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*Встановлено залежності складу сплаву Ni–P від умов електросинтезу. Показано, що утворення фосфору відбувається в результаті електровідновлення і диспропорціонування натрій гіпофосфіту за участю іонів водню. Збільшення швидкості цих реакцій призводить до збільшення вмісту фосфору в сплаві. На підставі цього встановлено, що підвищення температури і кислотності електроліту є сприятливими факторами для збільшення вмісту фосфору в сплаві Ni–P*

*Ключові слова: електросинтез, сплав Ni–P, метилсульфонатний електроліт, буферні властивості, утворення фосфору*

*Установлены зависимости состава сплава Ni–P от условий электросинтеза. Показано, что образование фосфора происходит в результате электровосстановления и диспропорционирования гипофосфита натрия при участии ионов водорода. Увеличение скорости этих реакций приводит к увеличению содержания фосфора в сплаве. На основании этого установлено, что повышение температуры и кислотности электролита являются благоприятными факторами для увеличения содержания фосфора в сплаве Ni–P*

*Ключевые слова: электросинтез, сплав Ni–P, метансульфонатный электролит, буферные свойства, образование фосфора*

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## EXAMINING THE EFFECT OF ELECTROSYNTHESIS CONDITIONS ON THE Ni-P ALLOY COMPOSITION

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### 1. Introduction

Alloys are widely used in different sectors of modern industry. Improved physical and mechanical characteristics provide durability and reliability during operation of alloys. Certain alloys possess catalytic and magnetic properties. Properties of alloys are determined by the nature of components and qualitative composition. Alloys of the iron group are characterized by the most varied palette of useful

exploitation properties. At various combinations of components such alloys possess high hardness, heat resistance, magnetic, catalytic, anticorrosion properties.

One of the most regulated methods of obtaining alloys with predicted properties is the electrosynthesis. Depending on the requirements to qualitative and quantitative composition of the alloy, the electrolysis is performed from specially selected electrolytes. The content of components in the alloy depends also on the parameters of conducting

the electrosynthesis. The ratio of alloy components can be significantly affected by a change in the electrolysis temperature and current density.

The electrolytic Ni–P alloy, related to the system metal-metalloid, possesses several valuable properties. This alloy is characterized by high hardness, electro catalytic activity and special magnetic properties. Phosphorus content determines the crystallographic state and properties of the Ni–P alloy. In order to form the alloy with specified composition, an understanding is required of the ways of influence on the kinetics of reactions of nickel and phosphorus extraction during alloy-formation. Given this, it is a relevant task is to establish the influence of electrosynthesis conditions on the Ni–P alloy composition.

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## 2. Literature review and problem statement

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The electrolytic Ni–P alloy is widely used to prevent corrosion of materials [1, 2]. Anti-corrosion properties of the Ni–P alloy are predetermined by its structure and depend on the content of phosphorus in the alloy. At the same conditions, a stationary potential of the alloy is more positive than the potential of nickel. Accordingly, corrosion current of Ni–P is less. Nanocrystalline precipitations with a content of phosphorus in the alloy of 5–9 % (by weight) are more corrosion resistant than the microcrystalline alloys containing a smaller amount of phosphorus [3]. Wear resistance of such alloys, compared with the amorphous alloys containing more than 9 % (by weight) of P, is better. Wear resistance of the Ni–P alloy is predetermined by high microhardness. The alloy hardness rises significantly after thermal treatment resulting in the formation of the Ni<sub>3</sub>P phase [4]. Authors of [5] found that the alloy with a phosphorus content exceeding 7.5 % (by weight) contains the Ni<sub>12</sub>P<sub>5</sub> and Ni<sub>2</sub>P phases. The formation of Ni<sub>12</sub>P<sub>5</sub> is explained by the specific conditions of electrosynthesis under conditions of the pulse electrolysis. Article [6] investigated the effect of sodium dodecyl sulfate on the morphology and roughness of the Ni–P alloy. It was established that precipitations, synthesized in the presence of a surface-active substance, possess lower roughness.

The Ni–P alloy acts as an electrocatalyst in the synthesis of hydrogen. The catalytic activity of Ni–P depends on the content of phosphorus and phase composition of the alloy [7]. A change in the temperature of electrosynthesis of the Ni–P alloy affects its catalytic activity. It was established in [8] that the largest catalytic activity is demonstrated by the alloy containing about 6 % (by weight) of phosphorus. In order to develop the surface of catalytically-active Ni–P, it was proposed to apply coprecipitation of the alloy with titanium dioxide [9].

