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**THE PROPERTIES OF CATHODE DEPOSITS
AT ELECTROCHEMICAL PROCESSING
OF ALLOY GS32-VI USING ACIDIC ELECTROLYTES**

Abstract. The article presents the results of studying the composition, surface morphology and granulometric analysis of cathodic precipitates obtained by electrochemical processing of the spent heat-resistant alloy GS32-VI using acid electrolytes the anodic dissolution of which was carried out using the electrochemical technological complex EHK-1012 (developed by Tetran, Russian Federation).

Studies of the properties of cathode sediments were carried out using the following methods of research, analysis and instrumentation: elemental analysis was carried out using an ICP mass spectrometer for isotope and elemental analysis of ELAN DRC-e (PerkinElmer, Canada); X-ray phase analysis - using a Shimadzu XDR 6000 diffractometer (Japan); particle size determination by the method of dynamic light scattering measurement - on the submicron particle size analyzer and the zeta potential Delsa™ Nano, PN A54412AA; the morphology of the sediment surface was studied using the scanning electron microscopy (SEM) method using the JEOL JSM 6390 L electron microscope.

It is shown that, depending on the nature of the electrolyte, cathode deposits of different chemical, phase composition can be obtained and differ in the size and morphology of the surface. In particular, it is possible to obtain cathode deposits of different chemical composition: from sulphate electrolyte, a powder with a nickel content of about 95%; from hydrochloric acid electrolyte - powder of composition Ni: Co: Al = 4: 1: 1; from nitric acid electrolyte - powder of composition Ni: Re: Co: Al = 3: 1: 1: 1. All the cathode deposits obtained had a dendritic structure, the development of which also depended on the nature of the electrolyte, the deposits with the most developed structure and the having smallest particle size obtained using a nitric acid electrolyte.

Keywords: electrochemical processing, heat-resistant nickel alloy, acid electrolyte, cathode deposit, morphology, dendritic, grain fineness.

Introduction. In recent years, the heat resisting alloys that are not losing the properties for a long time in the conditions of high temperatures and a complicated state were widely used. Such alloys are produced on aluminum, titanitic, iron, copper, cobalt and nickel bases.

Heat resisting alloys on a nickel basis that part rare and scattered elements, such as rhenium, tungsten, molybdenum and tantalum are of particular importance.

The amount of the fulfilled heat resisting alloys constantly grows. Problems of rational and integrated use of secondary raw material resources acquire the increasing value, relevance and prospects in the overall balance of production of metals both in the world and in the Republic of Kazakhstan. In particular, gas turbine engines, whose blades are made of nickel refractory alloys, are used not only in aviation, but also in thermal power plants, which are the main electricity producers in the Republic of Kazakhstan. A small amount of spent heat-resistant nickel alloys is formed due to the wear of individual parts of the internal combustion engines of motor vehicles, and the waste of heat-resisting alloys accumulates at the “Baikonur” spaceport.

The most economically significant is the waste of heat-resisting nickel alloys GS32, for which the composition is typical, %: Ni ~ 60, W and Co 5-10; Re and Ta 2-4; Nb 1.5-2; Mo, Cr and Al up to 5. One of the brightest representatives of this series - the alloy GS32-VI - is serially manufactured in VIAM [1, 2].

Existing technologies for processing waste heat-resisting nickel alloys can be divided into 4 groups:

- direct pyrometallurgical processing of waste of heat-resistant alloys on a nickel basis [3, 4];
- thermal oxidation technology to convert a number of components in water-soluble form [5, 6];
- hydrochemical technologies [7, 8];
- electrochemical techniques [9-12].

From the list of the indicated areas of processing of wastes of nickel-containing alloys doped with rare and scattered elements, and in particular rhenium, in recent years electrochemical and combined technologies have been increasingly used [13, 14].

When processing such alloys with electrochemical methods, electrolytes, cathode deposits and anode slimes are formed. Each of these products is either a valuable raw material for producing non-ferrous and rare metals (anode slimes and electrolytes) or finished products in the form of an individual or multicomponent metal powder (cathode deposit).

The purpose of the present studies is to determine the effect of the nature of the electrolyte on the composition and structural characteristics of the cathode deposits obtained in the course of electrochemical processing of the heat-resistant alloy GS32-VI.

