

# INVESTIGATION OF ELECTROCHEMICAL PROCESSES

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## OBTAINING of POWDERS of ELECTROLYTIC NICKEL- and RHENIUM-CONTAINING ALLOYS

**Abstract:** This paper contains results of studies on production of electrolytic Ni-Re-containing alloys. These alloys have more pronounced valuable technical characteristics than analogous casting alloys and can be used in powder metallurgy. The effect of some main electrolysis parameters (electrolyte composition, cathodic current density, temperature) on the chemical, phase composition, structure and yield by current efficiency (YCE) of Ni-Re-containing alloys deposited on a titanium cathode was determined. The alloys were analyzed using X-ray fluorescence, X-ray phase, chemical, electron-microscopic, and petrographic methods. Deposition of powder alloys from ammonium-sulfate electrolytes (average composition, g/dm<sup>3</sup>: 12-30 NiSO<sub>4</sub>, 1.0-7.2 NH<sub>4</sub>ReO<sub>4</sub>, 20-80 (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>, 6.6-15.0 H<sub>2</sub>SO<sub>4</sub>) showed that with increase of NiSO<sub>4</sub> concentration YCE increases from 56 up to 90 %. The average content in the alloys, wt. %: nickel – 47.5-54.9 and rhenium – 37.6-47.5. Increase in the concentration of NH<sub>4</sub>ReO<sub>4</sub> was associated with an increase in YCE from 45 up to 84%. The alloys contain in average, wt. %: 49.0–71.9 of nickel and 22.7–46.5 of rhenium. Increase in the current density (150–300 A/m<sup>2</sup>) leads to an increase in the YCE from 60 up to 95 %. The concentration of nickel in the alloys increases from 48 up to 63 (wt.%), whereas the concentration of rhenium decreases from 49 to 36 (wt.%). When the temperature of the electrolyte increased (30-60 °C), the YCE increased from 74 up to 95 %; the concentration of nickel in the alloys increased from 56 up to 61 (wt.%), and the concentration of rhenium varied in the range of 35–38 wt.%. When the composition of the electrolyte was modified (g/dm<sup>3</sup>: 2 NH<sub>4</sub>ReO<sub>4</sub>, 80 (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>) the concentration of nickel in the resulted alloys increases from 76 up to 93 (wt.%), the concentration of rhenium decreases from 19 to 1.7 (wt.%), and the yield by current efficiency of the alloys was from 51.7 to 85.4 % with increase in the temperature. When the temperature increased the alloy powders become more friable and are easily disintegrated. According to X-ray fluorescence analysis data, the alloy base is represented by solid Re solutions in Ni and Ni solutions in Re with particles of 8.2-16.6 nm in size irrespective of the electrolysis parameters.

**Key words:** powders, electrolytic alloys, nickel, rhenium, electrodeposition, electrolyte composition, current density, temperature, current efficiency.

**Introduction.** Recently, all over the world there is an increased interest to nickel-based superalloys which are used in aerospace engineering [1-4]. The countries with developed industries work actively for development of new construction materials on the basis of heat-proof nickel alloys containing additives of high-melting-point metals (rhenium, wolframium, etc.) using powder metallurgy methods. Many details made of powder alloys are characterized by better quality and a lower cost as compared to those made of traditional. Therefore, although the powder alloy production volume is low and makes up just 0.1% of the total metal production volume, they are of great importance in the national economy, and the scope of application thereof is extensive. Along with the use of powder alloys as raw materials for production of various components by powder metallurgical methods, powder alloys are used as a target material for application of high-melting-point metal coating by, for example, the ion-plasma method.

All known methods used for producing nickel-based superalloys are, generally, thermal methods and are used at high temperatures. However, there are works on electrical deposition of nickel-based alloys from aqueous solutions of electrolytes.

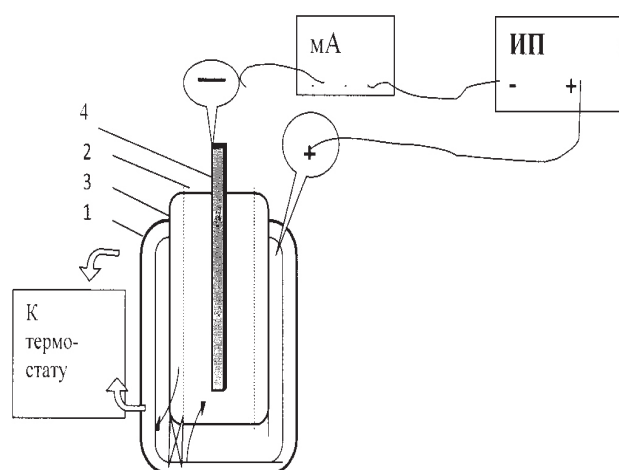
It is known that electrolytic alloys have more distinct valuable technical characteristics as compared to analogous casting alloys [5-6]. The works on electrolytic deposition of nickel alloys with rhenium and other high-melting-point metals from aqueous solutions are conducted in Russia, Kazakhstan, Japan, China, USA, Israel, and Poland [5-16].

