



Research article

Model of electrochemical deposition NiFe: experiments and theory

Tikhonov R.D.

SMC “Technological Centre”, RF, Moscow, Zelenograd, 124498,
e-mail: R.Tikhonov@tcen.ru



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Abstract

Enhancement of magnetic induction is of great interest to developer's magnetosensitivity integrated microsystems. To use films as a $\text{Ni}_{81}\text{Fe}_{19}$ of magnetic field concentrators significantly, due to heating of the electrolyte is excluded abnormal codeposition alloy components and reduced variation of process parameters to achieve optimal magnetic properties. Proposed chloride electrolyte pH adjusted with hydrochloric acid, which provides congruent electrochemical deposition of permalloy at heating and stirring. Magnetic properties of relevant films are received massive designs. Magnetic properties of permalloy films are very sensitive to rejection of the whole component of 4.26. Control of accuracy of preparation of chloride electrolyte for electrochemical deposition of NiFe conducted using spectrophotometry.

Keywords: permalloy, magnetic field concentrators, electrochemical deposition, chloride electrolyte, spectrophotometric monitoring.

Introduction

NiFe alloy has high maximum relative magnetic permeability $\mu \sim 100\,000$, low coercivity 1 Oe. These properties are manifested in a narrow range of changes in the composition 81% Ni [1]. The possibility of strengthening the magnetic induction to five orders of magnitude is of great interest to developers magneto sensitivity integrated microsystems [2].

Magnetic properties of permalloy vary if there are impurities in the alloy. Negatively on the properties of permalloy influence of impurity, which do not form solid solutions with alloy, such as carbon, sulfur and oxygen. Properties of permalloy sharply deteriorating mechanical stresses. To obtain high magnetic permeability requires high temperature processing in 1125 °C sheets and tapes after rolling. The combination of many factors influencing magnetic properties of permalloy, defines a wide range of their change and the difficulty of playing high magnetic parameters.

Applied electrolytes are related nickel and iron atoms as more than 4.26 for the alloy $\text{Ni}_{81}\text{Fe}_{19}$ and less. There is no electrolyte for electrochemical deposition congruent.

1. An empirical study of the process of electrochemical deposition permalloy

To obtain local structures of magnetic field concentrators researched magnetic properties of permalloy films with thick, obtained by the method of electrochemical deposition permalloy in local area using chip photoresist masks metallized surface of the silicon wafer [3]. These areas increase the magnetic field due to



magnetization, and serve as hubs, enabling low coercive force and high magnetic permeability of the lower bound of the range of tension of an external magnetic field, measured magneto sensitivity elements of integrated microsystems.

Heat the electrolyte improves wettability, aligns the thickness 20 μ of the hub by improving adhesion and reduces tension in the layer. Figure 1 presents the iron content dependence in permalloy film electrolyte temperature without mixing.

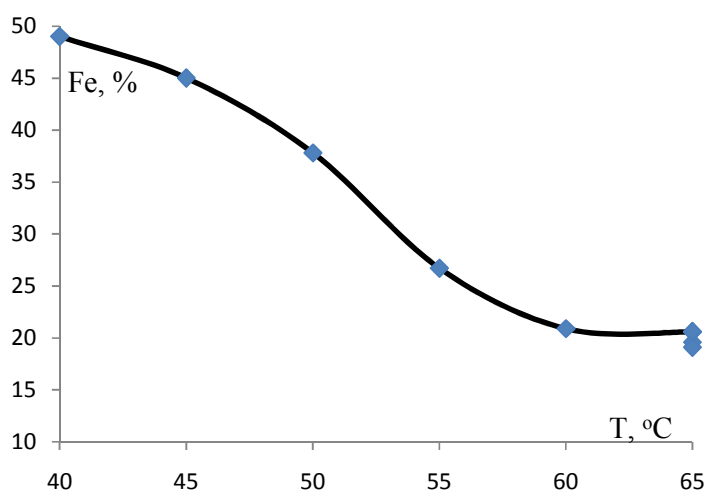


Figure 1. Iron content dependence of Fe in film of permalloy versus with electrolyte temperature T°C.

The composition of the beleaguered film is determined by the temperature of the electrolyte. At a temperature of 40°C composition of the films $Ni_{51}Fe_{49}$ and at temperatures of 60 and 65, respectively, $Ni_{79.1}Fe_{20.9}$, and $Ni_{79.4}Fe_{20.6}$. The temperature dependence of the composition is stabilized at 60°C. The film is very neat and clean, with almost uniform composition on the surface of the hubs.

Change the deposition rate at high temperatures the electrolyte provides much less dependent on current density that allows you to adjust the composition in a fairly narrow range of current change to get the tapes close in composition to permalloy $Ni_{81}Fe_{19}$.

The electrochemical installation with a galvanic bath for 3 L of electrolyte enables us to maintain its temperature automatically and to mix the solution during the whole deposition process. The electrodes of the installation are arranged vertically, the anode is of nickel foil, and the cathode is with a nickel ring electrode contacting the metallized platform of the silicon wafer.