Experimental. The objects of the present study were samples of the heat-resisting nickel alloy GS32-VI and cathode deposits obtained by electrochemical processing of this alloy.

Elemental analysis of the alloy GS32-VI and the resulting cathode precipitates was carried out using an ICP mass spectrometer for isotope and elemental analysis of ELAN DRC-e (Perkin Elmer, Canada).

Pre-weighed the alloy completely (without a solid residue) was dissolved in a strong solution of nitric acid. The alloy had the composition shown in table.

Elemental composition of the alloy GS32-VI

Composition, wt. %													
Ni	Co	W	Al	Cr	Re	Ta	Y	Nb	Mo	C	B	Ce	La
60,05	9,3	8,6	6,0	5,0	4,0	4,0	0,005	1,6	1,1	0,16	0,15	0,025	0,005

Electrochemical dissolution of the alloy GS32-VI was carried out using the electrochemical technological complex EHK-1012 (developed by Tetran LLC, Russian Federation), using an uncompensated method for measuring the potential [15]. The electrochemical cell consisted of a 300 ml volume of PTFE, the anode was a fragment of the blade from the heat-resistant alloy GS32-VI, area $S = 10 \text{ cm}^2$, cathode – titanium plate (VT1-0), area $S = 12 \text{ cm}^2$.

As electrolytes, solutions of mineral acids were used: sulfuric, hydrochloric and nitric with a concentration of 100 g/l, the choice of which was made based on literary data [16, 17]. The volume of electrolytes was 200 ml.

The process of electrochemical dissolution of the alloy GS32-VI was carried out at a controlled current of 2.0 A for 1-12 hours at a temperature of 20-32 °C without thermostating, the stirring was carried out with a magnetic stirrer of the MM-2A brand. At the end of the process, the electrolyte solution was analyzed for the content of nickel and rhenium in it. The resulting cathode pellet was washed and dried. Elemental and phase analysis of the resulting cathode deposits was then carried out and the morphology of their surface and the particle size distribution were determined.

The phase analysis of cathode sediments was performed using a Shimadzu XDR 6000 diffractometer (CuK α radiation, sample rotation, continuous (1 deg/min), stepwise (step 0.02 °, exposure 10 s) modes in the angle range 2 Θ 10-90.

Determination of cathode precipitate particle size was performed by dynamic light scattering measurement using a submicron particle size analyzer and the zeta potential Delsa TM Nano, PN A54412AA.

Morphology and crystal-chemical characteristics of cathode deposits were studied using the scanning electron microscopy (SEM) method using the JEOL JSM 6390 L electron microscope.

Results and discussion. During the electrochemical processing in the hydrochloric acid electrolyte, the content of nickel and rhenium increases steadily. The concentration of nickel at the end of the process was 15.06 g/l, and rhenium – 1.6 g/l.

The resulting cathode deposit was washed, dried and analyzed by X-ray phase analysis, which resulted in the identification and revealed that the main phase is Al_4CoNi_2 intermetallide.

The chemical analysis allowed to determine the weight ratio of the components of the cathode deposit – Ni:Co:Al = 4:1:1. In the composition of the cathode deposit, no other metals were detected. Consequently, chromium and rhenium in the anodic dissolution of the heat-resistant nickel alloy GS32-VI in the solution of hydrochloric acid are accumulated in the electrolyte.

It was of interest to study the effect of duration on electrolysis when using hydrochloric acid electrolyte on the composition of the cathode deposit (figure 1).