The electrochemical technique allows obtaining homogenous alloys at low temperatures, which is not always possible to achieve using high-temperature methods. Powder metallurgy provides for specific requirements to the powder form and sizes. A targeted adjustment of electrolysis parameters allows producing powder nickel-rhenium-containing alloys of the required form and size.

Development of the machine engineering and electronic industries in the Republic of Kazakhstan will require creation of corrosion-resistant and strong details for various devices and mechanisms, contacts in microelectronic circuits, etc. Therefore, development of a new technology for manufacture of powder nickel-rhenium-containing alloys from aqueous solutions by electrolytic method is of considerable scientific and practical interest.

This work is intended to determine the influence of main electrolysis parameters on the chemical, phase composition, structure and current efficiency of the deposited powder Ni-Re-containing alloys.

**Experimental Part.** Powder electrolytic Ni-Re-containing alloys were deposited on a titanium cathode in a thermostated electrochemical cell with a platinum anode which is schematically presented in Figure 1.



1 – water-jacket; 2 – membrane; 3 – anode;  
4 – cathode, ИП – power supply

Figure 1 – Scheme of a membrane thermostated electrochemical cell

The anode in the thermostated electrochemical cell was made of platinum in the form of cylinder with a cathode made of a titanium rod placed in the centre. In order to exclude mixing the cathode and anode reactions, the interelectrode spaces in the cell were separated by a MK-40 cationite membrane. The surface of the titanium cathode was processed mechanically and cleaned by fine sandpaper. Upon completion of the electrolysis the cathode with the deposit was washed in water and dried in air, weighted and the deposit was analyzed by X-ray fluorescence, X-ray phase, chemical, electron-microscopic, and petrographic methods. Nickel

and rhenium in the deposit were determined by atomic emission spectroscopy on Optima 2000 DV (USA, Perkin Elmer). X-ray fluorescence analysis was performed on a Venus 200 PANalytical B.V. spectrometer with wavelength dispersion. X-ray phase analysis of powder nickel-rhenium-containing alloys was performed on a D8 Advance (BRUKER) diffractometer with Cu K $\alpha$  radiation. The deposits were also analyzed using JEOL JXA-8230 electron probe micro-analyzer.

In the analyses solutions of sulphuric acid and nickel sulphate, ammonium sulphate, and ammonium perrhenate were used. The grade of sulphuric acid, nickel sulphate, and ammonium sulphate corresponded to the high grade and analytical reagent grade. Ammonium perrhenate was of AR-0 grade.

**Results and Discussion.** The deposit collected on the cathode as a result of electrocrystallization may be loose or thick layer consisting of numerous microcrystallites. The structure of the deposit depended on many factors such as the nature of the substance and solvent, concentration of impurities, adhesive properties of the deposits, temperature of the environment, diffusion conditions, etc. The alloy composition also depends on the cathode current density, electrolyte composition, temperature and mixing. Therefore, specific conditions must be created to electrolytic deposition of powder alloys of the specified composition.

Maintenance of electrolysis process in a membrane system will allow excluding use of platinum, that's why this was a basis for performing studies connected in development of a technology for producing powder electrolytic nickel-rhenium-containing alloys by membrane electrolysis.

Studies were performed in order to explore the influence of the electrolyte composition on the chemical and phase composition, structure and current efficiency of powders nickel-rhenium-containing alloys.

Sulphurous ammonium sulphate electrolyte (average composition, g/dm<sup>3</sup>: 12-30 NiSO<sub>4</sub>·7H<sub>2</sub>O, 1.0-7.2 NH<sub>4</sub>ReO<sub>4</sub>, 20-80 (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>, 6.6-15.0 H<sub>2</sub>SO<sub>4</sub>) was used as an electrolyte for deposition of powder Ni-Re-containing alloys by membrane electrolysis.

Experiments were performed for producing powder alloys from electrolytes (composition, g/dm<sup>3</sup>: 7.2 NH<sub>4</sub>ReO<sub>4</sub>, 40 (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>, 6.6 H<sub>2</sub>SO<sub>4</sub>) with different concentration of nickel sulphate (12; 15; 20; 30 g/dm<sup>3</sup>) with the cathode current density 200 A/m<sup>2</sup>, temperature 50°C, and four-hour-long process. Dark-grey and light-grey metalescent deposits were produced. When the concentration of nickel sulphate

is increased the current efficiency increases and is 56.6, 72.6, 82.7, and 89.8% in experiments 8, 5, 2, and 32, respectively. According to the results of X-ray fluorescence and chemical analyses of powder nickel-based alloys, no clear patterns of the relation between the content of nickel and rhenium in the cathode deposits and increase in the concentration of nickel sulphate in the electrolyte have been found. The average contents of nickel and rhenium in the deposits were 47.5-54.9 (wt.%) and 37.6-47.5 (wt.%), respectively.