Authors of [10] modified the magnetic properties of Ni–P by giving a specific geometric shape to the alloy. The alloy was synthesized in the form of nanotubes and nanowires. It was established that nanotubes and nanowires possess a seeming magnetic anisotropy caused by the anisotropy of the shape. It is shown that the use of Ni–P nanotubes as a soft magnetic material seems to be more promising.

Thus, depending on the composition and conditions of the electrosynthesis, an alloy for various purposes is received. The Ni–P alloy composition, in turn, depends on the type of the employed electrolyte. As a rule, electrosynthesis of the Ni–P alloy is performed from the sulfate [11, 12] or sulphamate [2, 13] electrolytes. At the same time, it was

proposed to apply in the modern technologies of the alloy electrosynthesis a new electrolyte based on methanesulfonic acid [14, 15]. Weak resistance and high solubility of the corresponding acidocomplexes of metals in the iron group [16] expands the range of concentrations and current densities, which provides for the electrosynthesis of alloys. Given this, it may prove rather useful to study dependences of the Ni–P alloy electrosynthesis from the methanesulfonate electrolyte in comparison with the sulphate electrolyte.

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## 3. The aim and objectives of the study

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The goal of present work is to establish the influence of electrosynthesis conditions on the Ni–P alloy composition, precipitated from the methanesulfonate electrolyte. This will make it possible to design a new technology for the targeted synthesis of the alloy with specified composition with predicted physical-chemical properties.

To accomplish the set goal, the following tasks have been set:

- to receive dependences of the content of phosphorus in the Ni–P alloy on the concentration of sodium hypophosphite, the electrolyte pH and temperature of the electrolysis in the methanesulfonate sulphate electrolytes;
- to identify ways of phosphorus formation in the Ni–P alloy electrosynthesis and to establish techniques to influence the speed of reactions that exert a decisive impact on the composition of the alloy.

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## 4. Materials and methods for examining the co-precipitation of nickel and phosphorus and the Ni–P alloy composition

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We conducted electrosynthesis of the Ni–P alloy from the methanesulfonate: 1.00 M Ni(CH<sub>3</sub>SO<sub>3</sub>)<sub>2</sub>, 0.30 M NaCl, 0.70 M H<sub>3</sub>BO<sub>3</sub>, X M NaH<sub>2</sub>PO<sub>2</sub> and the sulfate: 1.00 M NiSO<sub>4</sub>, 0.30 M NaCl, 0.70 M H<sub>3</sub>BO<sub>3</sub>, X M NaH<sub>2</sub>PO<sub>2</sub> electrolytes at current density of 20 mA/cm<sup>2</sup>. The electrosynthesis was carried out using the DC source BVP Electronics Home Tools 30V, 6A (Ukraine).

The pH indices of the solutions were controlled using the universal ionomer EV-74 (Belarus) with the combined electrode ESK-10601/7 (Belarus). The required pH indices were maintained by introducing to electrolyte the solutions of 0.1M CH<sub>3</sub>SO<sub>3</sub>H and 0.1 M NaOH.

Phosphorus content in the alloy was determined by the differential photocolometric method. Essence of the method is that the orthophosphates form with the salts of vanadium and molybdenum yellow compounds with the following composition P<sub>2</sub>O<sub>5</sub>·V<sub>2</sub>O<sub>5</sub>·22MoO<sub>3</sub>·11H<sub>2</sub>O. Optical density of the solution was measured using the photocolometer KFK-2-UKhL 4.2 (Russia) at a wavelength of 440 nm.

We prepared a molybdenum-vanadium reagent by mixing two solutions containing 100 g/l of ammonium molybdate and 6 g/l of ammonium metavanadate.

The Ni–P alloy was dissolved in diluted nitric acid (1:1), a volume of 25 ml. The resulting solution was poured in a measuring flask of 100 ml, next we added 10 ml of the 13-% solution of ammonium persulfate and kept the solution for 10 minutes. Upon the expiration of the allotted time, we added 5 ml of the concentrated nitric acid, 20 ml of molybdenum-vanadium reagent, and brought the volume to

100 ml. In 20–30 minutes, we measured optical density of the solution. The measurements were conducted relative to a comparison solution that contained all reagents except for the examined solution. The percentage of phosphorus content in the alloy was determined by formula:

$$\omega = 100 \frac{m_p}{m}, \quad (1)$$

where  $m_p$  is the weight of phosphorus, determined by calibration chart, g;  $m$  is the weight of the alloy, g.