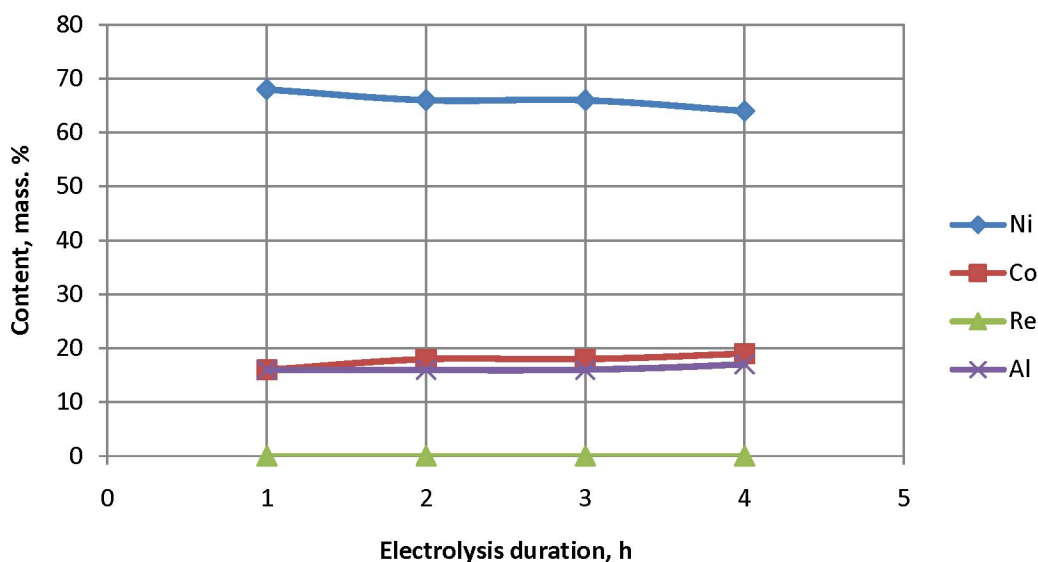


Figure 1 – Influence of duration of the electrolysis on composition of cathode deposit received when using hydrochloric acid electrolyte

It follows from figure 1 that the duration of electrolysis has practically no effect on the composition of the cathode deposit, there is a slight decrease in the content of nickel in the precipitate while simultaneously increasing the content of cobalt in it. The average weight ratio of the components of the cathode deposit also turned out to be equal to Ni: Co: Al = 4: 1: 1, that is, it did not depend on the duration of the process.

The resulting cathode deposit was fine, dark gray in color. Micrographs of the cathode deposit obtained by electrochemical processing of the heat-resisting alloy GS32-VI are shown in figure 2.

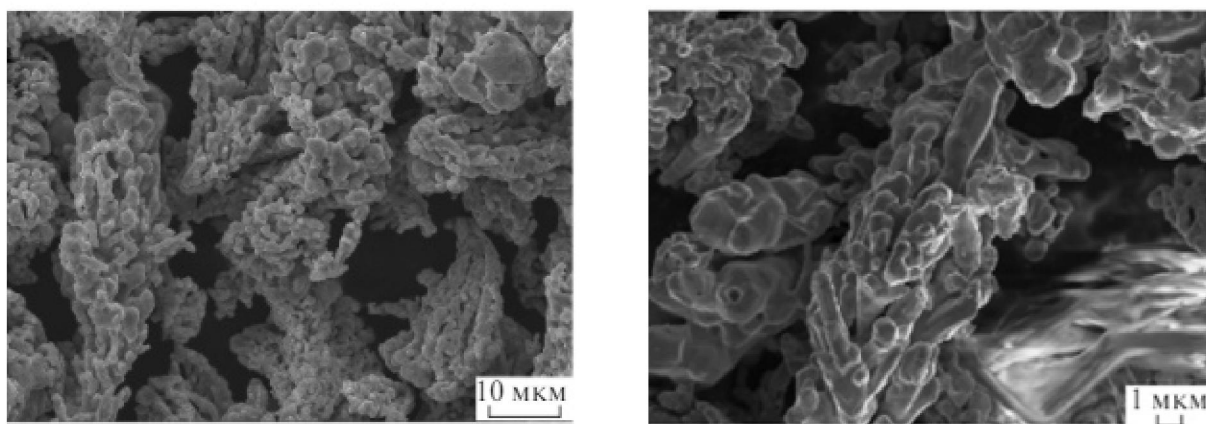


Figure 2 – Micrographs of the cathode nickel-containing deposit obtained by electrochemical processing of the alloy GS32-VI with the use of hydrochloric electrolyte

The SEM-images of the received cathode deposit at different magnifications show that the structure reminds dendrites, similar to corals, and separate particles of powder grow together among themselves.