The structure of powder alloys determined by petrographic analysis is shown in Figure 2. The higher is the concentration of nickel sulphate in the electrolyte, the lesser is the size of deposited powder particles, along with a change in the magnetic characteristics of the powders. Powder No 8 is strongly magnetic, No5 and No 2 are weakly magnetic, and No32 is not magnetic.

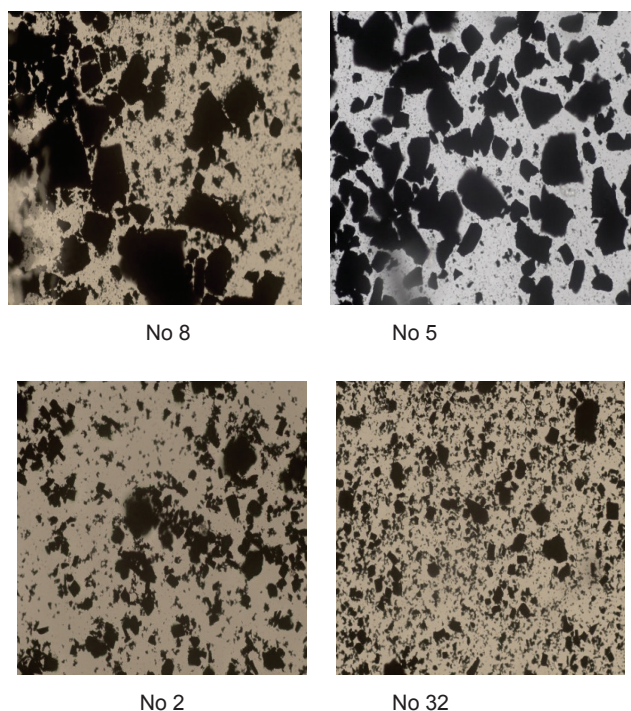


Figure 2 – Structure of powder nickel-rhenium-containing alloys depending on the concentration of nickel in the electrolyte according to the petrographic analysis data (200-fold magnification)

The data of the X-ray phase analysis (Table 1) show that the alloy deposits contain, primarily, solid solution of Re in Ni and solid solution of Ni in Re. The use of the method to determine an average size of particles based on the broadening of diffraction reflections on the X-ray pattern of alloys (the Sherrer equation) allows for crystallization of the produced Ni-Re alloys with isolation of finely-divided phases with the particle size 8.2 to 11.2 nm.

Table 1 – Results of X-ray phase analysis of powder alloys

Exp. No	Content of $\text{NiSO}_4$ in electrolyte, g/dm <sup>3</sup>	Compound Name
8	12	Nickel, syn Ni - 100.0%, Crystallite Size (Scherrer) 8.4 nm; Nickel (Cubic) Ni, Cell param.: Initial a=0.352 nm (Final a=0.352 nm); Nickel (Hexagonal) Ni, Crystallite Size 8.2 nm, Cell param.: Initial a=0.266 nm (Final a=0.262 nm); Rhenium (Hexagonal) Re, Crystallite Size 8.3 nm, Cell param.: Initial a=0.265 nm, c=0.445 nm (Final a=0.276 nm, c=0.446 nm); Rhenium (Cubic) Re, Crystallite Size 9.4 nm, Cell param.: Initial a=0.400 nm (Final a=0.395 nm). The deposited alloys contain phase of metallic Ni, solid solutions of Re in Ni and of Ni in Re.
5	15	Nickel (Cubic) Ni, Crystallite Size 8.3 nm, Cell param.: Initial a=0.356 nm (Final a=0.352 nm); Nickel (Hexagonal) Ni, Crystallite Size 8.2 nm, Cell param.: Initial a=0.263 nm, c=0.421 nm (Final a=0.262 nm, c=0.432 nm); Rhenium (Hexagonal) Re, Crystallite Size 8.4 nm, Cell param.: Initial a=0.274 nm, c=0.441 nm (Final a=0.276 nm, c=0.446 nm). The alloy deposits contain phase of metallic Ni, solid solutions of Re in Ni and solid solutions of Ni in Re.
2	20	Nickel (Cubic) Ni, Crystallite Size 8.3 nm, Cell param.: Initial a=0.355 nm (Final a=0.352 nm); Rhenium (Hexagonal) Re, Crystallite Size 8.4 nm, Cell param.: Initial a=0.268 nm, c=0.436 nm (Final a=0.276 nm, c=0.446 nm). The deposited alloys contain solid solution of Re in Ni and solid solution of Ni in Re.
32	30	Nickel (Hexagonal) Ni, Crystallite Size 8.3 nm, Cell param.: Initial a=0.264 nm, c=0.425 nm (Final a=0.262 nm, c=0.432 nm); Rhenium (Hexagonal) Re, Crystallite Size 11.2 nm, Cell param.: Initial a=0.274 nm, c=0.443 nm (Final a=0.276 nm, c=0.446 nm). The deposited alloy contain solid solution of Ni in Re.

