

**REPUBLIC OF AZERBAIJAN**

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**ABSTRACT**

of the dissertation for the degree of Doctor of Philosophy

**ELECTROCHEMICAL EXTRACTION OF THIN  
LAYERS OF RHENIUM TRIPLE ALLOYS**

Specialty: 2307.01 - **Physical Chemistry**

Field of science: **Chemistry**

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The work was performed at the laboratory "Electrochemistry and electrocatalysis of rhenium alloys" at the Institute of Catalysis and Inorganic Chemistry named after academician M. Nagiyev of the Ministry of Science and Education.

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## GENERAL CHARACTERISTICS OF THE WORK

**Relevance of the topic and degree of development.** Nanotechnology, one of the current trends in science, has entered a major stage of development. The development of this science field creates ample opportunities for scientists to conduct new fundamental scientific-research works.

The development of new methods for the production of inorganic substances with unique properties is the basis of modern material science. Chalcogen-containing semiconductor layers are widely used in semiconductor technology. Depending on the chemical and stoichiometric composition of rhenium ternary alloys, they show very interesting properties (photoconductivity, optoelectronics, semiconductors). However, chemical and electrochemical methods are used to obtain these semiconductor coatings. The advantage of both chemical and electrochemical methods is that in both cases the process is carried out at low temperatures and is free from evaporation at high temperatures. The electrochemical method has its own advantages. Thus, it is possible to carry out the electrolysis process accurately by electrochemical method, to adjust the thickness of the obtained layer. There is a wide circle method for obtaining these substances, among which the electrochemical method occupies one of the main places. It is known that rhenium has alloys with various metals, which also have a number of valuable properties: corrosion resistance, specific magnetic and mechanical properties, they are used as catalysts. On the other hand, the electrochemical production of these alloys from different electrolytes has many specific properties that affect the properties of the obtained substances. First of all, it is due to the fact that the amount of non-metallic compounds (oxygen, hydrogen, carbon, etc.) in the alloys obtained during the electrolysis process is very small. Therefore, it is of great scientific and practical importance to obtain thin layers of solutions of two or more metals with the required composition with semiconductor properties. There are alloys of various metals with semiconducting properties, which can be obtained by electrolysis using solutions of rhenium and related metal cations.

It is possible to obtain ternary alloys of rhenium with different composition by changing the concentration, temperature and pH of rhenium and metals in the solution. There is almost no information in the literature on the production of ternary alloys of rhenium based on chalcogen. For the first time, in the dissertation work, the process of obtaining thin layers of ternary alloys based on rhenium selenium was studied.

**Object and subject of research.** The main regularities of the process of obtaining nanolayers of rhenium-selenium-copper alloys with semiconducting properties and the acquisition of layers to be used in the technology.

**Goals and objectives of the study.** It consists of obtaining nano-layers in the rhenium-selenium-copper system with semiconductor properties by electrochemical precipitation and studying the physical and chemical properties of the obtained layers. Scientific research was carried out in the following stages:

- Study of the mechanism of electrolytic precipitation of rhenium, selenium, copper both separately and together
- Study of the influence of various factors in the production of rhenium-selenium-copper alloys
- Investigation of physical and chemical properties of thin layers of alloys obtained in the rhenium-selenium-copper system

**Research methods.** X-ray phase analysis of thin layers of Re–Se–Cu alloys was carried out in  $\text{CuK}\alpha$ -irradiation on a dusty “D2 Phazer” diffractometer of the German company “**Bruker Corporation**”, morphology of the obtained alloys was performed by SEM in JEOL JSM7600F scanning electron microscope, element analysis by detector OXFORD X-MAX 50. The phase composition of the surface structure of the sediments was determined with a Tesla BS-301 electron microscope and a «Cotece» M-46 microprobe. Magnetic properties were studied in an EPR device. Thermal resistance and oxidation of thin films were programmed by thermogravimetric analysis on a NETZCH STA 449F3A-0835 derivatograph in a temperature range of 20–900° C for 10° C/min. A computer-equipped IVIUMSTAT potentiostat was used to study the electrochemical process.

### **The general provisions of the defense.**

– For the first time, the process of electrolytic deposition of ternary alloys Re–Se–Cu from sulfate solutions by electrochemical way was carried out by potentiostatic, temperature-kinetic and voltamperometric methods. It was found that the process of electrolytic deposition of rhenium from sulfate solutions is mainly characterized by the concentration polarization and in given case the rate of the cathode process is characterized by the diffusion of ions on the surface of the cathode.

– As a result of the study of the process of rhenium precipitation from rhenium sulfate electrolytes, it was determined that the reduction of rhenium is mainly due to the formation of intermediates or rhenium oxides ( $\text{Re}_2\text{O}_5$ ,  $\text{ReO}_3$ ) and the process of electrolytic deposition of rhenium from sulfate solutions is mainly characterized by the concentration polarization.

– The process of electrolytic precipitation of selenium from sulfate solutions was studied, the effect of various factors on the precipitation process was studied and it was determined that the rate of electrochemical deposition of selenium depends on lighting and temperature.

– The study of the potentiostatic and cyclic voltammetric characteristics of the process of joint electrolytic deposition of rhenium and selenium from sulfate solutions shows that depolarization of both elements is observed during the joint electrolytic deposition of rhenium and selenium at the cathode. This is due to the chemical interaction between rhenium and selenium at the cathode, the formation of a chemical compound.

– The influence of various factors (current density, temperature, acidity, component density, etc.) on the composition of Re–Se–Cu ternary alloys was studied. It was found that with increasing current density, temperature and rhenium concentration, the quality of the layers improves, and the amount of rhenium in the alloy increases. Optimal conditions and electrolyte composition for the production of thin layers with the semiconductor properties have been proposed.

– Oxidation and thermal resistance of thin layers obtained in

the rhenium-selenium-copper system were studied. Complete oxidation of the electrochemically obtained Re–Se–Cu alloy is characterized by 3 exothermic and 2 endothermic reactions. Oxidation of the alloy is a complex mechanism.

– The SEM analysis of thin films samples obtained in the Re–Se–Cu system on a copper electrode at a potential of + 0.5V for 1800 seconds at 343 K was studied and it was determined that its dimensions varied in the range of 50-130 nm. In this case, the average size of spherical particles of different geometric shapes on the surface of the platinum electrode are ~ 20-25 nm.

The magnetic properties of thin layers of rhenium-selenium-copper alloys obtained by electrochemical methods from sulfate solutions were studied. It has been established that the thin layers obtained in the rhenium-selenium-copper system have paramagnetic properties. Al–(Re–Se–Cu) type diode structure was created on the basis of layers obtained in rhenium-selenium-copper systems, its dynamic and static voltamper characteristics were studied and it was found that these diode structures have “transformation and memory” effect.

**Scientific novelty of the research.** For the first time, using modern electrochemical research methods, the mechanism of deposition of rhenium, selenium and copper from their sulfate solutions was studied separately and together (potentiostatic, temperature-kinetic and voltammetric methods). It was determined that the process of electrolytic deposition of rhenium from sulfate solutions is mainly characterized by concentration polarization and diffusion of ions on the surface of the cathode.

The process of electrolytic deposition of selenium from sulfate solutions was studied and the nature of polarization was determined. Selenium deposition is characterized first by chemical and then by diffusion polarization. The process of electrolytic deposition of selenium is regulated by the reduction of divalent selenium particles formed in the electrolyte.

The study of the potentiostatic and cyclic voltammeter characteristics of the electrolytic co-deposition of rhenium and selenium from sulfate solutions shows that during the electrolytic co-deposition

of rhenium and selenium in the cathode, a new chemical compound based on rhenium is observed. This is due to the chemical interaction between rhenium and selenium in the cathode.

### **Theoretical and practical significance of the research.**

As a result of the research, it is possible to obtain a thin layer of rhenium ternary alloy by electrolysis in a short time, at low temperature and on various metals in the required composition, which can be used in semiconductor technology. It is possible to change the electrolyte composition during electrolysis to obtain high-quality alloys that meet the requirements of modern technology.

A number of physical and chemical properties of thin layers of rhenium-selenium-copper alloys obtained by electrochemical methods have been studied.