At bigger magnification, it is possible to distinguish the layered structure of a surface of dendrites that says that it is desirable to anneal the received cathode deposits for stabilization in the neutral atmosphere.

Particle size distribution and particle size of the cathode deposit, resulting in electrochemical processing GS32-VI alloy using hydrochloric electrolyte is shown in figure 3, from which it follows that the average particle size of the cathode deposit is about 40 microns.

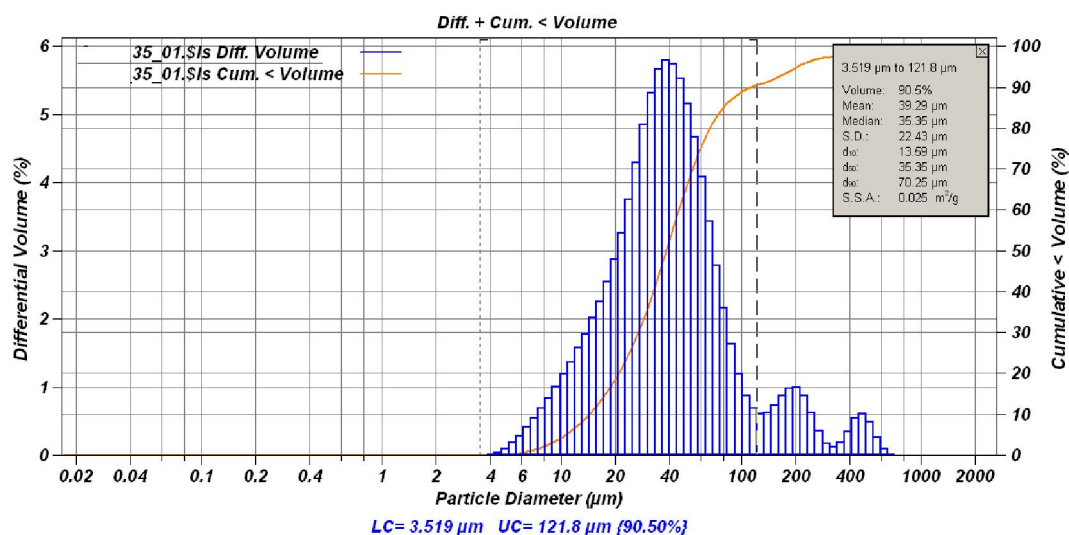


Figure 3 – Distribution of cathode deposit particles obtained using hydrochloric electrolyte

Thus, the use of hydrochloric electrolyte in the electrochemical processing of the heat-resistant GS32 alloy made it possible to obtain on the cathode a finely dispersed nickel-cobalt concentrate of a dendritic structure with a particle size of about 40 µm and a ratio of Ni: Co: Al = 4:1:1 components in it.

In the anodic dissolution of a heat-resisting GS32-VI alloy in a sulfate electrolyte under the conditions similar to those described above, the data presented in Fig. 4 were obtained, from which it follows that as the duration of electrolysis increases, the amount of nickel in the cathode deposit increases and the amount of cobalt decreases. Rhenium was not found in the composition of the cathode deposit, aluminum was in trace amounts. The main component of the cathode deposit was nickel, the content of which was 94-96%.