Experiments were conducted for producing powder alloys from electrolytes (composition, g/dm<sup>3</sup>: 12  $\text{NiSO}_4 \cdot 7\text{H}_2\text{O}$ , 40  $(\text{NH}_4)_2\text{SO}_4$ , 6.6  $\text{H}_2\text{SO}_4$ ) with various concentrations of ammonium perrhenate (1-7.2 g/dm<sup>3</sup>) at the cathode current density of 200 A/m<sup>2</sup>, temperature 50°C, and process duration for 4 hours. Dark-grey deposits were produced some of which were removed from the cathode in the form of film, but became more friable with increase of the concentration of ammonium perrhenate. No clear patterns of dependencies of current efficiency and nickel and rhenium content in the alloy powders were identified with an increase in the concentration of ammonium perrhenate in the electrolyte. When the concentration of ammonium perrhenate was increased (1; 3; 5; and 7.2 g/dm<sup>3</sup>) the current efficiency was 44.9; 83.6; 56.0; 82.7 %, respectively (experiments 40, 35, 13, and 2). Based on the results of X-ray fluorescence and chemical analysis of powder alloys, the average content of Ni and Re in the deposits is 49.0-71.9 wt. %: and 22.7-46.5 wt. %, respectively.



Figure 3 shows the structure of the powder alloys based on the petrographic analysis data. No specific pattern of influence of the concentration of ammonium perrhenate on the powder particle size was identified, still magnetic characteristics of powders change: strongly magnetic (samples 40 and 13), weakly magnetic (2), and non-magnetic (35).

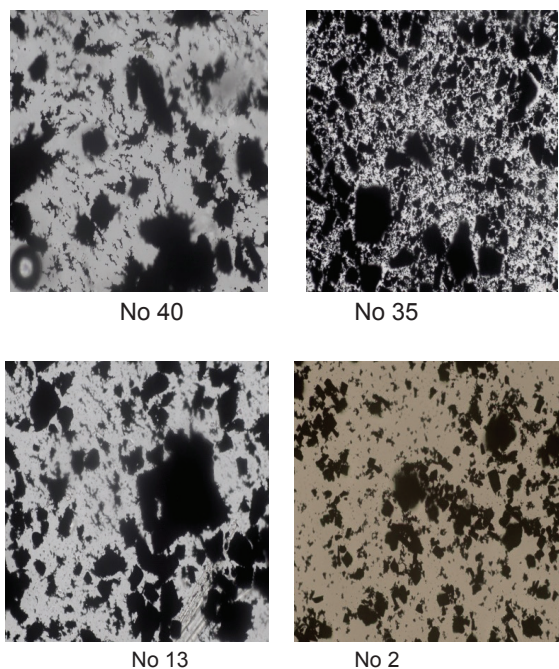


Figure 3 – Structure of powder nickel-rhenium-containing alloys depending on concentration of ammonium perrhenate in the electrolyte based on petrographic analysis data (200-fold magnification)

According to X-ray phase analysis data the deposited alloys contain solid solution of Re in Ni (samples 40 and 35) and solid solutions Re in Ni and Ni in Re (samples 13 and 2). The resulted alloys are crystallized with separation of finely-divided phases with particles size of 15.4-8.4 nm. Figure 4 shows X-ray pattern of the deposit of Sample 2.

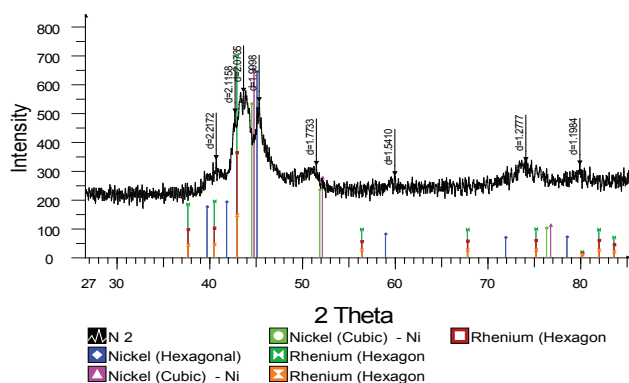


Figure 4 – X-ray pattern of powder nickel-rhenium-containing alloy of Sample 2

The study demonstrated influence of the cathode current density on the chemical and phase composition, structure and current efficiency of nickel-rhenium-containing alloys. Powder alloys were deposited from sulphuric ammonium-sulphate electrolytes (composition, g/dm<sup>3</sup>: 20 NiSO<sub>4</sub>·7H<sub>2</sub>O; 7.2 NH<sub>4</sub>ReO<sub>4</sub>; 40 (NH<sub>4</sub>)<sub>2</sub> SO<sub>4</sub>; 6.6 H<sub>2</sub>SO<sub>4</sub>) at 50 °C during 4-hours-long process. The cathode current efficiency was measured within the range of 150, 200, 250, 300 A/m<sup>2</sup>.