With the aim of eliminating unwanted contaminants and increasing the rate of growth of the thin film deposition process, the chloride electrolytes were studied. A chloride electrolyte with a ratio of Ni to Fe of 4.26 in the $Ni_{81}Fe_{19}$ alloy and an electrolytic composition of $FeCl_2 \cdot 4H_2O$ (5.8 g/l), $NiCl_2 \cdot 6H_2O$ (28 g/l), H_3BO_3 (30 g/l), and $C_6H_8O_7 \cdot H_2O$ (3 g/l) at pH 3.5, increased the speed of growth and thickness of the films by 2-3 times and increased the saturation induction. It is very important to regulate the pH of the electrolyte by adding hydrochloric acid, which prevents the formation of sludge and makes the electrolyte stable.

The magnetic properties of the films are illustrated by the magnetization curves for a 24 micron thick permalloy film covering a square section on a silicon plate with a diameter of 80 mm. The obtained values are a magnetic induction saturation B_s of 1.354 T, a relative magnetic permeability μ of 3303, and a coercivity H_c of 0.5 Oe.

To obtain high magnetic permeability and magnetic induction saturation of the permalloy at high temperature, annealing is applied followed by slow cooling to form the structure of the material at the macroscopic level, removing defects and internal grain stress that arise in the material during rolling. In films,



the structures do not have the large number of structural defects that occur from machining. The formation of an alloy structure occurs from electrochemical deposition of ions, which affects the formation of structures, and creates films with good magnetic properties without high-temperature annealing.

2. The exception is the influence of valency iron on electrodeposition of permalloy

Electrodeposition of NiFe-alloys galvanostatically from the solution implemented chloride electrolyte with pH = 1.5-2.5 at 70°C for determining the mechanism of co-precipitation. Effect of ferric ion on the permalloy deposition process shall be considered an act of ferric ion in source electrolyte. Elimination of ferric iron precipitates using boric acid and filtration and suppresses education trivalent iron ions Fe^{3+} hydrochloric acid electrolyte ensure stability, eliminate anomalous deposition process and congruent is a characteristic feature of codeposition of NiFe alloy in which the mol'nymi composition of the precipitate corresponds to composition of the electrolyte.

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

The spectral transmittance coefficient when sequentially adding attachments into a solution of ferric chloride hydrate is characterized by peaks of absorption of light at wavelengths of 345 nm and 945 nm. After additive of boric acid 80 g/l spectrogram in Figure 2 is not changed, then the light transmission is determined by solution of $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$ 90 g/l after filtering the absorption of light is reduced in areas away from the peaks of absorption.

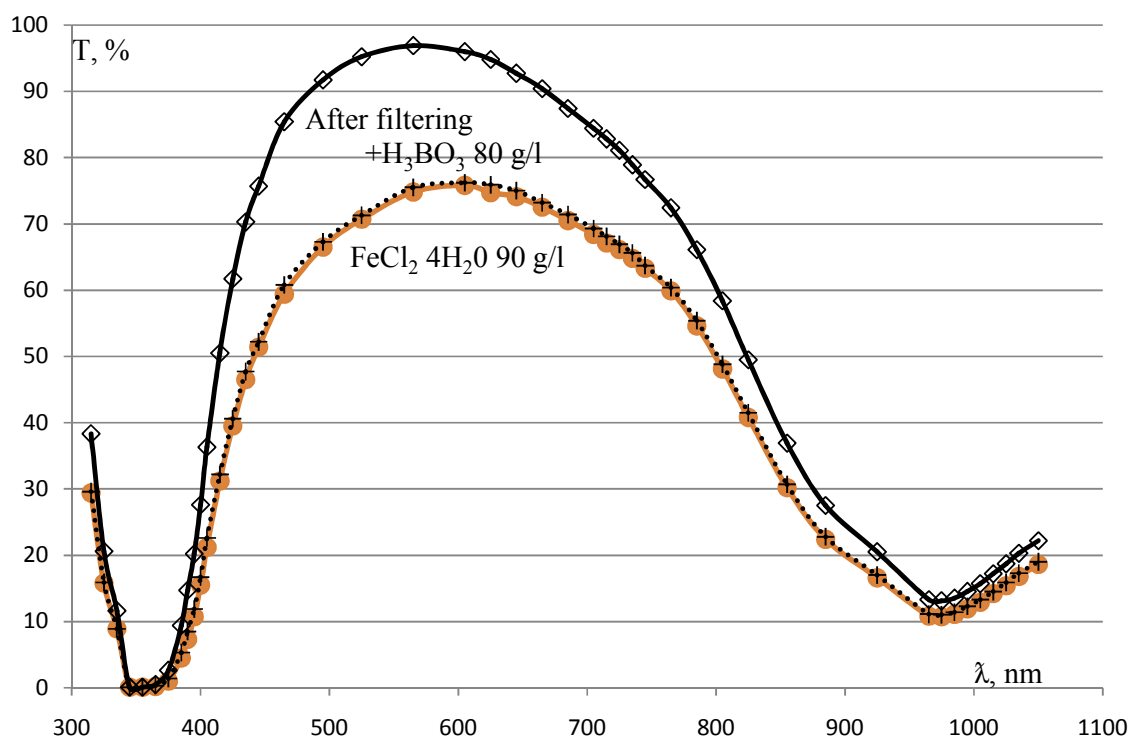


Figure 2. Dependence of optical transmittance T at wavelengths 315-1050 nm in aqueous solution with concentrations of $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$ 90 g/l, the addition of boric acid 80 g/l, and filtering solution.