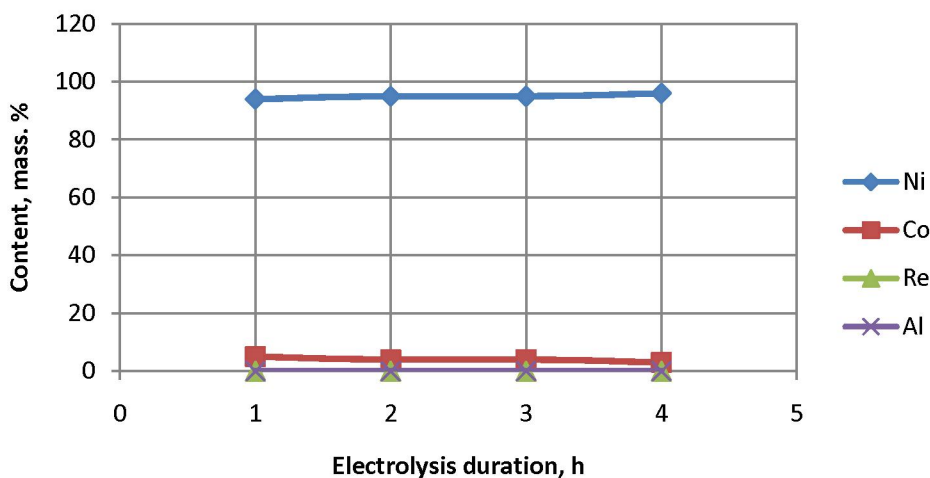


Figure 4 – Influence of the duration of electrolysis on the composition of cathode deposits obtained with the use of sulfate electrolyte

Microphotographs of the obtained cathode deposit during electrolysis from sulfate electrolyte at different magnifications are shown in figure 5.

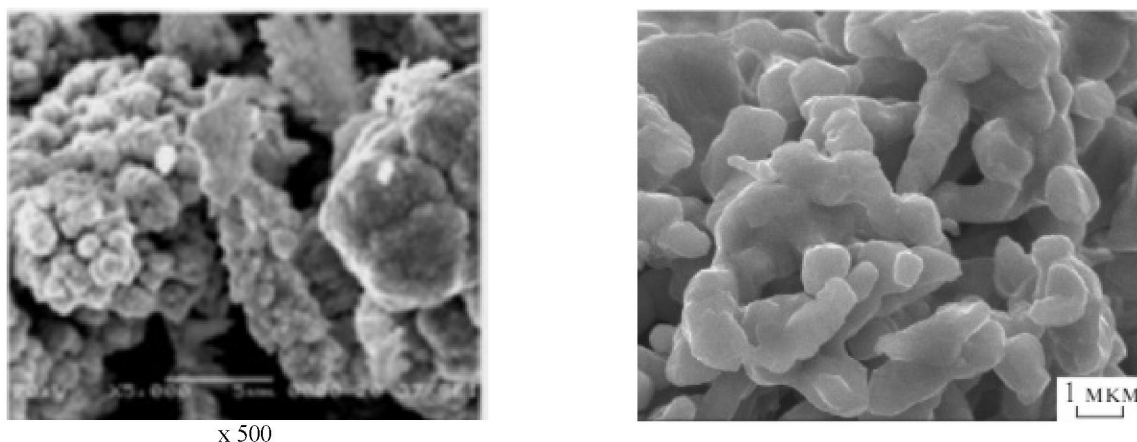


Figure 5 – Microphotographs of the cathode deposit of the heat resisting GS32-VI alloy received at electrochemical processing in sulfate electrolyte

Apparently, from the figure 5, the received deposit has dendritic structure which differs from the structure of the cathode deposit received at electrochemical processing of heat resisting GS32-VI alloy in the hydrochloric electrolyte: the structure remains the same dendritic, but particles among themselves grow together more.

Thus, at electrochemical processing of heat-resisting GS32-VI alloy with use of sulfate electrolyte as a part of a cathode deposit of dendritic structure rhenium isn't found, aluminum – trace quantities, a deposit represents a mechanical mix of two metals – nickel and cobalt, with nickel in the sediment averaging 95 %.

Electrochemical dissolution of the heat-resisting GS32-VI alloy using a nitrate electrolyte with a concentration of 100 g/l was carried out for 12 hours at a controlled current strength $I = 2.0$ A with the electrolyte agitated on a magnetic stirrer of the MM-2A brand. During the process, it was found that the nickel concentration in the electrolyte was 22.9 g/l, the rhenium concentration was 15.29 g/l.

Influence of duration on structure of the received cathode deposit by analogy with the above-described experiments is given in the figure 6 from which follows that as a part of a deposit there are all 4 elements – nickel, cobalt, rhenium and aluminum, and the content of cobalt, rhenium and aluminum are very close among themselves.