Light-grey metalescent deposits were obtained at current density of 150 A/m<sup>2</sup> and dark-grey deposits were obtained at higher current densities. The relationships between the current efficiency and content of nickel and rhenium in the resulted deposits and the cathode current density are shown in Figure 5. When the current density increases, the current efficiency increases and is 60.6; 82.7; 98.3; 94.9 %, respectively (experiments 22, 2, 25, 27). According to the results of X-ray fluorescence analysis of powder alloys with increase in the cathode current density the content of nickel in deposits increases from 48 to 63 % and decreases to 60 % at the current density of 300 A/m<sup>2</sup>; whereas the content of rhenium decreases from 49 to 33 % and then increases to 36 %.

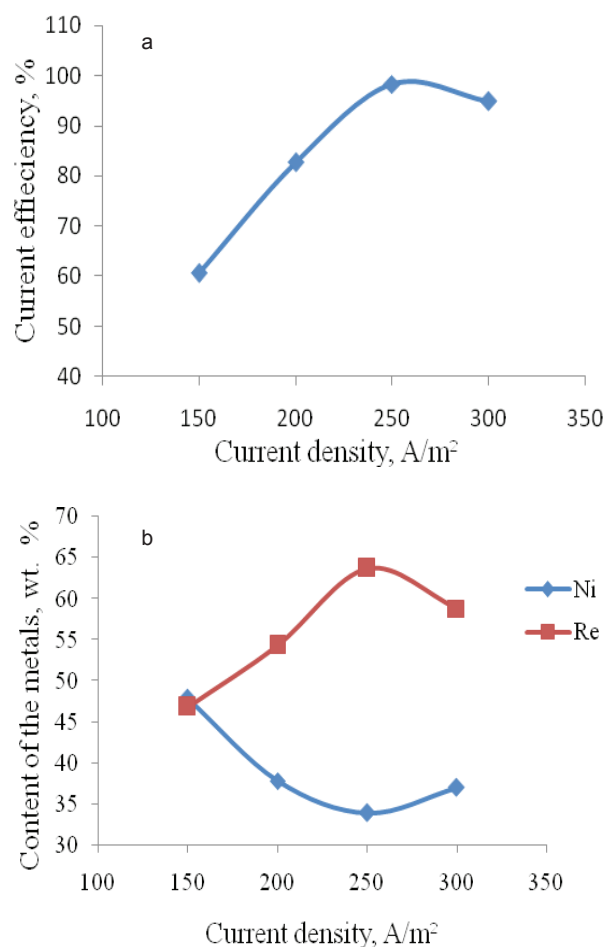


Figure 5 – Influence of cathode current density on alloy current efficiency (a) and content of the metals (Ni, Re) in the alloy (b)

According to X-ray phase analysis data all alloy deposits, irrespective of the cathode current density, contain solid solutions of Re in Ni and solid solutions of Re in Ni. The resulted alloys are crystallized with separation of finely-divided phases with particles size of 8.2-8.4 nm.

In the petrographic analysis no specific differences in the structure of powder nickel-rhenium-containing alloys depending on the current density were identified, however the powders deposited at the current density of 150 A/m<sup>2</sup> are weakly magnetic and become strongly magnetic when the current density increases.

The influence of temperature on the chemical and phase composition, structure and current efficiency of powder nickel-rhenium-containing alloys was studied. Sulphurous ammonium sulphate electrolyte (composition, g/dm<sup>3</sup>: 20 NiSO<sub>4</sub>·7H<sub>2</sub>O; 7.2 NH<sub>4</sub>ReO<sub>4</sub>, 40 (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>, 6.6 H<sub>2</sub>SO<sub>4</sub>) was used as an electrolyte for deposition of powder Ni-Re-containing alloys by membrane electrolysis. Powders were deposited at the cathode current density 200 A/m<sup>2</sup> over 4 hours; the electrolyte temperature was varied within the range of 30, 40, 50, and 60°C.

When the electrolyte temperature increases the current efficiency increases from 74 to 95 %. The colour of deposits changes from light-grey to dark-grey. Cathode deposit which was obtained at 60 °C in form of film is easily disintegrated.

The petrographic analysis showed that the powders became more finely-divided and their magnetic properties increase with increase in the temperature.