**Approbation and application.** The main content of the dissertation is reflected in 29 scientific works and 2 patents. 14 of them are articles (10 abroad), 15 theses. The main results of the dissertation work were reported at the following International and Republican scientific conferences: Materials of the conference dedicated to the 75<sup>th</sup> anniversary of the corresponding member of ANAS H.S.Mammadov (Baku 2002), Scientific conference dedicated to the 95<sup>th</sup> anniversary of Academician M.F.Nagiyev (Baku 2003), materials of the conference dedicated to the 90<sup>th</sup> anniversary of the corresponding member of ANAS Z.H.Zulfugarov (Baku 2004), Materials of the republican conference "Organic reagents in analytical chemistry" dedicated to the 95<sup>th</sup> anniversary of Prof. A.A.Verdizade (Baku, 2009), "Modern methods in theoretical and experimental electrochemistry" II, VI, VII, IX, X, XI International scientific conference, (Ivanovo 2010, 2014, 2015, 2017, 2019, 2020), "Eurasian Symposium on Innovations in Catalysis and Electrochemistry" dedicated to the 100<sup>th</sup> anniversary of Acad. D.V.Sokolsky, (Kazakhstan, Almaty 2010), FGUP Gintsvetmet Institute Rhenium. Scientific Research, Process Developments. Practical Applications: Transactions of the International Conference (Moscow 2013, 2016), Proceedings of the Republican Scientific Conference dedicated to the 80<sup>th</sup> anniversary of the Institute of Catalysis and Inorganic Chemistry named after M. Nagiyev (Baku 2016), International conference "Nagiyev readings" dedicated to the

110<sup>th</sup> anniversary of Academician M.Nagiyev (Baku 2018), XXI Mendeleev Congress of General and Applied Chemistry (St. Petersburg 2019)

**Name of the organization where the dissertation is performed.**

The dissertation work was carried out in the laboratory "Electrochemistry and electrocatalysis of rhenium alloys" of the Institute of Catalysis and Inorganic Chemistry named after M. Nagiyev of ANAS.

**The total volume of the dissertation with a sign, indicating the volume of the structural units of the dissertation separately.**

The dissertation consists of an introduction (10648 signs), 4 chapters (Chapter I–70625 signs, Chapter II–9175 signs, Chapter III– 48378 signs, Chapter IV–40925 signs) and general results (2256 signs). The volume of the dissertation consists of 159 pages of computer text. There are 56 figures, 9 tables and 142 references. There are 182007 marks in the dissertation.

**Researcher's personal contribution.** The applicant fully participated in the implementation of the dissertation, the solution of the issues. Recently, he has been directly involved in summarizing the work done in this field in the literature review, setting up electrolysis, explaining the mechanism of the process by drawing polarization curves, summarizing the emerging ideas and results, and writing articles.

## MAIN CONTENT OF THE WORK

The **introductory part** reflects the relevance of the dissertation topic, the main purpose of the work, scientific novelty, and practical significance.

The **first chapter** provides a comparative analysis of the scientific literature on the dissertation. In the literature review, the properties of rhenium ternary alloys, the main regularities of the process of electrolytic precipitation from different electrolytes were discussed.

**In the second chapter** - the chemical reagents used in the work, the rules of preparation of solutions and electrodes, methods of analysis of the obtained sediments, methods of drawing polarization curves, methods for determining the physical and chemical properties of rhenium and Re–Se–Cu alloys are given. Reagents required for



research:  $\text{SeO}_2$  (k.t.),  $\text{CuCl}_2$ ,  $\text{KReO}_4$  (k.t.),  $\text{NH}_4\text{ReO}_4$  (k.t.)  $\text{H}_2\text{SO}_4$  (k.t.). The alloy is obtained using a platinum and copper electrode with an area of  $4 \text{ cm}^2$  as a cathode. Polarization curves are drawn using a platinum electrode with an area of  $0.15 \text{ cm}^2$ . Before use, the electrodes are polished, degreased and washed in 30% nitric acid. To draw the polarization curves, the surface was precipitated from suitable solutions using a platinum electrode coated with selenium, rhenium and rhenium-selenium. Experiments were performed using a silver-chloride electrode as a reference electrode and a platinum wire as an auxiliary electrode.

The kinetics of the combined electrolytic deposition of rhenium, selenium and copper was studied using potentiostatic and cyclic voltamperometric methods. Cyclic voltamperometric curves were plotted on the P-5827M potentiostat, and potentiostatic curves were plotted on the P-5827M potentiostat using the KSP-4 potentiometer using the PDP4-002 recorder. The kinetics and mechanism of the process of electrochemical obtaining of Re-Se-Cu thin films using the computer-equipped IVIUMSTAT electrochemical analyzer potentiostat were studied. Polarization curves were drawn with the help of a glass electrolyzer with a volume of 50 ml, covered with a glass shirt. The temperature was regulated with an accuracy of  $\pm 0.1^\circ\text{C}$  using a thermostat U-10. With the help of a copper coulometer, the current yield was determined by the weight method and calculated taking into account the composition of the sediment.

Phase composition of the surface structure of rhenium and its chalcogenic alloys was determined with a Tesla BS-301 electron microscope and a «Cotece» M-46 microprobe. The porosity of the coatings was determined by scratching the surface and bending the electrode at an angle of  $90^\circ\text{C}$ , and the adhesion of the sediments to the cathode surface was determined by checking with filter paper. The analysis of the composition of the Re-Se-Cu alloy is as follows: the electrochemically obtained precipitate at the cathode is dissolved by heating in 10 ml of full-strength nitric acid, after evaporation several times in a water bath, 5N  $\text{H}_3\text{PO}_4$  is added to the solution. To separate Rhenium from selenium, it extracted with isoamyl alcohol.

Se and Re were determined separately on the SPECORD-50 PLUS device by the urea method.

Copper determination in rhenium-selenium-copper system was carried out on AA 280 FS Fast Sequential Atomic Absorption Spectrometer - Varian device. X-ray studies of thin layers of alloys obtained in the Re-Se-Cu system were carried out on the UDR-50 UM diffractometer in the chamber RKD-57,3 in  $\text{CuK}\alpha$ -radiation. The thickness of thin layers of Re-Se-Cu alloys was calculated in the microscope MII-4 by the method of interferon. The morphology of alloys obtained in the Re-Se-Cu system by electrochemical SEM was studied.

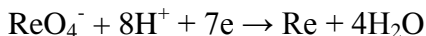
The surface of the electrode was examined at various magnifications in the JEOL JSM7600F scanning electron microscope to determine the joint deposition, as well as the corresponding element analysis by means of the detector OXFORD X-MAX 50. The sample was scanned at an average electronic voltage of  $\sim 15$  kEv. Thermal resistance and oxidation of thin layers obtained by electrochemical sulphate solutions in the Re – Se – Cu system were studied by thermogravimetric analysis. The thermogram is programmed on a NETZCH STA 449F3A-0835 derivatograph in a temperature range of 20–900°C for 10°C/min. The magnetic properties of nano-layers obtained in the Re – Se – Cu system were studied in an EPR device.

**The third chapter**-presents the experimental results of electrochemical precipitation of rhenium-selenium-copper alloys from sulfate solution

In order to study the process of joint electrolytic precipitation of rhenium, selenium and copper, it is first necessary to study the process of precipitation of these metals separately from the given electrolytes. For this purpose, the kinetics and mechanism of the process of precipitation of rhenium, selenium and copper from given solutions have been studied by potentialstatic, voltammetric methods [4, 12].

The main electrolyte for the electrolytic precipitation of rhenium from acidic solutions is sulfate solution. It is known that rhenium sulfate occurs in the form of perrenate ions ( $\text{ReO}_4^-$ ) in solutions, and its

reduction takes place in several stages. The final equation of the process is as follows:

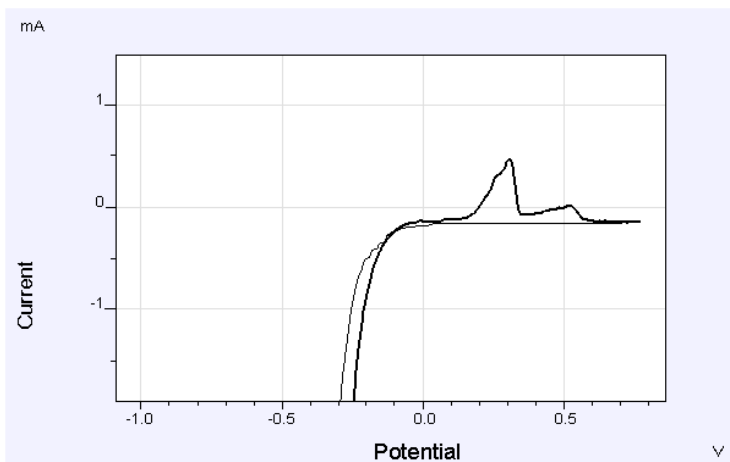


It is known from the literature and our research that the reduction of rhenium in sulfate solutions consists of several stages. The effect of rhenium concentration, temperature, acidity, electrode material in the electrolyte on the electrolytic precipitation of rhenium from sulfate solutions was studied [7]. A wave is observed in the polarization curves, which confirms the gradual reduction of rhenium. It is likely that  $\text{Re}_2\text{O}_5$  and  $\text{Re}_2\text{O}_3$  oxides are obtained during the reduction of rhenium [16]. Electrolytic precipitation of rhenium from sulfate solutions begins at a potential of + 0.3 V, and thin black layers of rhenium are obtained on the electrode.

Polarization curves of rhenium at different acids were plotted and it was found that as the acidity of the solution increases, the reduction of rhenium becomes more difficult and the rhenium collapses with high polarization. It was found that by increasing the pH in the solution, the process of electrolytic deposition of rhenium from the sulfate electrolyte slows down. The increase in pH slows down the process of electrolytic deposition of rhenium by the fact that at different pH values in the solution, intermediate products are formed on the surface of the electrode, which passivate the surface of the electrode and cause the potential to shift negatively.