Clears the filtering solution from precipitates, improved light transmission without changing spectral characteristics, i.e. clears from the precipitates that give light absorption without changing spectrum. Sludge



appeared with the additive of boric acid and when filtered precipitates as yellow with white foam complexion. Interaction of sludge of hydroxide iron (III) with a solution of hydrochloric acid leads to characteristic for $\text{Fe}(\text{OH})_3$ the dissolution of sediment and the formation of a yellow solution of iron (III) chloride. Filter cleans the electrolyte from insoluble hydroxide trivalent iron.

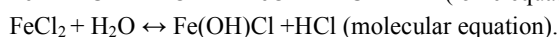
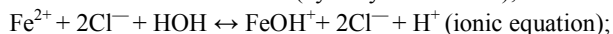
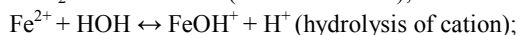
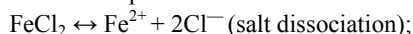
3. Hydrolysis of ferric chloride

The interaction of ions of salts with water leads to the formation of a weak electrolyte resulting from the hydrolysis of salt [4]. Salt ions bind with water-generated ions, either hydrogen ions H^+ or OH^- . According to the Le Chatelier principle, a decrease in the concentration of the ions should lead to further disintegration of water molecules into ions. If an ion participates in the formation of ions from the electrolyte, then another ion accumulates in the solution, and the pH changes.

To strengthen salt hydrolysis, the solution is diluted and heated. According to the law of mass action, supplying one of the products of the hydrolysis to the solution decreases the hydrolysis of the salt. When a product of hydrolysis is removed, the hydrolysis of the salt is enhanced.

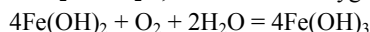
The dissolution of ferric chloride in water [5] in sludge can precipitate iron(II) hydroxide ($\text{Fe}(\text{OH})_2$). Pure iron(II) hydroxide produces a white insoluble substance in water. The hydrolysis of ferric(II) chloride occurs in acidic water in the presence of cations. The hydrolysis equation is as follows:

The first step is as follows:

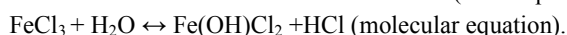
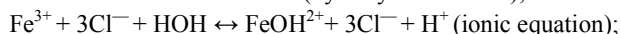
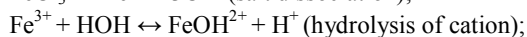
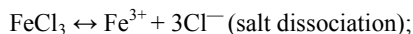


Is formed principal salt FeOHCl .

Iron(II) hydroxide has a grayish-green color, does not dissolve in water, and quickly darkens due to oxidation. Like all other compounds of divalent iron, iron(II) hydroxide has restorative properties, and in the presence of O_2 and H_2O , the dissolved oxygen is dissolved to iron(III) hydroxide over time when standing in air:



The hydrolysis of ferric(III) chloride occurs in acidic water in the presence of cations. The first step is as follows:

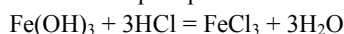


Is formed principal salt FeOHCl_2 .

Iron(III) hydroxide, with the formula $\text{Fe}_2\text{O}_3 \cdot n\text{H}_2\text{O}$, has a reddish-brown color and is not soluble in water.

Admixture of trivalent iron indicated in the passport on chemical bivalent iron chloride and determines chemical storage time.

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



Validating the action of hydrochloric acid on residues from our solution showed the presence of such an effect.

After filtering solution there is no $\text{Fe}(\text{OH})_3$ and addition of hydrochloric acid lowers the pH. The electrolyte is illumined and stabilizes.

Nickel chloride hydrolyzed completely and forms two charge ions.

Analyzing the solubility of $\text{Fe}(\text{OH})_2$ and $\text{Ni}(\text{OH})_2$ and the dissociation constants of FeOH^+ and NiOH^+ in [5] yields the following conclusion. As reviewed the mechanisms imply the existence of nickel deposition, inhibiting substances containing the less noble ion Fe in order to reduce the deposition rate of more noble Ni for



NiFe alloy deposition. The fivefold increase in pH in the cathode layer was directly measured [5]; the NiFe alloy deposition depending on the current density increased from 1 to 5 mA/cm².

In FeCl₂ solutions with pH = 3-4, the braking mechanism of the deposition of nickel iron hydroxides [3] cannot yield anomalous deposition.