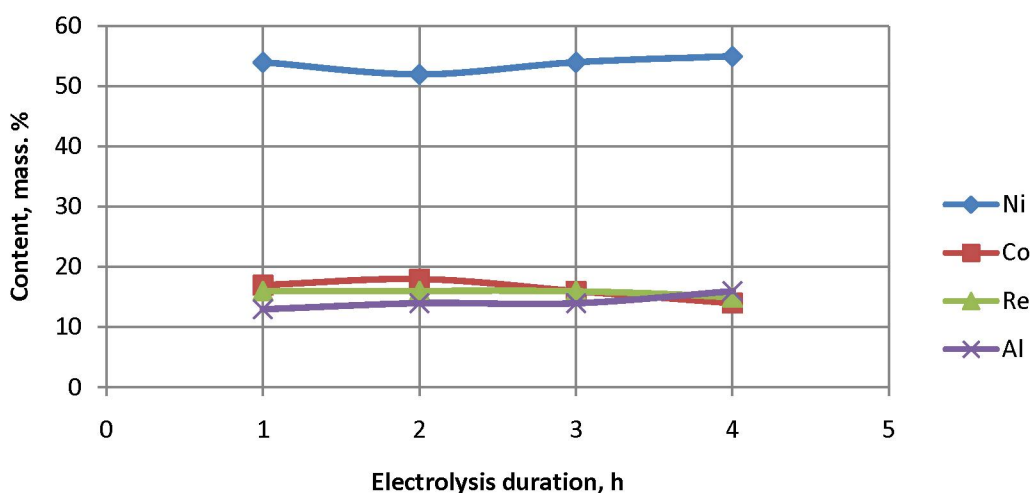


Figure 6 – Influence of duration of the electrolysis on composition of cathode deposit obtained by using nitrate electrolyte

The chemical analysis of a cathode deposit in case of electrolysis from nitrate electrolyte has allowed to define a ratio of components: Ni:Re:Co:Al = 3:1:1:1. The X-ray phase analysis of a cathode deposit has allowed to establish that the main phases are intermetallics of $Al_{14}Co_3Ni_3$ and AlRe.

Micrographs of the cathode deposit received at electrochemical processing of heat resisting alloy in nitrate electrolyte by concentration of 100 g/l are provided on figure 7 from where follows that the dendritic structure of a cathode deposit was more expressed, dendrites are branched strongly out, the fusion of the particles occurs along the lines of the syngony of the crystals formed and the dendritic "branch".

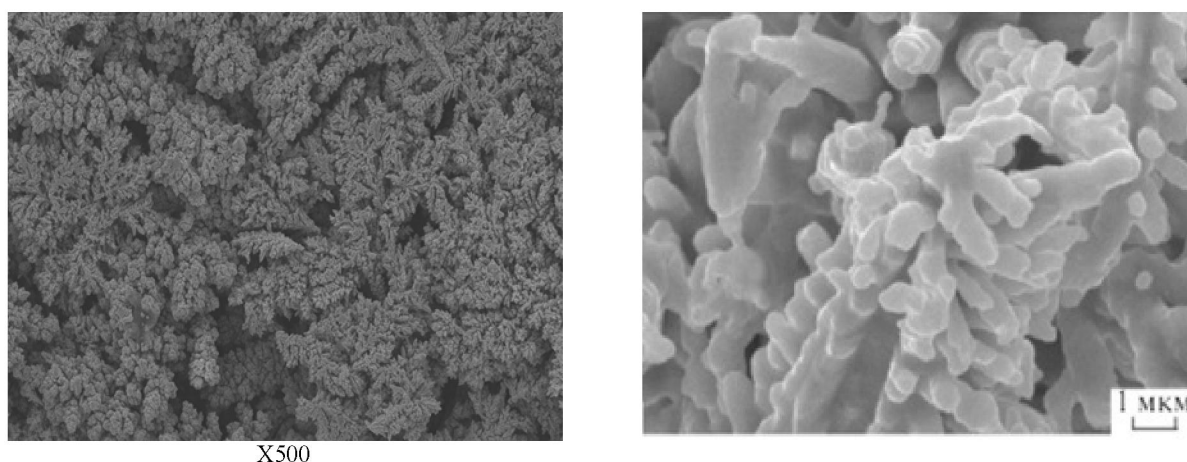


Figure 7 – Micrographs of a cathode deposit of the heat resisting GS32-VI alloy received at electrochemical processing in nitrate electrolyte

The particle size distribution of the cathode deposit obtained using the nitrate electrolyte is shown in figure 8, from which it is seen that 99% of grains cathode deposit are in the range from 0.040 to 0.2 μm [18].