At the same time, polarization curves are drawn at different values of potential change, and two accurate waves are observed in the cathode polarization curve. One of them has a potential of 0.2-0.3 V, and the other has a potential of (-0.3)-(-0.4) volts. The existence of these waves, can be explained by the stepwise mechanism of perrenat- ions reduction. This is also confirmed by the anode polarization curves. The potential of 0.1 - 0.2 V in the anode wave can be attributed to the dissolution of rhenium, which is a bright expression of the dissolution of  $\text{ReO}_3$  and  $\text{ReO}_2$ .

Figure 1 shows voltamperometric cyclic polarization curves from rhenium sulfate solutions on a platinum electrode.



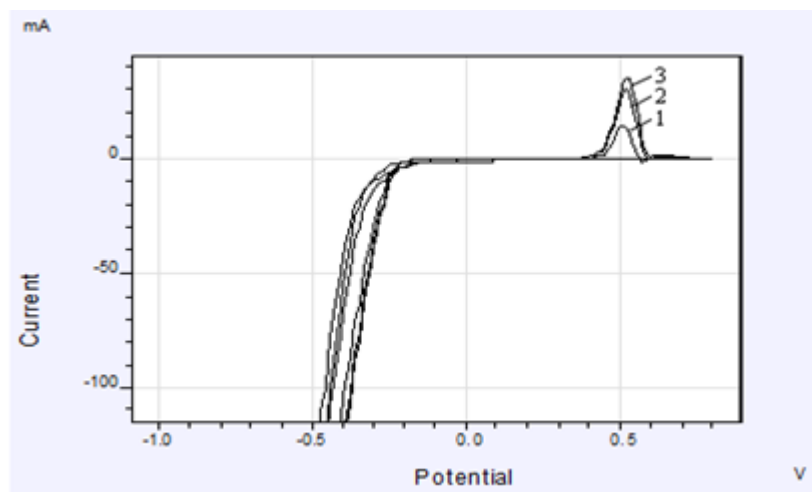
**Figure 1. Voltamperometric cyclic polarization curves from rhenium sulfate solutions on a platinum electrode. Electrolyte content mol/l:  $3,5 \cdot 10^{-3}$   $\text{KReO}_4 + 2,0 \text{ H}_2\text{SO}_4$ ;  $t=75^\circ\text{C}$ ;  $\text{pH}=0,4$  ;  $V=0,005$  v/s.**

When looking at the voltamperometric cyclic polarization curves of rhenium sulfate solutions on the rhenium electrode, the maximum is observed in the ice polarization curves, which refers to the reduction of rhenium. The reduction of rhenium in a given electrolyte begins at 0.15 volts, and the reduction of perrenate ions to metallic rhenium is gradual.

By electrolysis on a platinum electrode, it was studied that the anode polarization curve shows that the rhenium is the anode solution, and the cathode polarization curve shows the cathode deposition of rhenium on the electrode. Deposition and anode dissolution processes occur at different potentials depending on the concentration of rhenium in the solution [6]. The oxidation current of rhenium in acidic solutions begins at a potential of -0.5 V, resulting in a sharp maximum at the voltammeter curves.

Figure 2 shows the voltamperometric cyclic polarization curves from sulfate solutions of different concentrations of rhenium on a platinum electrode. As can be seen from the curves, depending on the concentration of rhenium, the height of the wave generated in the volt-ampere curves increases. This confirms that the process of precipitation

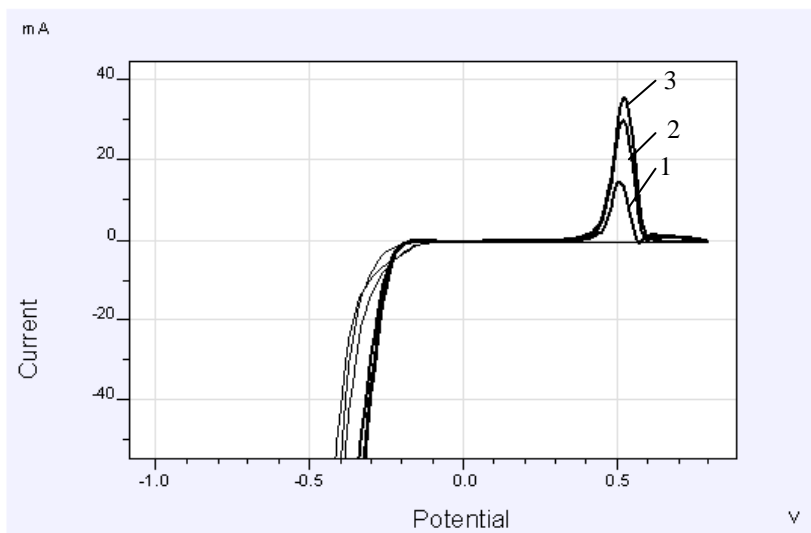
and anode dissolution takes place at different potentials and different current densities, depending on the concentration of rhenium.



**Figure 2. Voltamperometric cyclic polarization curves of rhenium sulfate solutions of different concentrations on a platinum electrode. Electrolyte content mol / l: 1– $3,5 \cdot 10^{-3}$   $\text{KReO}_4 + 2,0 \text{ H}_2\text{SO}_4$ ; 2– $5,0 \cdot 10^{-3}$   $\text{KReO}_4 + 2,0 \text{ H}_2\text{SO}_4$ ; 3– $8,0 \cdot 10^{-3}$   $\text{KReO}_4 + 2,0 \text{ H}_2\text{SO}_4$ ;  $t=75^\circ\text{C}$ ;  $\text{pH}=0,4$ ;  $V=0,005 \text{ v/s}$**

The influence of various factors: current density, temperature, pH of the solution, concentration of rhenium in solution, sulfuric acid, concentration of ammonium sulfate, various electrodes was studied to obtain better and thinner layers of rhenium from sulfate electrolyte.

One of the main factors influencing the electrolytic precipitation of rhenium from acid solutions is temperature. Figure 3 shows the voltamperometric cyclic polarization curves from rhenium sulfate solutions on a platinum electrode at different temperatures. At this time, the temperature changed in the range of  $25\text{--}90^\circ\text{C}$ . As can be seen from the polarization curves, as the temperature increases, the height of the rhenium anode curve increases and rhenium becomes easier to dissolve.



**Figure 3. Voltamperometric cyclic polarization curves from rhenium sulfate solutions on a platinum electrode at different temperatures. Electrolyte content mol / l:  $3,5 \cdot 10^{-3}$   $KReO_4 + 2,0 H_2SO_4$ ; pH=0,4;  $V=0,005$  v/s Temperature ( $^{\circ}C$ ), 1–25, 2–45, 3–75.**

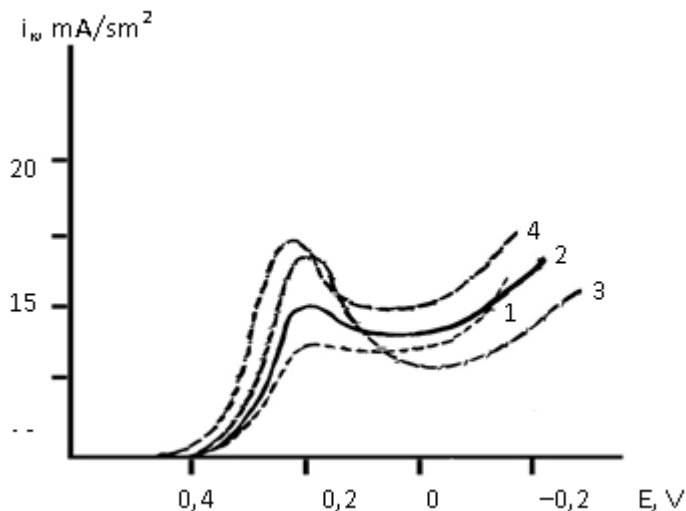
Long-term studies have shown that the process of electrolytic precipitation of rhenium from acidic solutions, the quality and appearance of the layer is highly dependent on temperature.

Temperature-kinetic method was used to clarify the mechanism of electrolytic precipitation of rhenium from sulfate solutions. Using this method, the nature of polarization in the cathode process was determined.

At different constant values of the cathode potential, a graph of  $\lg i_k - 1/T$  is constructed, the effective activation energy of the electrode process is calculated based on a straight line dependence on the constant potential of the cathode. It was found that at low values of potential, precipitation from rhenium sulfate solution is observed by chemical polarization. At later potential values, the precipitation of rhenium is accompanied by mixed polarization.