Fe sulfate has an observed [6] absorption peak at 480 nm, whereas Ni can be divided into several peaks at 393, 657 and 720 nm. The changing spectrometer data over time showed that the peak of Fe rapidly increases and dominates the spectrum within 25 days. The growth of the Fe peak is interpreted as the result of the oxidation of Fe²⁺ ions to Fe³⁺. Over 20% of the originally existing Fe²⁺ ions disappeared due to deposition or oxidation, and the ion content of Fe³⁺ increased. After electrodeposition the Fe peak at 480 nm was reduced, i.e., some of the Fe³⁺ ions disappeared from the electrolyte. The absorption spectrum indicated that the instability of the electrolyte was associated with a change in the valence of iron from Fe²⁺ to Fe³⁺ and deposition mainly consisted of deposited Fe³⁺.

The effects of ferric iron in electrolyte conclude. There is reason to assert that abnormal codeposition is defined by the presence of bivalent iron and iron hydroxides with iron valence changes of two to three in the electrolyte. The charge of trivalent iron ions leads to their preferential deposition compared to nickel. Removing ferric iron (Fe³⁺) from chlorine electrolyte solves the problem of anomalous codeposition and obtains reproducible films with compositions specified by electrolyte-congruent electrochemical deposition at 70°C.

4. Effect of temperature of the electrolyte on the composition of the permalloy films

It is shown Figure 1 that the electrochemical permalloy composition is strongly dependent on the temperature of the electrolyte without filtering.

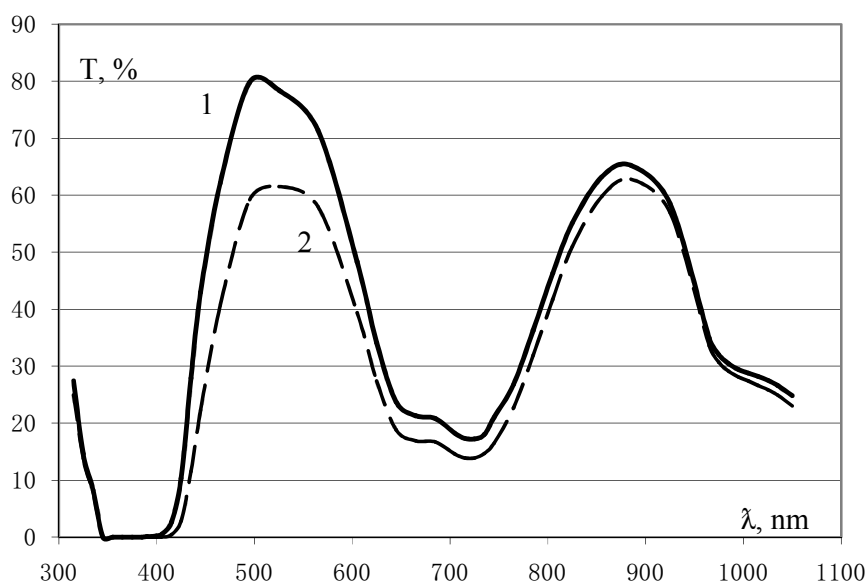


Figure 3. The dependence of the optical transmittance T at wavelengths 315-1050 nm electrolyte concentrations FeCl₂ 4H₂O-11 g/l; NiCl₂ 6H₂O-56 g/l, H₃BO₃ -25 g/l, C₇H₅NaNO₃S-1.5 g/l, HCl-2.7 ml/l 1- initial and 2- after 12 process and aging 10 months.

Heating of chloride electrolyte at a temperature of 70°C eliminates abnormal NiFe alloy components codeposition due to ionization once charged ions (Fe²⁺Cl)⁺ and Fe²⁺ ions. Identified a new phenomenon full ionization of ferric chloride in the electrolyte. Chloride electrolyte hydrochloric acid pH correction ensures the normal electrochemical deposition of permalloy. Chloride electrolyte composition with concentrations of C_{Ni}/C_{Fe} = 4.26 provides obtaining of permalloy films Ni₈₁Fe₁₉ in electrochemical deposition.



This work obtained a very interesting composition dependence on the temperature for the electrolyte with filtering for the following composition, g/l: FeCl₂ 4H₂O-11; NiCl₂ 6H₂O-56; H₃BO₃-25; C₇H₅NaNO₃S-1.5; and HCl-2.7 ml/l. Optical transmittance of the electrolyte is represented in Figure 3.

Holding 12 processes at the electrolyte temperature of 60°C and 70°C strongly modifies the iron content. Figure 4 shows that the sequence of processes and current does not affect the composition, which is determined only by the temperature. The permalloy films deposited at the electrolyte temperature of 60°C contain 39.4% iron. The permalloy films deposited at the electrolyte temperature of 70°C contain 19.2% iron. Based on the results of the X-ray spectral analysis for the deposition of the same electrolyte, all the values of current showed twice higher iron content at 60°C.