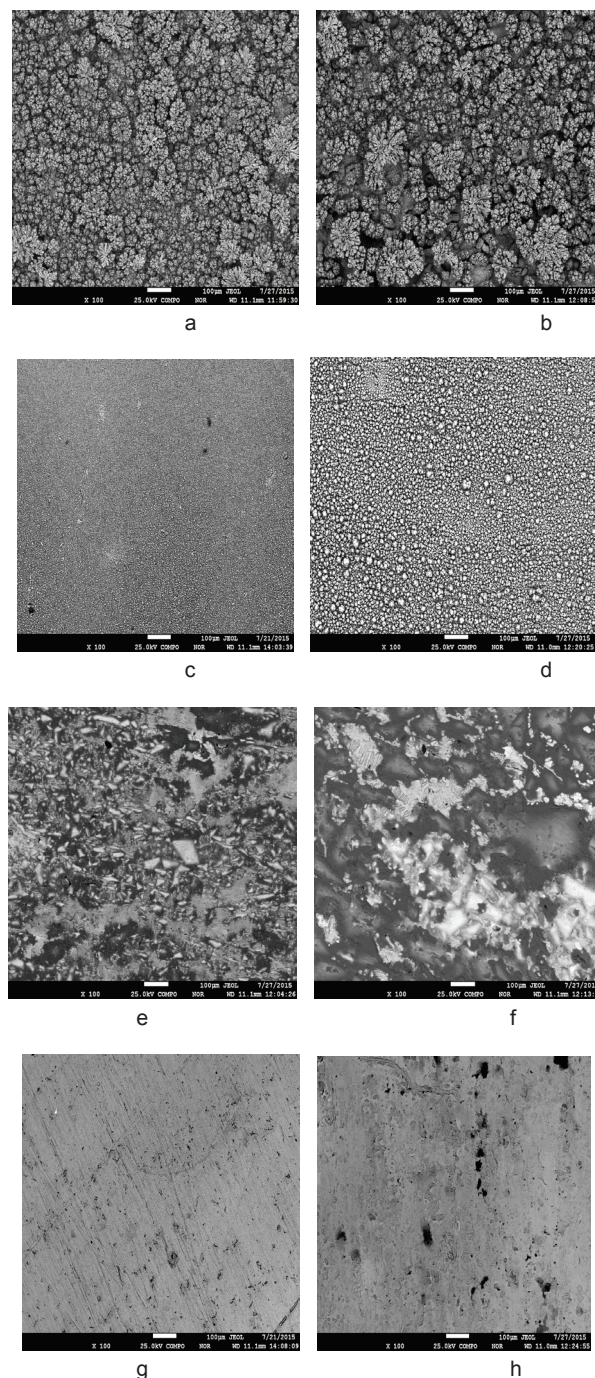
The X-ray fluorescence and chemical analysis of powder alloys deposited from electrolytes, depending on their temperature, showed that the content of nickel increased from 56 to 61 % and the content of rhenium ranged from 35 to 38 % when the temperature increased.

Based on the X-ray phase analysis data, all deposits of alloys, irrespective of the temperature, contain solid solutions of Re in Ni and Ni in Re. The produced alloys are crystallized with separation of finely-divided phases with a particles size of 8.2-16.6 nm.

Experiments were conducted for depositing powder Ni-Re-containing alloys by membrane electrolysis within the temperature range from 30 to 60 °C in somewhat modified conditions: the alloys were deposited from an electrolyte with a modified composition (g/dm<sup>3</sup>: 20 NiSO<sub>4</sub>·7H<sub>2</sub>O; 2 NH<sub>4</sub>ReO<sub>4</sub>, 80 (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>, 6.6 H<sub>2</sub>SO<sub>4</sub>) and the process time reduced to 3 hours. Dark-grey cathode deposits with different densities were obtained. According to the X-ray phase analysis data the powder alloys are solid solutions of rhenium in nickel (a cubical lattice with  $a = 0.35\text{-}0.36$  nm) and nickel in rhenium (hexagonal lattice with  $a = 0.27$  nm,  $c = 0.44$  nm). The powder

alloys deposited within the temperature interval 40-60 °C were more friable. Depending on the process temperature (30; 40; 50; 60 °C) the current efficiency of the powder alloys was 51.7; 56.4; 85.4; 52.0 %, respectively.

The microstructure of the powder alloys (Figure 6) and their chemical composition (Table 2) was analyzed on JEOL JXA-8230 electron probe micro-analyzer.