When constructing the calibration chart, we used standard solutions with the known content of phosphorus in the form of phosphate ion.

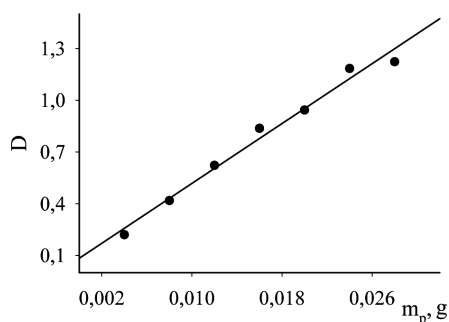


Fig. 1. Calibration chart of dependence of optical density of the solution ( $D$ ) on the phosphorus content  $m_p$

Electrochemical studies into co-precipitation of nickel and phosphorus into the alloy were conducted using the IPC-Pro potentiostat (Russia) in a thermostated four-chamber glass cell. Platinum plate served as a working electrode. As an auxiliary electrode during deposition of the Ni–P alloy, we applied nickel of the NPA-1 brand. Reference electrode – chloride silver electrode of the EVL-IMI brand (Belarus). Potentiodynamic measurements were carried out when the potential sweep rate was 2 mV/s.

### 5. Results of examining the influence of electrosynthesis conditions on the alloy Ni–P composition

Electrosynthesis of the Ni–P alloy was carried out in the presence of phosphorus donor in the electrolyte. In the present work, a source of phosphorus in the alloy is the hypophosphite-anion. An increase in the concentration of sodium hypophosphite in the electrolyte naturally results in an increase in the content of phosphorus in the alloy (Fig. 2). At concentrating the electrolyte by sodium hypophosphite, initial sharp jump gives way to a smooth change in the composition of the alloy. Depending on the type of electrolyte used, phosphorus content in the alloy, obtained under the same conditions, is different. The Ni–P alloys, received from the methanesulfonate electrolyte, has less amount of phosphorus compared with the precipitations, synthesized in the sulphate electrolyte.

A change in the electrolyte pH has a significant impact on the composition of the alloy. Fig. 3 shows that a decrease in pH contributes to increasing phosphorus content in the alloy. This trend is maintained in both the methanesulfonate and sulphate electrolyte. The above-mentioned difference in the composition of alloys received from the methanesulfonate and sulfate electrolyte is observed as well at larger

acidity of the electrolyte. The Ni–P alloy synthesized in the methanesulfonate electrolyte at pH 2 contains a quarter less phosphorus than the alloy obtained from the sulfate electrolyte.

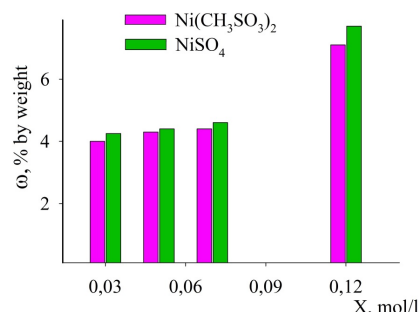


Fig. 2. Dependence of phosphorus content in the Ni–P alloy on the concentration of sodium hypophosphite in the methanesulfonate and sulphate electrolytes at pH 3

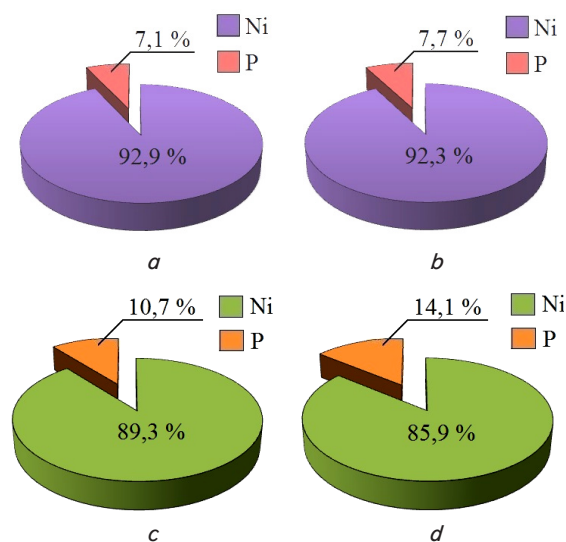


Fig. 3. Composition of the Ni–P alloy received at  $X=0.12$  from: *a* – methanesulfonate electrolyte with pH 3; *b* – sulfate electrolyte with pH 3; *c* – methanesulfonate electrolyte with pH 2; *d* – sulfate electrolyte with pH 2