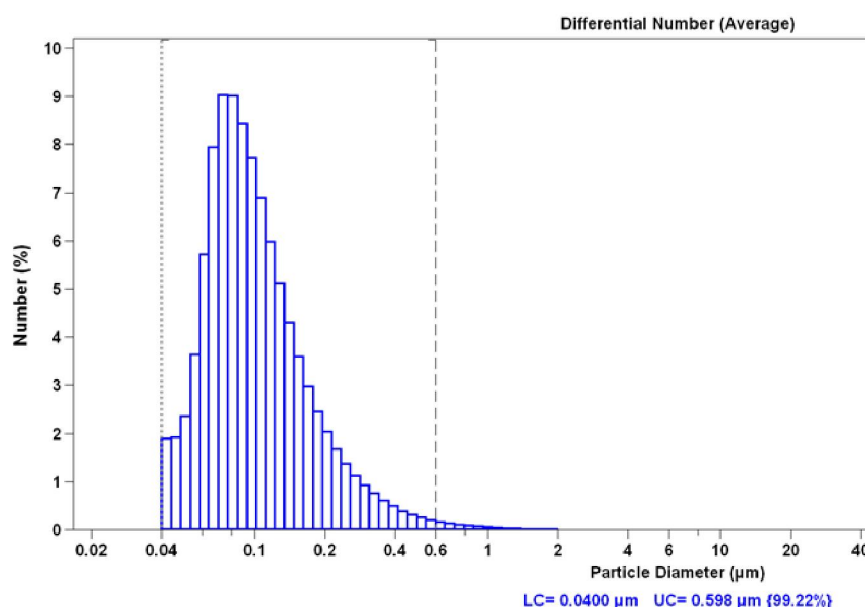


Figure 8 – The particle size distribution of the cathode deposit obtained using the nitrate electrolyte

Thus, in the electrochemical dissolution of the GS32-VI alloy in a nitrate electrolyte, a cathode deposit of a dendritic structure with a particle size of 0.04 to 0.2 μm is formed, which is a mixture of the following $Al_{14}Co_3Ni_3$ and AlRe compounds with a component ratio of Ni:Re:Co:Al = 3:1:1:1.

Conclusions:

1. It is established that depending on the choice of the nature of electrolyte at electrochemical processing of heat resisting GS32-VI alloy it is possible to receive cathode deposits of various chemical composition: from hydrochloric electrolyte – a deposit of structure of Ni:Co:Al = 4:1:1; from sulfate electrolyte – a deposit with the content of nickel about 95%; from nitrate electrolyte – a deposit of structure of Ni:Re:Co:Al = 3:1:1:1.

2. The nature of the electrolyte affects the morphology of the dendritic cathode deposit obtained - the development of the structure increases with the use of acid electrolytes with a concentration of 100 g/l in a row: $H_2SO_4 < HCl < HNO_3$.

3. The particle size distribution of cathode deposit also depends on the chosen electrolyte – the finest deposit (0,04-0,2 microns) turns out at electrochemical processing of GS32-VI alloy with use of nitrate electrolyte.

4. In general, the process of electrochemical dissolution of the heat-resistant alloy GS32-VI in the galvanostatic regime is most effective when using solutions of nitric acid as electrolyte.

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СВОЙСТВА КАТОДНЫХ ОСАДКОВ ПРИ ЭЛЕКТРОХИМИЧЕСКОЙ ПЕРЕРАБОТКЕ СПЛАВА ЖС32-ВИ С ИСПОЛЬЗОВАНИЕМ КИСЛОТНЫХ ЭЛЕКТРОЛИТОВ

Аннотация. В статье представлены результаты изучения состава, морфологии поверхности и гранулометрического анализа катодных осадков, полученных при электрохимической переработке отработанного жаропрочного сплава ЖС32-ВИ с использованием кислотных электролитов, анодное растворение которого проводили с использованием электрохимического технологического комплекса ЭХК-1012 (разработан ООО ИП "Тетран", Российская Федерация).