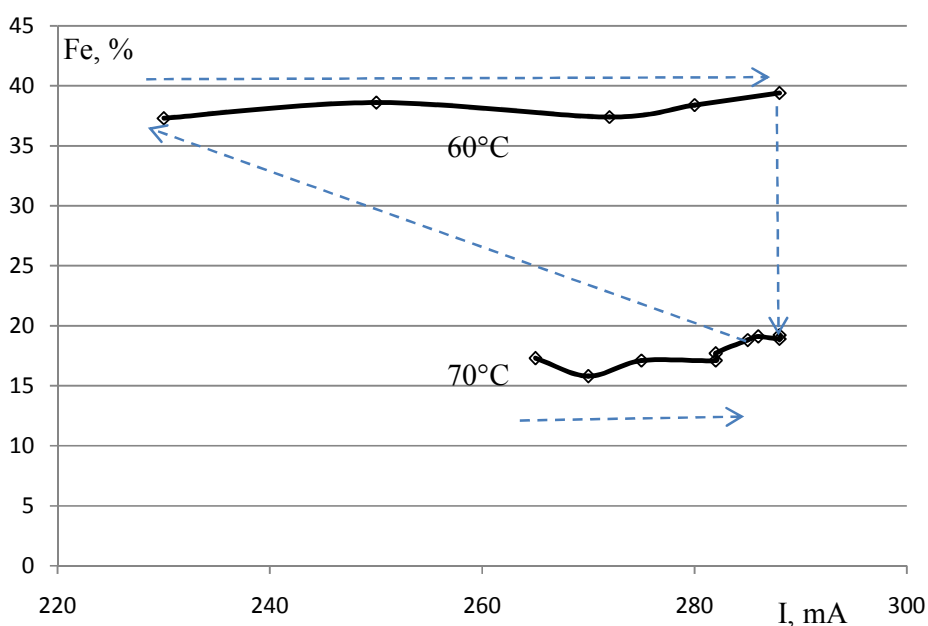


Figure 4. Iron content dependence in permalloy films on the current in chloride electrolyte with $C_{Ni}/C_{Fe} = 4.26$ at temperatures of 60°C and 70°C, pH = 1.9.

The film deposition speed of different compositions depending on the current is shows a slight dependence on the current magnitude, after growing simultaneously for samples obtained at the temperatures of 60 and 70°C. The electrochemical equivalent of permalloy deposition for the electrolyte has a single value for the films based on the growth rate. However, doubling of the iron content in the film at a lower temperature may be associated with the charge value of the iron ion. Voltage drop between electrolytic cathode and anode at a temperature of 70°C compared to 60°C decreases, which indicates that the greater the conductivity of the electrolyte through the charge of ions.

This concept is confirmed by the pH dependence on the FeCl₂ 4H₂O from concentration. pH is decreased by dissolving FeCl₂, which means that as a result of the dissociation of salts in water, ions and hydroxyls are generated. A complete dissociation of the formed Fe(OH)₂ and 2HCl is obtained without altering the pH. Upon single ionization of FeCl₂ as (Fe²⁺Cl)⁺ for the second chlorine ion, in collaboration with the water, Cl⁻ + H₂O forms HCl and excess OH⁻. According to the Le Chatelier principle, reduction of the concentration of ions should lead to further disassociation of water molecules into ions. If a molecule participates in the formation of ions from the electrolyte, then another ion accumulates in the solution and the pH changes.



Electrochemical equivalents for metals with double valence give a natural change in the deposition rate relative to their valence. When the current is determined by the electrochemical charge ions, iron, nickel and cobalt are precipitated 1.5 times slower than in the bivalent case.

With this ratio of electrolyte components, the concept of anomalous deposition changes from suppression of the deposition of nickel iron hydroxide to doubling of the deposition rate for iron ions (FeCl^+) compared to the doubly charged Fe^{2+} . At the temperature of 70°C , a concentration ratio change occurs at the expense of full ionization of singly charged ions (FeCl^+), and the deposition of the electrolyte with doubly charged Fe^{2+} is observed, corresponding to the normal, conventional notion of the deposition of nickel-iron alloy.

A new phenomenon of full ionization of ferric chloride in the electrolyte is observed at the temperature of 70°C .

The reviewed explanation of the anomalous deposition of nickel-iron alloy based on the hydrolysis of ferric chloride not only explains this phenomenon, which was discovered 60 years ago, but also provides the solution to the problem of obtaining films under the common reaction of normal structure deposition. At the temperature of 70°C , the composition of the chloride electrolyte with a concentration ratio of $C_{\text{Ni}}/C_{\text{Fe}} = 4.26$ led to the obtainment of electrochemical permalloy $\text{Ni}_{81}\text{Fe}_{19}$ films. Numerous previous studies with a wide range of electrolyte composition had different relative contents of ions (FeCl^+) and Fe^{2+} that led to a large variation in the entire film.

5. Ionic balance in the hydrolysis of FeCl_2

[7] Discusses the ionic equilibrium in the FeCl_2 electrolyte through thermodynamic calculations of equilibrium constants, taking into account the adverse reactions, as well as mass and charge balance equations.