Let us consider the effect of temperature on composition of the Ni–P alloys received by the electrosynthesis from the methanesulfate and sulfate electrolytes. Phosphorus content in the alloy, synthesized at a temperature of 313 K from the methanesulfonate electrolyte with 0.07 mol/l  $\text{NaH}_2\text{PO}_2$  is 3.37 % (by weight). An increase in the temperature of electrolyte to 333 K leads to an increase in the alloy phosphorus content to 4.35 % (by weight).

An increase in the electrolysis temperature has a larger impact on the change in the composition of the Ni–P alloy when using the methanesulfonate electrolyte. The difference in the compositions of alloys, precipitated at 313 K and 333 K from the sulfate electrolyte is negligible (4.1 and 4.63 % (by weight), respectively). The patterns established for the electrolytes containing 0.07 mol/l of  $\text{NaH}_2\text{PO}_2$ , are similar in the solutions, more concentrated by sodium hypophosphite. Thus, the Ni–P alloy, precipitated at 313 K from the methanesulfonate electrolyte in the presence of 0.12 mol/l of  $\text{NaH}_2\text{PO}_2$ , contains 5.2 % (by weight). At 333 K, phosphorus content increases to 7.0 % (by weight). For the precipitations, syn-

thesized in the sulphate electrolyte under these conditions, the phosphorus content values make up 6.55 % (by weight) and 7.68 % (by weight).

The alloy Ni–P composition is determined by the rates of formation of nickel and phosphorus during electrolysis. The electrodeposition process of the Ni–P alloy is characterized by voltampere dependences, shown in Fig. 4.

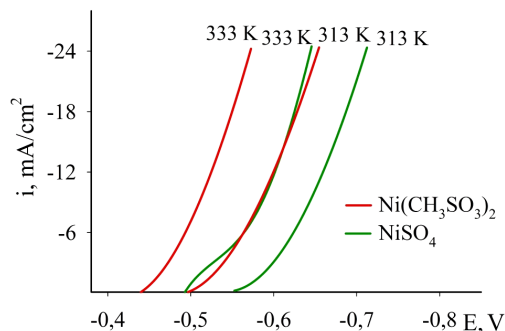


Fig. 4. Voltammograms of the Ni–P alloy deposition from the methanesulfonate and sulfate electrolytes at  $X=0.12$

Voltammograms shift towards the region of more positive potentials when the electrolysis temperature rises, both for methanesulfate and sulfate electrolytes. This indicates that a change in the temperature affects the kinetics of proceeding processes.

When conducting the electrosynthesis under galvanostatic mode, influence of the electrolysis parameters on the composition of the alloy will manifest itself mainly through a change in the rate of phosphorus formation.

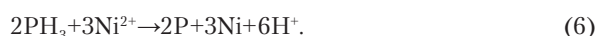
## 6. Discussion of results of examining the effect of electrosynthesis conditions on the alloy Ni–P composition

Let us consider reactions that proceed in the Ni–P alloy electrosynthesis. The electrodeposition of nickel follows the reaction:



Formation of phosphorus can take place along several pathways. Phosphorus gets into the nickel matrix either in the form of nickel phosphides or in the atomic state [17, 18]. Phosphide formation occurs as a result of redox reaction involving nickel (II) ions and phosphine [19–21]. Atomic phosphorus can be formed as a result of cathodic reduction of hypophosphite anion [22]. In the course of the Ni–P alloy electrosynthesis from the electrolyte containing a hypophosphite-anion, the disproportionation reaction may also take place [22].

Therefore, the probable reactions of phosphorus formation in the examined system take the following form:



The electrosynthesis of atomic phosphorus is possible during electroreduction of hypophosphite-anion by reaction (3) or in the course of reactions (5) and (6). In order to obtain the same amount of phosphorus, four times less amount of electricity is required in the first case. As shown (Fig. 2), phosphorus content in the examined alloys reaches 7 % (by weight). Comparison of limiting current magnitudes of the electroreduction of hypophosphite anion (Fig. 5) with the total density of electrosynthesis current of the Ni–P alloy testifies in favor of the reaction (3).