Temperature, °C: a, e – 30; b, f – 40; c, g – 50; d, h – 60; a, b, c, d – solution facing deposit surface; e, f, g, h – cathode facing deposit surface

Figure 6 – Microstructure of cathode deposits of Ni-Re alloys obtained at different temperature (100-fold magnification)



Table 2 – Chemical composition of cathode deposits of Ni-Re alloys deposited from electrolytes at different temperature (JEOL JXA-8230 micro-analyzer)

Sample number	Temperature electrodeposition of powders, °C	Content, wt. %			
		Ni	Re	C + O	Ti
11	30	79,11	8,35	3,54	-
12	40	76,96	19,43	3,21	-
7	50	93,91	1,69	4,40	-
13	60	90,83	4,84	4,44	-

The obtained results showed that, in general, when the temperature increase, the content of nickel in Ni-Re powders increases from 76 to 93 wt. %, and the content of rhenium decreases from 19 to 1.7 wt. %. When the temperature increases, the powder alloys become more friable and easily disintegrated.

**Conclusions.** The influence of some main electrolysis parameters (electrolyte composition, cathodic current density, temperature) on chemical and phase composition, structure and current efficiency of powder Ni-Re-containing alloys deposited on the titan cathode was determined. Deposition of powder alloys from sulphurous ammonium sulphate electrolytes (average composition, g/dm<sup>3</sup>: 12-30 NiSO<sub>4</sub>·7H<sub>2</sub>O, 1.0-7.2 NH<sub>4</sub>ReO<sub>4</sub>, 20-80 (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>, 6.6-15.0 H<sub>2</sub>SO<sub>4</sub>) showed that increase in the nickel sulphate concentration (from 12 to 30 g/dm<sup>3</sup>) is associated with increase in the current efficiency from 56 to 90%; the average content of nickel and rhenium in the alloys is 47.5-54.9 (wt.%) and 37.6-47.5, respectively. Based on the X-ray fluorescence data the alloys are based on solid solutions of Re in Ni and Ni in Re with the particles size 8.2-11.2 nm. When the concentration of ammonium perrhenate increased (from 1 to 7.2 g/dm<sup>3</sup>), the current efficiency was from 45 to 84 %; the average content of nickel and rhenium in the deposits was 49.0-71.9 wt.% and 22.7-46.5 wt. %, respectively. The alloys were based on solid solutions of Re in Ni and Ni in Re with the particles size 15.4-8.4 nm.

Increase in the current density (from 150 to 300 A/m<sup>2</sup>) was associated with an increase in the current efficiency from 60 to 95 %. The analysis of the powder alloys showed that increase in the cathode current density associated with the increase in the content of nickel from 48 to 63 (wt.%) and decrease in the content of rhenium from 49 to 36 (wt.%). According to X-ray fluorescence data the alloy deposits contain solid solutions of Re in Ni and solid solutions of Re in Ni with particles size 8.2 to 8.4 nm irrespective of the cathode current density.

When the temperature of electrolyte increases from 30 to 60°C, the current efficiency increases from 74 to 95%. Petrographic analysis showed that with increase in the temperature the powders became more finely-divided, and their magnetic properties increased. The analysis showed that when temperature increased, the content of nickel in the powders increased from 56 to 61 (wt.%), and the content of rhenium varied within the range of 35 to 38 (wt.%). When powder Ni-Re-containing alloys were deposited from an electrolyte with the modified composition (g/dm<sup>3</sup>: 20 NiSO<sub>4</sub>·7H<sub>2</sub>O; 2 NH<sub>4</sub>ReO<sub>4</sub>, 80 (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>, 6.6 H<sub>2</sub>SO<sub>4</sub>), the content of nickel in the obtained Ni-Re powders increased from 76 to 93 (wt.%), and the content of rhenium decreased from 19 to 1.7 (wt.%) with increase in the temperature. Depending on the process temperature the current efficiency of powder alloys was 51.7 to 85.4%. With increase in the temperature the powder alloys are more friable and easily disintegrated. The X-ray fluorescence analysis showed that the alloy deposits contain solid solutions of Re in Ni and Ni in Re with the particles size 8.2 to 16.6 nm, irrespective of the temperature.