The solution of FeCl_2 4 mol/l at room temperature mostly contains singly charged ions ($\text{Fe}^{2+}\text{Cl}^+$), a smaller content of Fe^{2+} ions, a small amount of neutral molecules and a very slight amount of hydroxide ions ($\text{Fe}^{2+}\text{OH}^+$). Addition of hydrochloric acid increases the content of the singly charged ions ($\text{Fe}^{2+}\text{Cl}^+$) and FeCl_2^0 neutral molecules and reduces the amount of doubly charged Fe^{2+} ions.

When used for the deposition of permalloy, the FeCl_2 concentration on the order of 0.1 mol/l solution contains equal concentrations of singly charged ions ($\text{Fe}^{2+}\text{Cl}^+$) and fewer Fe^{2+} ions, with the difference in the concentration of these ions increasing upon the addition of hydrochloric acid. The contents of the neutral molecules FeCl_2^0 and hydroxide ions ($\text{Fe}^{2+}\text{OH}^+$) are considerably smaller than those of the ($\text{Fe}^{2+}\text{Cl}^+$) and Fe^{2+} ions. For these calculations, the hydroxide and ($\text{Fe}^{2+}\text{OH}^+$) deposition should not have a significant impact.

When the electrolyte is heated, increasing ionization is observed because the number of intermediate ions ($\text{Fe}^{2+}\text{Cl}^+$) can be smaller than the number of Fe^{2+} ions. The content of neutral molecules becomes very small.

6. Electrochemical deposition of NiFe alloy films of chloride electrolyte containing low concentration of salts

Performing the NiFe alloy electrochemical deposition using iron ions with changing electrolyte temperature [1, 3] allows for a new approach to the fabrication of the electrolyte. The charge of the ions in the electrolyte may depend not only on the temperature but also on the iron concentration. It is well-known that reduction of the concentration leads to strengthening of the electrolytic dissociation of salts [8].

For the content of iron in permalloy films obtained by diluting chloride electrolyte, it should be found that films with permalloy $\text{Fe}_{19}\text{Ni}_{81}$ are obtained by the use of simple electrochemical chloride electrolyte with $C_{\text{Ni}}/C_{\text{Fe}} = 4.26$ and concentrations of Fe^{2+} -0.004 mol/l. Consequently, at these concentrations of nickel and iron in molten chloride, the nickel and iron ions with charges Ni^{2+} , and Fe^{2+} have the same amounts in the cathode discharge.

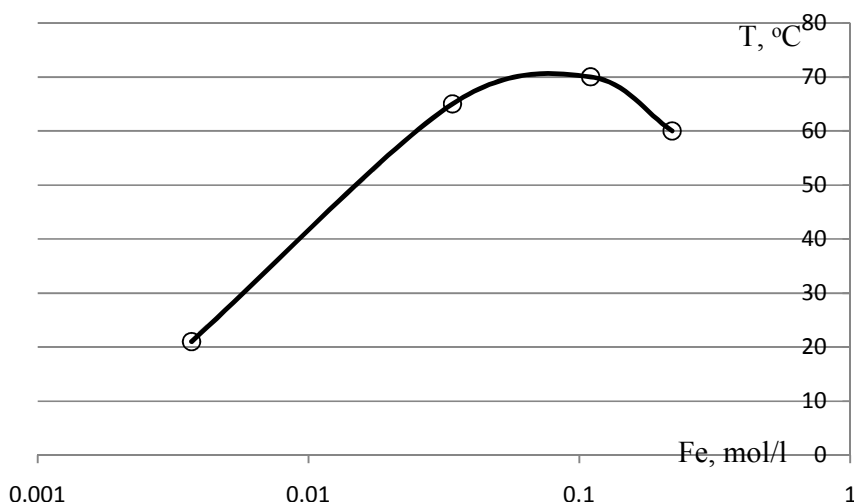


Figure 5. Electrochemical deposition of NiFe alloy films containing 19% iron from electrolytes with concentrations of Fe of 0.0037 mol/l at a temperature point, 0.034 mol/l at a temperature of 65°C, 0.11 mol/l at a temperature of 70°C, and 0.22 mol/l at a temperature of 60°C.

Obtaining weak solutions of permalloy films not only increases the induced electrolytic salts and ion content but also leads to doubly charged ions instead of singly charged ions.

Electrochemical deposition of permalloy films from chloride electrolyte with $C_{Ni}/C_{Fe} = 4.26$ and concentrations of Fe^{+2} -0.004 mol/l at room temperature allows one to obtain films with congruent electrolyte composition.

The use of dilute chloride electrolyte for NiFe deposition with a ratio of $C_{Ni}/C_{Fe} = 4.26$ confirms the principle that the charge of iron ions influences the composition of permalloy films.