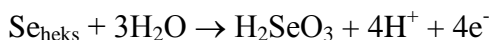
The appearance and quality of rhenium vary depending on the current density. In this case, depending on the current density in the

solution (current density 5–20 mA/cm<sup>2</sup>), the current output of rhenium varies in the range of 20-45% [14]. The electrolyte proposed below and the optimal electrolysis conditions were used to obtain thin layers of rhenium from sulfate solutions. The composition of the electrolyte is given below (mol / l):  $5 \cdot 10^{-3}$  KReO<sub>4</sub> + 2,0H<sub>2</sub>SO<sub>4</sub>,  $i_k=1-20$  mA/sm<sup>2</sup>,  $t=75^{\circ}\text{C}$ ,  $\text{pH}=0,4$

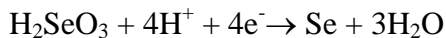


**Figure 4. Polarization curves of selenium on a platinum electrode at different temperatures (<sup>0</sup>C): 1 - 25; 2 - 45; 3 - 65; 4 - 75°C.**

The electrochemical production of selenium and its coatings is promising and of great scientific interest. The semiconducting properties of selenium and the mechanism of deposition depend on the composition of the electrolyte, the conditions of electrolysis, the condition of the electrode surface, temperature and current density [4]. The standard potential of selenium is 0.74 V:



The most suitable electrolyte for the deposition of selenium from an acidic environment is sulfuric acid. Electrolytic precipitation of selenium from selenite acid in any modification goes on this reaction:



The effect of selenium concentration, temperature and potential change rate in the electrolyte on the course of polarization curves was studied. As can be seen from Figure 4, the reduction of selenium from the acid electrolyte begins at a potential of +0.36 V, and a wave of +0.25 V is observed in the polarization curve, which is also observed during the deposition of selenium from an alkaline environment [1]. However, the difference is that the electrolytic deposition of selenium is more positive in an acidic environment than in an alkaline one. It is likely that the reduction of selenite ion in an acidic environment follows the following scheme: the first stage of the electrode process is determined by the formation of elemental selenium. In the second stage, divalent selenium is formed. In the third stage of the electrode process, hydrogen is formed.

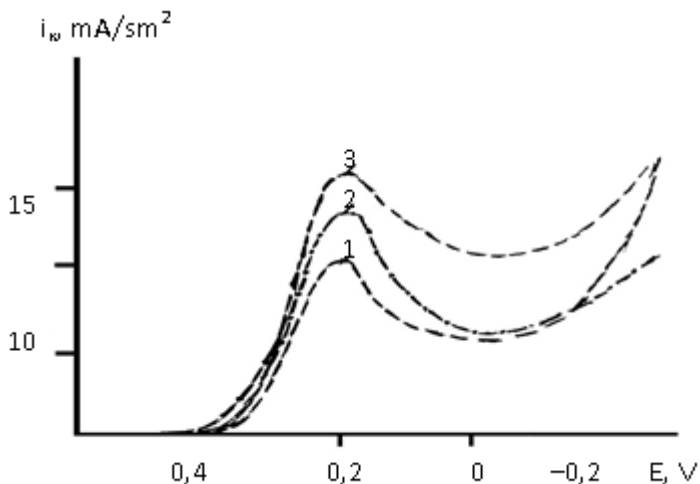
Temperature has a special effect on the electrolytic deposition and quality of selenium. Figure 4 shows the polarization curves of selenium at different temperatures. As can be seen from the figure, the reduction of  $\text{SeO}_3^{2-}$  ion becomes easier with increasing temperature, and red amorphous selenium is obtained on the cathode surface at a temperature of 25–45<sup>0</sup>C, and gray hexagonal selenium at a temperature of 45–75<sup>0</sup>C.

By increasing the concentration of selenium oxide in the solution, the rate of reduction of selenium at the cathode increases and the sedimentation potential shifts to the positive side. In this case, a wave is observed in the polarization curve at a potential of + 0.3 V, and an elemental selenium layer is obtained on the electrode surface (Figure 5). There is a direct relationship between the concentration of selenium in the solution and the limiting current. This indicates that as the limiting current is generated, the potential of the electrode shifts to the negative side as the amount of selenium in the area around the electrode decreases, and hydrogen is released during electrolysis.

By studying the effect of temperature on the process, it was determined that the amount of selenium in the cathode increases with increasing temperature, and a fine crystalline precipitate is obtained.



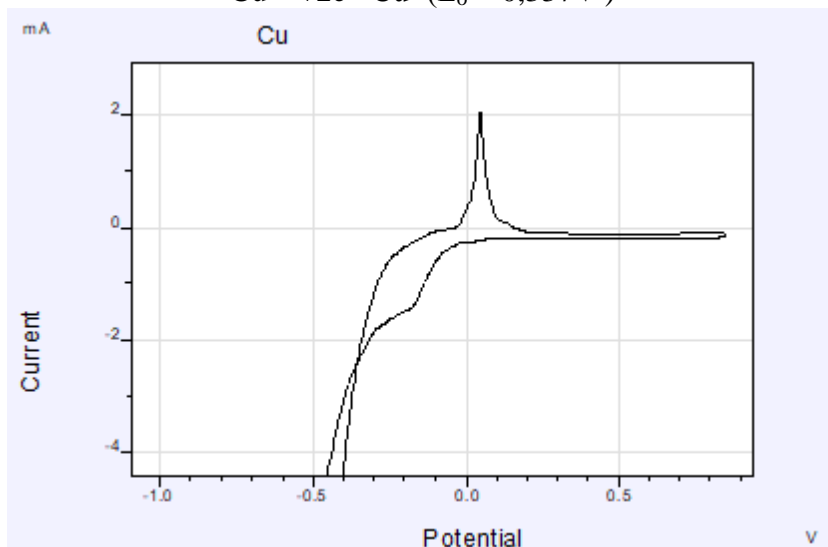
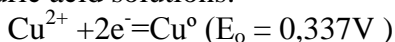
The construction of the  $\lg i_k - 1/T$  dependence curve is to determine the nature of the cathode potential. In this case, the activation energy was calculated based on the obtained linear dependence. The deposition of selenium from these solutions up to a potential of 0.2 V is mainly accompanied by chemical polarization, and from 0.35 V by mixed polarization, and the temperature has a significant effect on the speed of the process under study. When the temperature in the electrolyte is 20-90°C, a limiting current is observed in the polarization curves, the value of the limiting current varies with temperature and the rate of the reduction reaction of selenium increases. In this case, an increase in temperature accelerates the change in cathode potential and the electrolytic deposition of selenium to varying degrees.



**Figure 5. Polarization curves of selenium on a platinum electrode at different concentrations (mol / l): 1 - 0,25  $\text{SeO}_2 + 2,0\text{H}_2\text{SO}_4$  ; 2 - 0,35  $\text{SeO}_2 + 2,0\text{H}_2\text{SO}_4$ ; 3 - 0,40  $\text{SeO}_2 + 2,0\text{H}_2\text{SO}_4$ .  $t = 75^\circ\text{C}$ .**

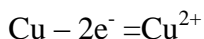
In order to study the Re-Cu-Se system electrochemically, the process of electrolytic deposition of copper from that electrolyte must be studied in detail. Therefore, the process of electrolytic deposition of copper from the given solutions was studied. Copper and its alloys (chalcogenides) are widely studied in the literature. The cath-

ode process of Cu (II) reduction on the Pt cathode from the sulfate solution was studied. It is known that copper is formed by reducing copper ions in sulfuric acid solutions:



**Figure 6. Cyclic polarization curves (mol / l) of a copper on a platinum electrode:  $6 \cdot 10^{-4} \text{CuCl}_2 \cdot 2\text{H}_2\text{O} + 2\text{H}_2\text{SO}_4$ ;  $t=75^\circ\text{C}$ ;  $V=0,005\text{VS}^{-1}$ ;  $\text{pH}=0,4$**

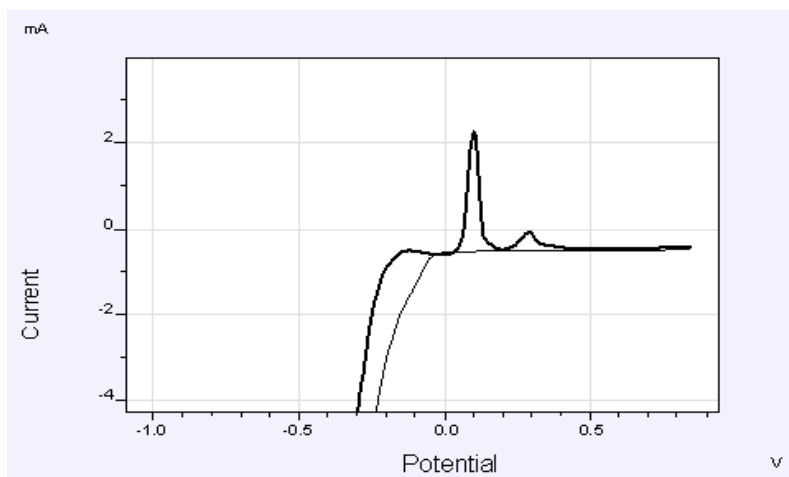
To study the process of electrolytic precipitation from copper sulfate solutions, we drew cyclic polarization curves. For this purpose, a copper anode curve was drawn, and these curves are given in Figure 6 [18]. A wave is interviewed in the copper anode curve (potential 0.1V), which is the copper oxidation curve.