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#### ТҮЙІНДЕМЕ

Құрамында никель-ренийі бар қорытпалардың электролиттік ұнтақтарын алу бойынша зерттеулерлер нәтижелері келтірілген. Бұл қорытпалар ұқсас құймалы қорытпаларға қарағанда айқын құнды техникалық сипаттамаларға ие, және де ұнтақты металлургияда қолданылуы мүмкін. Электролиздің кейбір негізгі көрсеткіштерінің (электролит құрамы, катодтық тоқ тығыздығы, температура) химиялық және фазалық құрамына, құрылымына, титан катодына отырғызылатын никель-рений құрамды қорытпалардың тоқ бойынша шығымына (ТШ) әсері анықталған. Қорытпалар рентгенофлуоресценттік, рентгенофазалық, химиялық, электронды-микроскопиялық, петрографиялық әдістермен зерттелген. Аммоний-сульфатты электролиттерден қорытпалар ұнтағын отырғызу (орташа құрамы, г/дм<sup>3</sup>: 12-30 NiSO<sub>4</sub>, 1,0-7,2 NH<sub>4</sub>ReO<sub>4</sub>, 20-80 (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>, 6,6-15,0 H<sub>2</sub>SO<sub>4</sub>) никель сульфатының концентрациясының көбеюімен тоқ шығымы 56 – дан 90 % - ға дейін жоғарлайтынын көрсетті. Қорытпалардың орташа құрамы, мас. %: 47,5-54,9 Ni, 37,6-47,5 Re. Аммоний перренаты концентрациясының өсуімен тоқ шығымы 45 – тен 84 % -ды құрайды. Қорытпалардың орташа құрамы, мас. %: 49,0-71,9 Ni, 22,7-46,5 Re. Тоқ тығыздығының өсуімен (150-300 А/м<sup>2</sup>) тоқ шығымы 60 - дан до 95 % - ға жоғарылайды. Қорытпаларда Ni құрамы 48 -дан 63 мас. % -ға жоғарылайды; Re - 49 -дан 36 мас. % - ға төмендейді. Электролит температурасының жоғарылауымен (30-60 °C) ТШ 74 - тен 95 % - ға көтеріледі; қорытпадағы Ni құрамы 56 - дан 61 мас. % - ға өседі, Re - 35-38 мас. % шамасында болады. Электролит құрамын өзгерту кезінде (г/дм<sup>3</sup>: 2 NH<sub>4</sub>ReO<sub>4</sub>, 80 (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>) температура жоғарылауымен алынған балқымаларда Ni 76 -дан 93 мас. % - ға өседі, Re – 19 - дан 1,7 мас. % - ға

төмендейді; Қорытпалардың тоқ шығымы 51,7 - дан 85,4 % - ды құрайды. Температураның жоғарылауымен қорытпалар ұнтағы борпылдақ және тез үгілгіш келеді. РФТ нәтижесі бойынша электролиз параметрлеріне қарамастан қорытпалар негізін түйіршік өлшемі 8,2-16,6 нм болатын Re - дің Ni - гі және Ni - дің Re – гі қатты ерітінді болып табылады.

**Түйін сөздер:** ұнтақтар, элетролиттік қорытпалар, никель, рений, электрлік отырғызу, электролит құрамы, тоқ тығыздығы, температура, тоқ бойынша шығымы.

### РЕЗЮМЕ

Приведены результаты исследований по получению порошков электролитических Ni-Re-содержащих сплавов. Эти сплавы по сравнению с аналогичными литейными сплавами обладают более ярко выраженными ценными техническими характеристиками и могут быть использованы в порошковой металлургии. Определено влияние некоторых основных параметров электролиза (состав электролита, катодная плотность тока, температура) на химический, фазовый состав, структуру и выход по току (ВТ) осаждаемых на титановом катоде Ni-Re-содержащих сплавов. Сплавы анализировали рентгенофлуоресцентным, рентгенофазовым, химическим, электронно-микроскопическим, петрографическим методами. Осаждение порошков сплавов из аммонийно-сульфатных электролитов (средний состав, г/дм<sup>3</sup>: 12-30 NiSO<sub>4</sub>, 1,0-7,2 NH<sub>4</sub>ReO<sub>4</sub>, 20-80 (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>, 6,6-15,0 H<sub>2</sub>SO<sub>4</sub>) показало, что с повышением концентрации сульфата никеля ВТ возрастает от 56 до 90 %. Сплавы в среднем содержат, мас. %: 47,5-54,9 Ni, 37,6-47,5 Re. С ростом концентрации перрената аммония ВТ составляет от 45 до 84 %. Сплавы в среднем содержат, мас. %: 49,0-71,9 Ni, 22,7-46,5 Re. С ростом плотности тока (150-300 А/м<sup>2</sup>) ВТ возрастает от 60 до 95 %. В сплавах содержание Ni возрастает от 48 до 63 мас.%; Re - снижается от 49 до 36 мас.%. С повышением температуры электролита (30-60 °С) ВТ возрастает от 74 до 95 %; содержание Ni в сплавах повышается от 56 до 61 мас.%, Re - колеблется в пределах 35-38 мас.%. При изменении состава электролита (г/дм<sup>3</sup>: 2 NH<sub>4</sub>ReO<sub>4</sub>, 80 (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>) в полученных сплавах с ростом температуры возрастает содержание Ni от 76 до 93 мас.%, Re – снижается от 19 до 1,7 мас.%; ВТ сплавов составил от 51,7 до 85,4 %. С ростом температуры порошки сплавов получают более рыхлыми и легко измельчаются. По данным РФА независимо от параметров электролиза основу сплавов представляют твердые растворы Re в Ni и Ni в Re с размером зерен 8,2-16,6 нм.

**Ключевые слова:** порошки, электролитические сплавы, никель, рений, электроосаждение, состав электролита, плотность тока, температура, выход по току.

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