iron hydroxide using boric acid and filtration suppresses trivalent iron ions Fe^{3+}, and a hydrochloric acid electrolyte ensures stability and a reproducible permalloy film deposition.

The deposition process of the chloride electrolyte, cooked with iron precipitates with boric acid and the subsequent stabilization of hydrochloric acid, gives new properties, such as the independence of the whole from the current density and the excess deposition of nickel over iron. This opens new possibilities for obtaining the desired composition of permalloy films by specifying the composition of the electrolyte [32].

COMPETING INTERESTS

Author has declared that no competing interests exist.

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