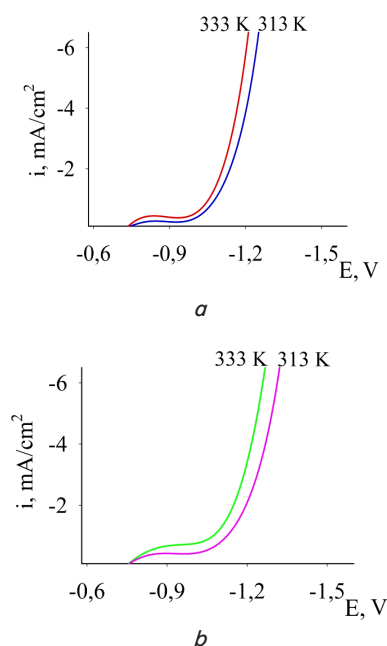


Fig. 5. Cathode voltammograms, obtained in electrolytes without the  $\text{Ni}^{2+}$  ions, at  $X=0.12$  and pH 5: *a* – methanesulfonate electrolyte; *b* – sulfate electrolyte

An increase in temperature contributes to an increase in the rate of hypophosphite anions electroreduction (Fig. 5). This leads to increased phosphorus content in the Ni–P alloy. Limiting current density of the electroreduction of hypophosphite-anions in the sulphate electrolyte exceeds that in the methanesulfonate electrolyte. This agrees well with the fact that phosphorus content in the alloy, received from the sulfate electrolyte, is larger.

It should be noted that the alloy Ni–P electrosynthesis takes place in the region of potentials, which does not correspond to the electroreduction of hypophosphite-anions. Obviously, in this case, there occurs a chemical disproportionation reaction of hypophosphite-anions by reaction (4). Thus, phosphorus is formed by electrochemical and chemical way. Because the rate of reactions (3) and (4) depends on the concentration of hydrogen ions, an electrolyte pH decrease should lead to an increase in the rate of phosphorus formation. Indeed, dependences of the Ni–P alloy composition on the electrolyte pH, shown in Fig. 3, do confirm this. An increase in temperature contributes to the reduction of diffusion difficulties during hydrogen ions transport to the near-electrode layer. In accordance with the electrochemical (3) and (4) chemical reactions, it also provides for an increase in the rate of phosphorus formation.

The increased phosphorus content in the alloys obtained from the sulfate electrolyte is due to the elevated acidity of the near-electrode layer. Sulfate electrolyte possesses higher

buffer properties than those in the methanesulfonate electrolyte [11]. This is due to the presence in the electrolyte of hydrosulfate anions, which act as the donors of hydrogen ions. As a result, the near-electrode layer pH in the sulphate electrolyte is lower than that in the methanesulfonate electrolyte. In accordance with reactions (3) and (4), the rate of phosphorus formation in the sulphate electrolyte is higher, which is selected by the composition of the alloy.

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## 7. Conclusions

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1. We received dependences of the Ni–P alloy composition on the concentration of sodium hypophosphite in the methanesulfonate and sulphate electrolytes. An increase in the concentration of the donor of phosphorus atoms in the electrolyte contributes to increasing phosphorus content in the alloy. An increase in the acidity and temperature of the electrolyte leads to the formation of alloys with a larger content of phosphorus. At the Ni–P alloy electro-synthesis from the methanesulfonate electrolyte, phosphorus content in the alloy is less than when using the sulfate electrolyte.

2. It was established that phosphorus formation during the Ni–P alloy electro-synthesis takes place in two ways. Electrochemical reaction matches direct formation of phosphorus during electroreduction of hypophosphite-anion. The chemical synthesis of phosphorus occurs as a result of the disproportionation reaction of hypophosphite-anion on the cathode surface. Both reaction of phosphorus formation proceed with the consumption of hydrogen ions. Therefore, a change in the parameters of electrolysis, leading to an increase in acidity of the electrode layer, increases the rates of phosphorus formation reactions. This, in turn, affects the composition of the Ni–P alloy. That is why an increase in temperature and acidity of the electrolyte leads to increased phosphorus content in the alloy. It is shown that under identical conditions phosphorus content in the Ni–P alloy received in the methanesulfonate electrolyte is less than in the sulphate electrolyte. This is linked to the lower buffer properties of the methanesulfonate electrolyte. Established dependences are required for designing the new technologies of electro-synthesis of the Ni–P alloy with specified composition from the methanesulfonate electrolyte.

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