However, it is known that for the joint electrolytic precipitation of Cu and Se, a solution of both metals is added to the solution and the precipitation process is studied (Figure 7).

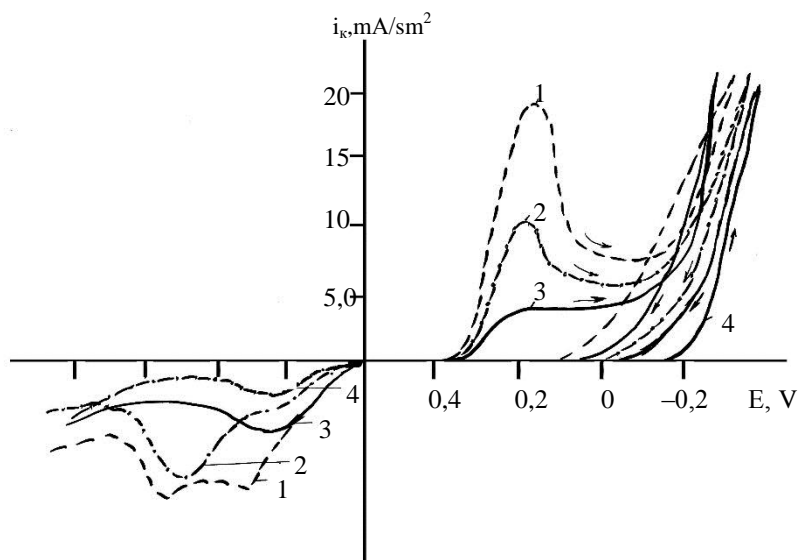
In order to study the process of joint electrolytic deposition of rhenium and selenium from sulfate electrolytes and to clarify the mechanism and kinetics of deposition, general polarization curves were drawn. Figure 8 shows these polarization curves. It is known

that the standard electrode potential of selenium in acid solutions is + 0.36V, and rhenium is + 0.74V. Polarization measurements show that the deposition of rhenium begins at (0.28) V in the absence of selenium, while the reduction of selenium begins at (0.32) V. The electrolytic precipitation of rhenium and selenium together begins at a more positive potential than the precipitation of these metals separately. This indicates that both elements are separated by depolarization, which confirms the formation of a chemical compound ( $\text{ReSe}_2$ ) or solid solution on the cathode.



**Figure 7. Voltammetric cyclic polarization curves of selenium and copper together on a platinum electrode, electrolyte composition (mol / l):  $6 \cdot 10^{-4} \text{CuCl}_2 \cdot 2\text{H}_2\text{O} + 9 \cdot 10^{-4} \text{SeO}_2 + 2\text{H}_2\text{SO}_4$ ,  $t=75^\circ\text{C}$ ;  $V=0,005\text{VS}^{-1}$ ;  $\text{pH}=0,38$**

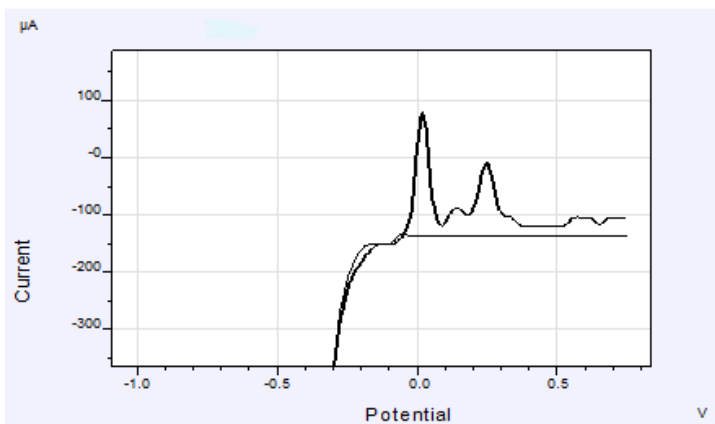
During electrolytic deposition with selenium, the potential of rhenium shifts to the positive side, which is due to the depolarization effect of the alloy between the components (Figure 8). The reason for the facilitation of the reduction of metal ions in the formation of alloys is the change in the free partial energy of the components of the alloy. Optimal electrolysis conditions for obtaining high-quality semiconductor Re–Se coatings from sulfate electrolytes were determined and the influence of various factors on the sedimentation process was studied.



**Figure 8. Cyclic polarization curves (mol / l) of selenium and rhenium on a platinum electrode:**

**1 –  $0,18\text{NH}_4\text{ReO}_4 + 0,07\text{SeO}_2 + 2,0\text{H}_2\text{SO}_4$ , 2 –  $0,07\text{SeO}_2 + 2,0\text{H}_2\text{SO}_4$ , 3 –  $0,18\text{NH}_4\text{ReO}_4 + 2,0\text{H}_2\text{SO}_4$ , 4 –  $2,0\text{H}_2\text{SO}_4$ , temperature -  $75^\circ\text{C}$ .**

The study of the electrolytic precipitation process of Re– Se alloys shows that the quality of the obtained coatings depends on the current density, temperature, acidity of the solution, the individual concentration of the components, the total concentration and the effect of complexing agents. Temperature also has an effect on the composition and quality of Re-Se alloy. It was found that the amount of rhenium in the alloy increases with increasing temperature in both electrolytes. Thin plate-shaped, high-quality sediments are obtained from sulfate electrolytes at  $75\text{--}80^\circ\text{C}$  [13]. At a temperature of  $95^\circ\text{C}$ , the alloy Re-Se obtained on the cathode contains amorphous selenium, which degrades the quality of the sediment. The effect of acidity of the solution and ammonium sulfate on the composition and quality of Re-Se alloys was studied [3]. It was found that as the concentration of sulfuric acid in the electrolyte increases, the amount of rhenium in the alloy first increases and then does not change.



**Figure 9. Voltammetric cyclic polarization curves on rhenium-selenium-copper alloy platinum electrode. Electrolyte composition (mol / l):  $6,9 \cdot 10^{-4} \text{KReO}_4 + 9 \cdot 10^{-4} \text{SeO}_2 + 6 \cdot 10^{-4} \text{CuCl}_2 \cdot 2\text{H}_2\text{O} + 2\text{H}_2\text{SO}_4$ ,  $t=75^\circ$ ;  $V=0,005\text{VS}^{-1}$ ;  $\text{pH}=0,1$**

In order to study the joint deposition in the Re-Cu-Se system, an electrolyte consisting of all three components was taken and polarization curves were drawn. Figure 9 shows the voltammetric polarization curves of the Re-Cu-Se joint precipitation process. When looking at the voltamperometric polarization curves of Re-Cu-Se, 3 waves are observed here compared to the Re-Se and Cu-Se anode curves. The following conclusions can be drawn from the obtained anode curves: The first wave observed in the drawn polarization curves can be attributed to the oxidation of Cu, the second wave observed in the polarization curve can be attributed to selenium oxidation, and the third wave can be attributed to the anode solution of the new ternary Re-Se-Cu alloy. The obtained wave proves that a ternary Re-Se-Cu alloy is obtained in the studied solution and at the determined potential. Morphology and composition analysis of electroplated thin layers of Re-Cu-Se were performed on a platinum electrode [25]. Based on the data presented in this paper, an initial model of the mechanism of electrolytic deposition of thin layers of Re-Se-Cu is proposed. Here are three main stages of electrochemical research:

Stage I – reduction and adsorption of selenium particles on the surface of the platinum electrode;

Stage II – interaction of  $\text{Cu}^{2+}$  ions with oxidized compounds of adsorbed selenium;

Stage III – Formation of Re–Se–Cu alloy and thin layers of Re–Se–Cu are formed by the interaction of  $\text{Cu}^{2+}$  and  $\text{Se}^{4+}$  ions with rhenium ions.

**Table 1. Dependence of the composition and quality of the rhenium-copper-selenium layer on the concentration of rhenium. Electrolyte composition (mol / l):  $2 \text{H}_2\text{SO}_4$ ,  $t= 75^\circ\text{C}$ ,  $i_k= 20 \text{ mA/cm}^2$ .**

Electrolyte, mol / l			$i_k$ , $\text{mA/cm}^2$	Composition of the alloy, %			Appearance of the layer
KReO <sub>4</sub>	CuCl <sub>2</sub>	SeO <sub>2</sub>		Re %	Se %	Cu%	
$3,5 \cdot 10^{-3}$	$1,5 \cdot 10^{-3}$	$2,6 \cdot 10^{-2}$	20	46	14	40	gray, lustreless
$4,6 \cdot 10^{-3}$	$1,5 \cdot 10^{-3}$	$2,6 \cdot 10^{-2}$	20	47	16	37	dark gray, lustreless
$5,0 \cdot 10^{-3}$	$1,5 \cdot 10^{-3}$	$2,6 \cdot 10^{-2}$	20	48	18	34	black gray, shiny
$5,4 \cdot 10^{-3}$	$1,5 \cdot 10^{-3}$	$2,6 \cdot 10^{-2}$	20	50	20	30	black, shiny
$6,5 \cdot 10^{-3}$	$1,5 \cdot 10^{-3}$	$2,6 \cdot 10^{-2}$	20	54	24	22	black, shiny

The influence of various factors on the composition of the obtained alloy was studied [29]. Thus, the electrolysis process depends on the concentration of components, temperature, pH of the solution, the rate of change of potential, the material of the electrode, etc. effects have been studied [26]. Table 1 shows the dependence of the composition of the Re–Se–Cu alloy on the concentration of rhenium in the electrolyte. As can be seen from the table, the concentration of rhenium in the solution affects the quality and properties of the rhenium-copper-selenium alloy. As the amount of rhenium in the solution and the current density increase, the amount of rhenium in the alloy increases.