The effect of the temperature of the electrolyte on the ability to obtain permalloy films containing 19% iron is presented for selected concentrations of iron in solution in Figure 5. At low concentrations of nickel and iron chlorides, the room temperature deposition rate is significantly less than that at higher concentrations and 70°C.

7. Model discharge of iron and nickel ions on the cathode

Experimental results obtaining permalloy films specified composition [9, 10] find scientific justification as an exception at the cathode deposition from chloride electrolyte once charge ions of bivalent and trivalent iron.

Have not been considered in the literature component ratio of job in the electrolyte is equal to a specified composition component in the film. Has not been the stabilization of the electrolyte. None of the researchers linked the anomalous deposition of iron ions with charge. Therefore, the deposition of the permalloy films was not repeatable process.

Electrochemical deposition of NiFe depends on the charge of ions of Fe in the electrolyte. A charge ion depends not only on temperature but also on the concentration of Fe. Decreased concentration is well known to exacerbate electrolytic salt dissociation. To ascertain the nature of the events taking place in chloride electrolyte, we studied and deposition of thin films at room temperature.

At the probability current density, the discharge of ions depends on their concentration, and contributions to electric current from their charge. When you change the current density occur a change the contribution of different ions and the change in the composition of the sediment. Discharge of ions at the cathode determines current cathode. Content of ions in the electrolyte sets the composition of sediment. One



charge ion gives you more sediment than two charge ion. Changing the current changes the ratio of the ions in the sludge. This determines the dependence of the whole sediment from current.

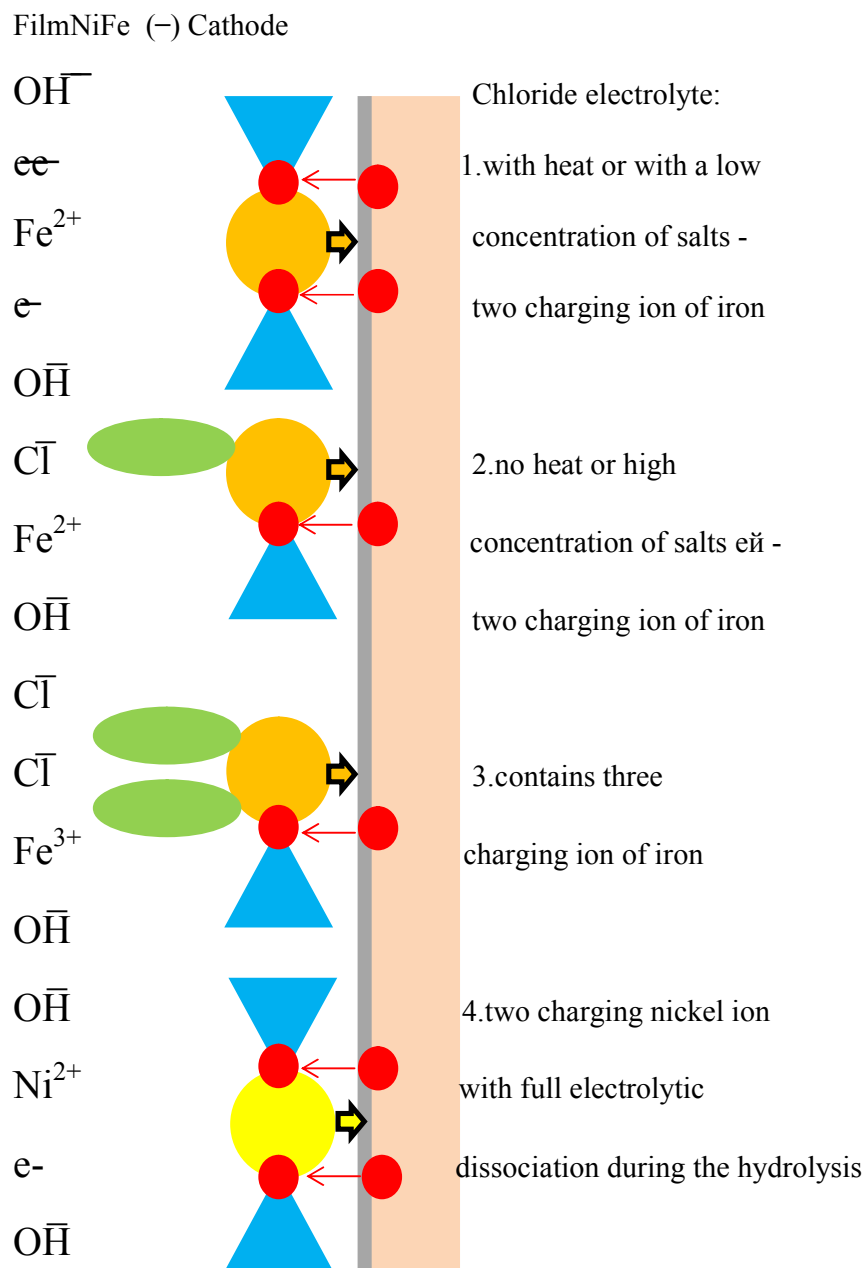


Figure 6. The discharge of ions of iron and nickel on the cathode at electrochemical deposition of permalloy.