Исследования свойств катодных осадков проводились с использованием следующих методов исследований, анализа и приборного обеспечения: элементный анализ проводили с использованием ICP масс-спектрометра для изотопного и элементного анализа ELAN DRC-e (PerkinElmer, Канада); рентгенофазовый анализ – с использованием дифрактометра Shimadzu XDR 6000 (Япония); определение размеров частиц методом измерения динамического светорассеяния – на анализаторе размера субмикронных частиц и дзета-потенциала Delsa™ Nano, PN A54412AA; морфологию поверхности осадков изучали с использованием метода растровой и сканирующей электронной микроскопии (РЭМ и СЭМ) на электронном микроскопе JEOL JSM 6390 L.

Показано, что в зависимости от природы электролита могут быть получены катодные осадки различного химического, фазового состава и отличающиеся между собой по крупности и морфологии поверхности. В частности, можно получить катодные осадки различного химического состава: из сернокислого электролита - порошок с содержанием никеля около 95 %; из солянокислого электролита - порошок состава Ni:Co:Al = 4:1:1; из азотнокислого электролита - порошок состава Ni:Re:Co:Al = 3:1:1:1. Все полученные катодные осадки имели дендритную структуру, развитость которой также зависела от природы электролита, причем наиболее развитой структурой и наименьшим размером частиц обладали осадки, полученные при использовании азотнокислого электролита.

Ключевые слова. электрохимическая переработка, жаропрочный никелевый сплав, кислотный электролит, катодный осадок, морфология, дендритность, гранулометрический состав.

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ҚЫШҚЫЛ ЭЛЕКТРОЛИТТЕРДІ ҚОЛДАНУМЕН ЖС32-ВИ ҚОРЫТПАСЫН ЭЛЕКТРОХИМИЯЛЫҚ ӨНДЕУ КЕЗІНДЕ КАТОД ТҮНБАЛАРЫНЫҢ ҚАСИЕТТЕРІ

Аннотация. Мақалада анодты ерітуді ЭХК-1012 ("Тетран", Ресей) электрохимиялық комплекс көмегімен қышқыл электролиттерді қолданумен ЖС32-ВИ қайталама қызуға берік қорытпасын электрохимиялық өндеу кезінде алынған катод тұнбаларының құрамын, беттік морфологиясын және гранулометриялық талдау нәтижелері көрсетілген.

Катод тұнбаларының қасиеттері келесі талдау әдістерінің көмегімен жүзеге асырылды: элементтік талдауды изотопты және элементті анализге арналған ICP масс-спектрометрмен ELAN DRC-e (PerkinElmer, Канада); рентгенфазалық анализ – Shimadzu XDR 6000 (Жапония) дифрактометрмен; бөлшектердің мөлшері – субмикронды бөлшектердің мөлшерін және дзета-потенциалды талдауға арналған Delsa™ Nano, PN A54412AA; тұнбалар бетінің морфологиясын растрлық және сканерлеуші электронды микроскопия (РЭМ и СЭМ) JEOL JSM 6390 L электрондық микроскопкемеімен.

Электролит табиғатына тәуелді әр түрлі химиялық, фазалық құрамды және өзара ірілігі мен беттік морфологиясы жағынан айрықша катодты тұнбаларды алуға болады. Соның ішінде, түрлі химиялық құрамды катодты тұнбаларды алуға болады: күкіртқышқылды электролиттерден – никель бойынша құрамы 95 % ұнтақ; тұзқышқылды электролиттерден – Ni:Co:Al = 4:1:1 құрамды ұнтақ; азотқышқылды электролиттерден – Ni:Re:Co:Al = 3:1:1:1 құрамды ұнтақ. Барлық алынған катодты тұнбалар дендритті құрылымды болды, оның жетілгендігі де электролит табиғатына тәуелді, бұған қоса ең жетілген құрылымды және бөлшек мөлшері ең кіші тұнбалар азотқышқылды электролиттерді қолдану кезінде алынды.

Түйін сөздер: электрохимиялық өндеу, никель құрамды қызуға берік қорытпасы, қышқылды электролит, катодты тұнба, морфология, дендриттік, гранулометриялық құрамы.

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