One of the main factors influencing the composition of the alloy is temperature and current density. It was found that with an increase in current density from  $15 \text{ mA/cm}^2$  to  $20 \text{ mA/cm}^2$ , the amount of rhenium in

the alloy increases from 40% to 54%. Better quality alloys have a current density of 20 mA/cm<sup>2</sup>. Table 2 shows the temperature dependence of the composition of Re–Cu–Se alloy. It was found that better quality thin films are obtained at 75 °C, on the Pt and Ni electrodes.

**Table 2. Temperature dependence of the composition and quality of the rhenium-selenium-copper layer. Electrolyte content (mol / l): 2 H<sub>2</sub>SO<sub>4</sub>, t= 75°C, i<sub>k</sub>= 20 mA/cm<sup>2</sup>.**

Electrolyte, mol / l			temperature °C	Alloy composition, %			Appearance of the layer
KReO <sub>4</sub>	CuCl <sub>2</sub>	SeO <sub>2</sub>		Re %	Se %	Cu%	
6,5·10 <sup>-3</sup>	1,5·10 <sup>-3</sup>	2,6·10 <sup>-2</sup>	25	44	30	26	dark gray, lustreless
6,5·10 <sup>-3</sup>	1,5·10 <sup>-3</sup>	2,6·10 <sup>-2</sup>	45	47	29	24	dark gray, lustreless
6,5·10 <sup>-3</sup>	1,5·10 <sup>-3</sup>	2,6·10 <sup>-2</sup>	65	50	27	23	dark gray, shiny
6,5·10 <sup>-3</sup>	1,5·10 <sup>-3</sup>	2,6·10 <sup>-2</sup>	75	54	24	22	dark gray, shiny
6,5·10 <sup>-3</sup>	1,5·10 <sup>-3</sup>	2,6·10 <sup>-2</sup>	90	54	24	22	black, shiny

The microstructure of thin layers of rhenium-selenium-copper alloys obtained by electrochemical methods at different current densities was studied. During the study of electrolysis conditions, it was determined that the most suitable temperature for the production of thin layers of quality Re–Se–Cu coatings is 75-80<sup>0</sup>C. At this temperature, thin layers of black-gray, dense, shiny, small crystalline of Re–Se–Cu alloy are obtained on the surface of the cathode. As the temperature of the electrolyte increases, the quality and structure of the sediment also change. Thus, as a result of the conducted experiments, the following electrolyte and optimal regime were proposed for the electrochemical method of obtaining thin layers with semi-conducting properties from sulfate solutions on a platinum electrode in the rhenium-selenium-copper system.

The composition of the electrolyte is given below (mol / l):  
 $6,9 \cdot 10^{-4} \text{KReO}_4 + 9 \cdot 10^{-4} \text{SeO}_2 + 6 \cdot 10^{-4} \text{CuCl}_2 \cdot 2\text{H}_2\text{O} + 2,0 \text{H}_2\text{SO}_4$ ,  
 $i_k = 1-20 \text{mA/cm}^2$ ,  $t = 75^\circ \text{C}$ ,  $\text{pH} = 0,4$

**The fourth chapter** presents the physicochemical properties of rhenium-selenium-copper alloys: electrolyte dispersion, thermal resistance of the obtained layers, magnetic properties, voltammetric characteristics, and SEM analysis, X-ray phase analysis results are given.

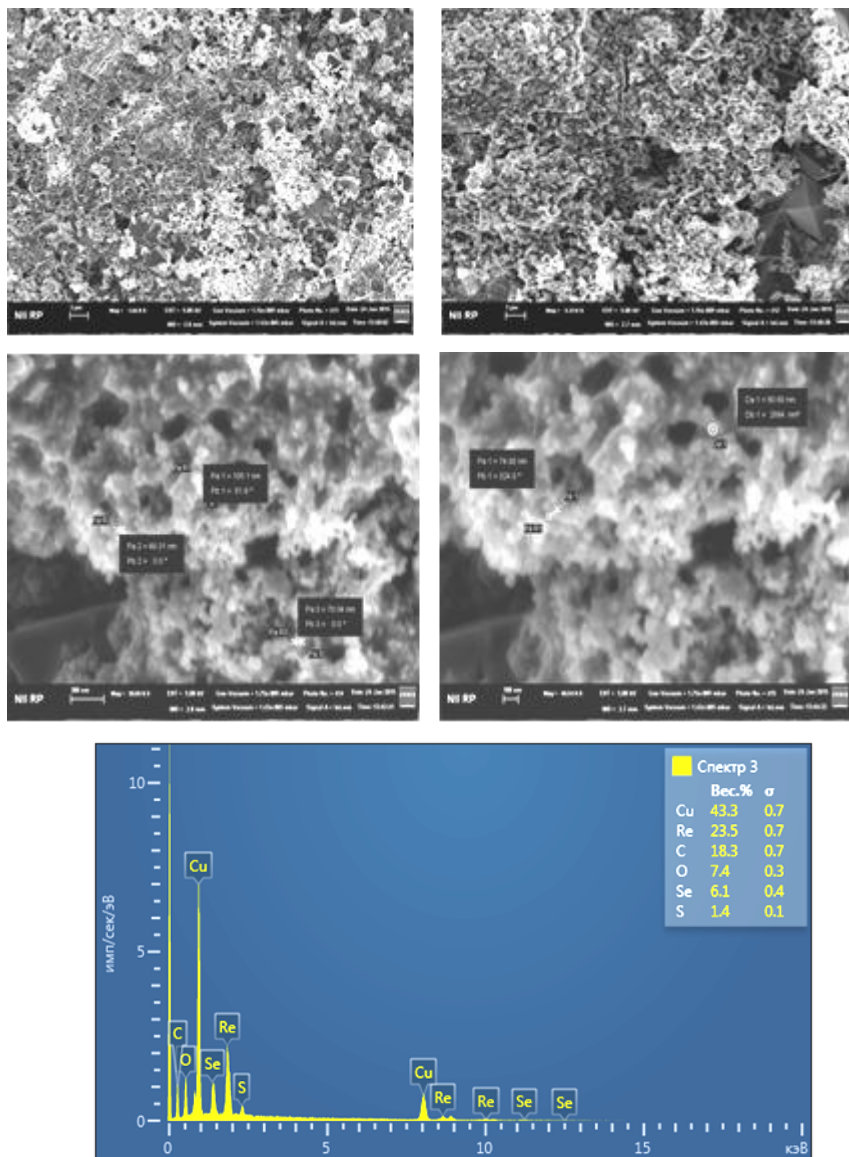
As is known, the microstructure, X-ray structure, thickness, type of conductivity and properties of alloys obtained by electrolysis vary depending on the mechanism of alloy formation.

The morphology of the thin layers obtained electrochemically in the Re–Se–Cu system was studied on various electrodes: copper, platinum and nickel electrodes. It was found that the size of the thin layers obtained in the Re–Se–Cu system varies depending on the material of the electrode. Thus, the layers obtained from the same electrolyte on different electrodes at the same current density differ in size and composition. Therefore, the size of the substances obtained on the platinum electrode varies from 18 to 22 nm, and the size of the substances obtained on the copper electrode varies from 50 to 60 nm.