In the same spirit nickel and iron ions occurs congruent permalloy deposition. The composition of the film does not depend on current.



Conditional discharge mechanism of nickel and iron ions at the cathode is shown in Figure 6, with an indication of the charge of ions and the characteristics of chloride electrolyte with heat, cleaning or with low salt concentrations of ferric chloride.

8. Composition of permalloy films deposition from optimized chloride electrolyte

Electrochemical deposition of chloride electrolyte and content of nickel and iron atoms in relation to 4.26 corresponding alloy $Ni_{81}Fe_{19}$ at a temperature of $70^{\circ}C$ gave the composition dependence of permalloy films in the current range 270-410 mA, shown in Figure 7. Spectrogram of the electrolyte is presented in Figure 3.

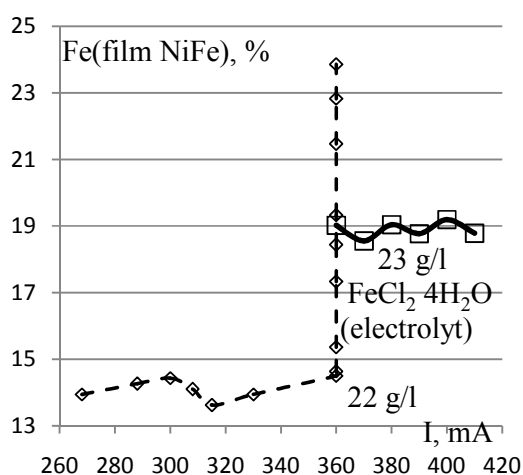


Figure 7. Electrochemical deposition of NiFe from chloride electrolyte.

Changing the current process does not lead to a noticeable change in the content of iron and nickel film. Current value is selected when changing the content of iron and nickel due to additives in electrolyte $FeCl_2 \cdot 4H_2O$ leads to modify the content of the film.

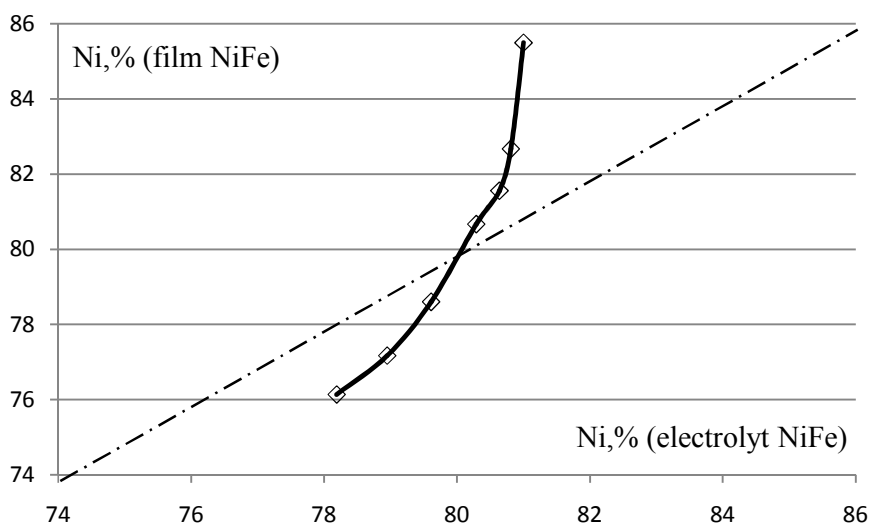


Figure 8. The dependence the composition of the NiFe film from the electrolyte composition.



Nickel in NiFe film is higher than in the electrolyte, as seen in Figure 8. An equal deposition of nickel towards electrolyte indicated a dashed stroke. Therefore, nickel is precipitated better than iron and anomalous codeposition is not observed.

Increased concentrations of ferric chloride hydrate at 4.5% to compensate for accelerated deposition of nickel and get corresponding alloy permalloy film composition $\text{Ni}_{81}\text{Fe}_{19}$.

Conclusion

The researchers during the 60 years NiFe alloy plating and receive preferential deposition of iron in relation to nickel in all formulations of the electrolyte. Applied electrolytes are characterized by a wide range of molar relationship of nickel and iron. None of the researchers linked the anomalous character of electrodeposition of iron ions with the main feature-the existence of variable valence iron one and two values in the charge of ions during the hydrolysis of iron salts.

Selection of chloride electrolyte with attitude $C_{\text{Ni}}/C_{\text{Fe}} = 4.26$, development of technology of preparation of electrolyte and determination of optimal electrolyte temperature revealed the mechanism of abnormal deposition because of incomplete ionization of atoms of iron and ensure that in the film the relationship $C_{\text{Ni}}/C_{\text{Fe}} = 4.26$ at room temperature, without mechanical stress, with uniform structure and high magnetic parameters without high temperature annealing. The congruent deposition when posting the charge of ions in the electrolyte allows getting.

Detailed results of the study described in the book [11].

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