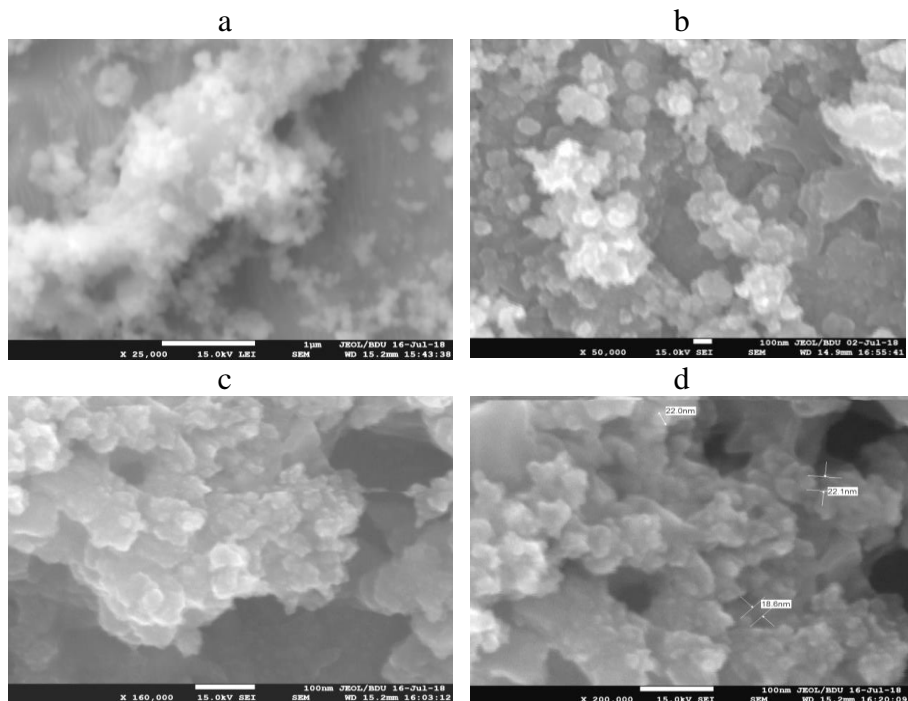
The results of the obtained experiments are given in Figure 10 (morphology of the obtained layers and elemental analysis of the composition of the obtained layers). As can be seen from the results of the element analysis, it was determined that the layers are homogeneous everywhere in terms of their composition. As a result of research, it was found that the morphological structure of the layers obtained on both platinum and nickel electrodes is the same.

To determine the formation of thin layers of Re–Se–Cu and the co-deposition of the main components on the platinum electrode, the surface of the electrode was examined at various magnifications in the JEOL JSM7600F scanning electron microscope as well as by element analysis. The sample was scanned at an average electronic voltage of  $\sim 15 \text{keV}$ . In a series of increments of 25,000 (a), 5,000 (b), 160,000 (c) and 200,000 (d), as shown in Figure 11, agglomerates with an average size of  $\sim 20-25 \text{nm}$  of spherical particles of various geometric shapes are observed on the surface of the electrode.



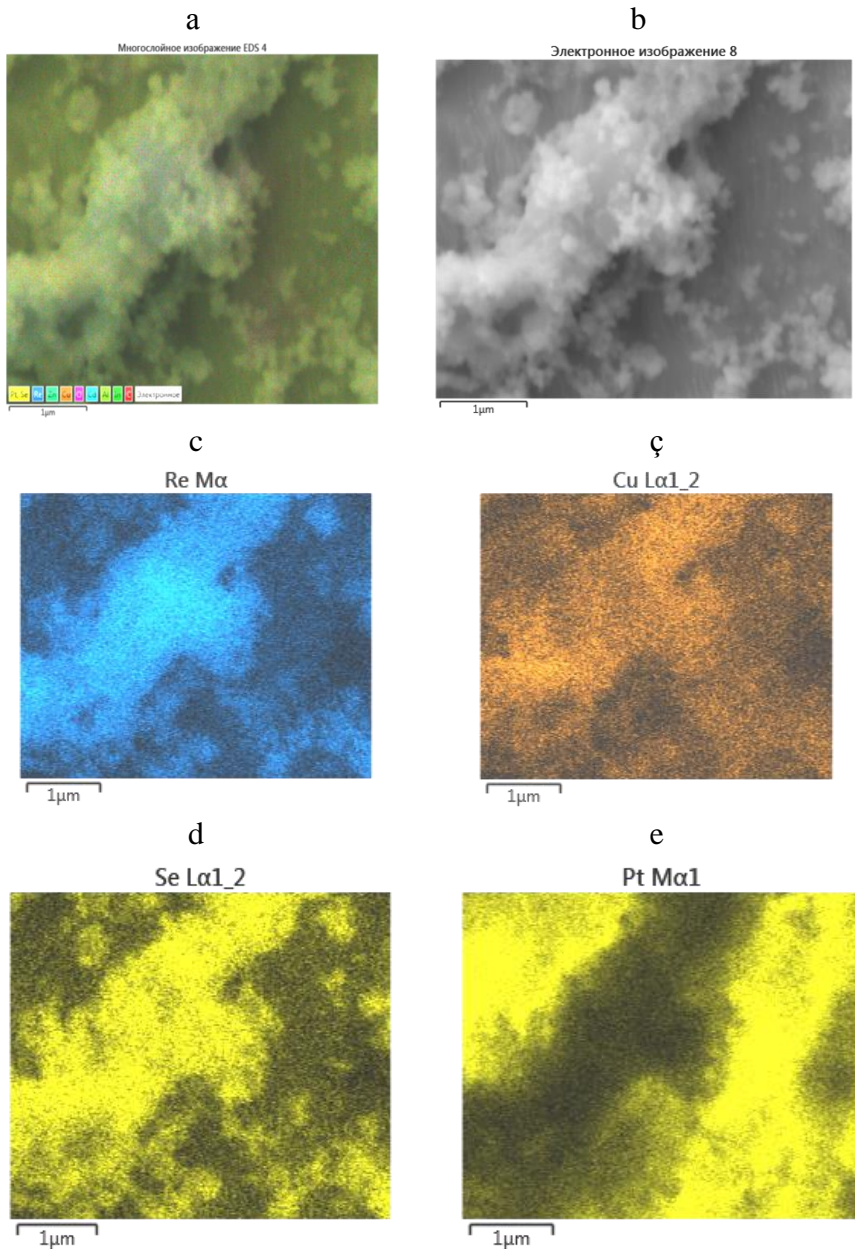


**Figure 10. Morphology and elemental analysis of samples of electrochemically obtained thin layers in a Re – Se – Cu system at 343 K for 1800 seconds on a copper electrode with a potential of + 0.5 V using SEM.**



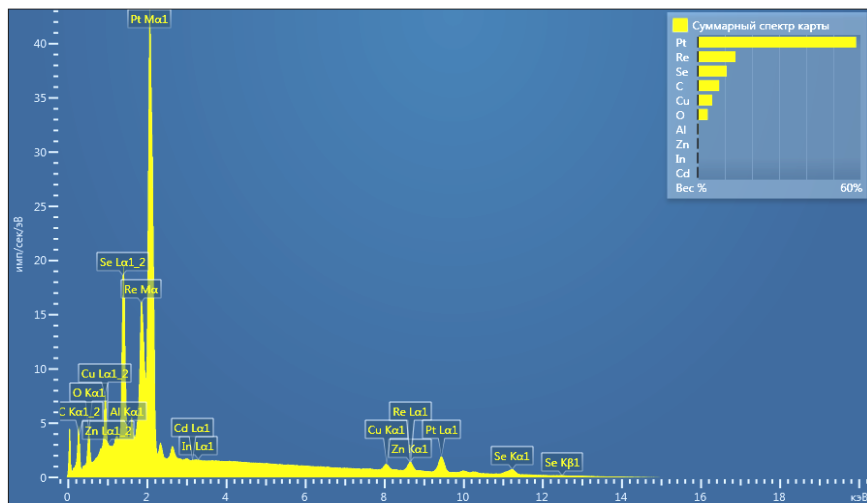
**Figure 11. Electrochemical imaging of thin layers of Re-Cu-Se under an SEM microscope magnified x25000 (a), 50000 (b), 160000 (c) and 200000 (d) times.**

The images obtained at low magnitudes show an even distribution of agglomerates on the platinum electrode. This fact is also confirmed by the map of the distribution of elements in Figure 12 (a – small map, b – electronic image, c, ç, d, e – Re, Cu, Se and Pt location corresponds to the layer). As can be seen from the presented map, the observed content of agglomerates is shown as the main element content of the alloy, and the course of the technological process allows us to conclude that the obtained clusters are the main elements of the Re–Se–Cu system [22]. The spectral characteristics of SEM in Figure 13 also allow for quantitative analysis of the indicated components of a given system. As can be seen from the weight percentage distribution diagram, the amount of components in the system can be shown as Re-12%, Cu-5%, Se – 10%. The presence of characteristic peaks in the spectrum of carbon and oxygen is explained as a residue in the extraction process [27].



**Figure 12. Morphology of alloys obtained by electrochemical Re-Se-Cu system by SEM microscope.**

When studying the X-ray structure of thin films obtained on the platinum electrode in the Re–Se–Cu system by electrochemical means, it was found that they contain oxide compounds. In order to clean the layers from various oxides, the substances obtained by electrochemical means in the Re – Se – Cu system were burned at a temperature of 500<sup>0</sup>C for 2 hours and an X-ray was taken after combustion. At this time, there were no changes in the radiograph.

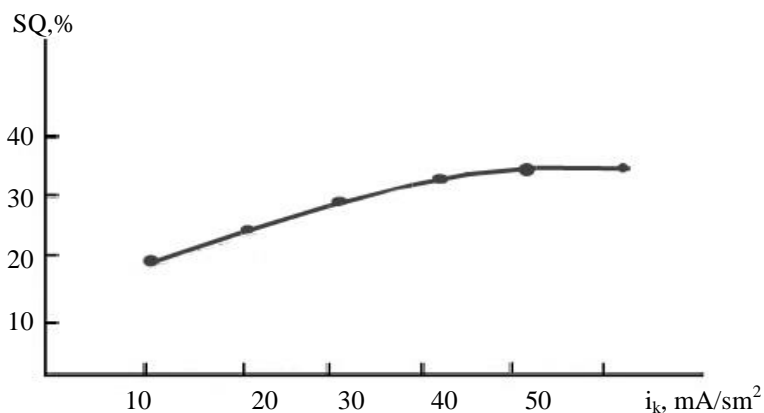


**Figure 13. Morphology and element analysis of electrochemically obtained alloys in the Re-Se-Cu system by SEM microscope.**

Depending on the composition of the electrolyte and the conditions of electrolysis, the current distribution on the electrode (the ability of the electrolyte to scatter) may be different. Proper current distribution during the metal deposition process determines the possibility of obtaining thin layers of the same thickness and quality on the entire surface of the cathode. The Herring and Blum method, which has a wide field of application, was used to determine the dispersancy of the electrolyte used in the Re-Se-Cu system [5].

Figure 14 shows the current density dependence of the electrolyte scattering capacity for the formation of thin layers in the Re–Se–Cu system. For this purpose, two copper cathodes and a platinum-plated anode were used to determine the dispersion capacity of the

electrolyte. In this case, one anode is installed 5 times farther from the other cathode. When the value of one of the parameters changes during the process, the value of the other is kept constant. In this case, the dispersion of the electrolyte and the current distribution were measured and determined to be positive [28]. Thus, at a current density of  $4.0 \text{ mA} / \text{cm}^2$  and a temperature of  $750\text{C}$ , the distribution capacity of the electrolyte is 24%. When the current density is more than  $10 \text{ mA} / \text{cm}^2$ , the yield increases according to the current and the dispersion capacity of the electrolyte increases to 35%. The type of conductivity of thin films obtained electrochemically in the Re-Cu-Se system was determined by the thermoprobe method and it was determined that they have a "p" type conductivity [24].

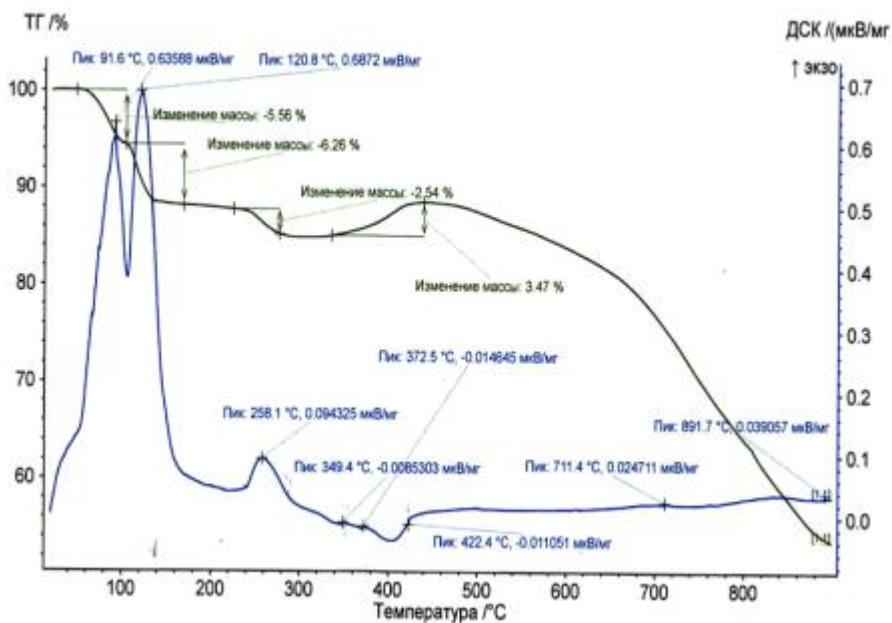


**Figure 14. Dependence of the scattering capacity of the electrolyte on the current density for the formation of thin layers in the Re–Se–Cu system. Electrolyte composition (mol/l):  $7 \cdot 10^{-3} \text{ KReO}_4 + 1,25 \cdot 10^{-2} \text{ SeO}_2 + 1,2 \cdot 10^{-3} \text{ CuCl}_2 + 2.0 \text{ H}_2\text{SO}_4$**

Thermal stability and oxidation of Re – Se – Cu alloy obtained by electrochemical sulphate solutions were studied by thermogravimetric analysis. For this purpose, the thermogram was measured in an inert atmosphere at a temperature of  $20\text{--}900^\circ\text{C}$  and a heating rate of  $100 \text{ C} / \text{min}$ .

It was found that Re-Se – Cu alloys obtained by electrochemi-

cal methods are thermally resistant to the reaction medium. The highest weight loss is 6.25% and 120.8 ° C. Complete oxidation of the electrochemically obtained Re – Se – Cu alloy is characterized by 3 exothermic and 2 endothermic reactions. Oxidation of the alloy is a complex mechanism. The influence of various factors, the concentration and nature of the components in the alloy, the properties of the oxide layers, temperature, changes in crystal structure, etc. plays a big role. According to research, it can be concluded that the oxidation process takes place in a diffusion mode. An increase in temperature is observed with an increase in the rate of oxidation of the alloy, regardless of the composition of the alloy. The first exothermic process begins at 100–120°C and characterizes the detachment of water. The second exothermic process is observed at 250–280°C, which is probably the oxidation of rhenium at that temperature. Figure 15 shows a thermogram of nanoparticles obtained electrochemically in the Re–Se–Cu system.



**Figure 15. Thermogram of electrochemically obtained layers from sulfate solutions in Re-Cu-Se system**

Al-(Re–Se–Cu) type diode structure was created on the basis of layers obtained in rhenium-selenium-copper systems, its dynamic and static voltamper characteristics were studied and it was found that these diode structures have “transformation and memory” effect. Coatings made of these alloys also have a bipolar controllable transformation effect, which makes them suitable for use in semiconductor conversion devices.

The magnetic properties of thin layers of rhenium-selenium-copper alloys obtained by electrochemical methods from sulfate solutions were studied. It has been established that the thin layers obtained in the rhenium-selenium-copper system have paramagnetic properties [23].

A patent entitled "Method of obtaining a thin-layer coating based on rhenium" was obtained from the dissertation work. (Patent I 2018 0016) [31].

The nano-layers obtained by the electrochemical method in the Re-Se-Cu system were exhibited at the institute's exhibition. Figure 16 shows examples of thin layers obtained on copper (a) and platinum (b) electrodes in the Re – Se – Cu system.



**Figure 16. Examples of thin layers obtained on copper (a) and platinum (b) electrodes in the Re – Se – Cu system.**

The research was carried out with the financial support of the Science Development Foundation under the President of the Republic of Azerbaijan. - Grant № EIF-2013-9 (15) -46 / 19/4-M-37

## RESULTS

1. Using modern electrochemical research methods, the mechanism of deposition process of rhenium, selenium and copper from their sulfate solutions was studied separately and together (potentiostatic, temperature-kinetic and voltammetric methods). It was determined that the process of electrolytic precipitation of rhenium from sulfate solutions is mainly characterized by concentration polarization and diffusion of ions on the surface of the cathode.
2. The process of electrolytic deposition of selenium from sulfate solutions was studied and the nature of polarization was determined. Selenium deposition is characterized first by chemical and then by diffusion polarization. The process of electrolytic deposition of selenium is regulated by the reduction of divalent selenium particles formed in the electrolyte.
3. The effect of various factors (current density, temperature, total density, acidity, component density, etc.) on the composition and quality of the semiconducting rhenium-selenium-copper alloy was studied. It was determined that with the increase of current density, temperature and density of rhenium, the amount of rhenium in the alloy increases and the quality of the layers improves.
4. The study of the potentiostatic and cyclic voltammeter characteristics of the co-electrolytic deposition of rhenium and selenium from sulfate solutions shows that during the co-electrolytic deposition of rhenium and selenium in the cathode, a new chemical compound based on rhenium is observed. This is due to the chemical interaction between rhenium and selenium in the cathode.
5. The Re-Cu-Se system from sulfate solutions was studied electrochemically, and based on the conducted experiments, the optimal conditions and composition of the electrolyte were proposed for obtaining nano-layers from sulfate electrolytes. A patent entitled "Method of obtaining a thin layer coating based on rhenium" was obtained (Patent I 2018 0016).
6. The structures of the layers obtained from sulfate solutions in the rhenium-copper-selenium system were studied by X-ray method and as a result of the electrolysis process,  $\text{ReSe}_2$  chemical compound and Re-Cu-Se ternary alloy were obtained. The conductivi-



ty of the nanosheets obtained in the Re-Cu-Se system was determined by the thermoprobe method and it was found that they have "p" type conductivity.

7. Magnetic properties (paramagnetic) and VAX characteristics of thin layers of rhenium-selenium-copper alloys obtained from sulfate solutions by electrochemical method were studied. It was determined that there is a "transformation and memory" effect in the thin layers obtained in the rhenium-selenium-copper system.

**The main results of the dissertation were published in the following scientific works:**

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