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ABSTRACT

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Keywords: rhenium alloys, thin coatings, electrochemical deposition, alloys, current density.

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ABSTRACT

The dependence of various factors (current density, temperature, concentration of rhenium, acidity of a solution, nature of an electrode etc.) for electrodeposition of rhenium from chloride-sulfate solutions and obtaining thin rhenium coatings were stidued. The dependence of current yield of rhenium from chloride-sulfate solutions on current density was investigated. It was determined that when current density changes by 10-25 mA/sm² current yield of rhenium varies between 15-60% and electrolysis must be performed at low current densities to obtain thin rhenium coatings from chloride-sulfate solutions. Electrodeposition of rhenium at different temperatures was confirmed and it was determined that when the temperature increases, deposition of rhenium becomes easier, and wave height in rhenium curve rises. Studies have determined that high-quality and thin rhenium coatings are produced at 75°C. The electorlyte with the following composition was proposed to obtain thin rhenium coatings from chloride-sulfate solutions. The composition of the electrolyte is as follows (mol/l): 0,01KReO$_4$+2H$_2$SO$_4$+2HCl,i_k=1−4 mA/sm² , t=75°C , pH=0,5.

Keywords: rhenium alloys, thin coatings, electrochemical deposition, alloys, current density.

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I. INTRODUCTION

The future development of science and technology is possible thanks to the study of new nanomaterials with new properties and creation of devices on their basis, which meet modern requirements. For this purpose, the production of new semi-conductor nanomaterials and their practical application are of great importance [1-3]. Presently, there are different methods for producing nanomaterials among which electrochemical method is considered to be the most effective and promising for obtaining these materials. Due to the high level of development in science and technology the production of high efficient materials from high quality and existing materials using less material is one of the topical issues of a modern life. For this purpose recently, production of nanoparticles of several substances, their physical and chemical properties allows using them as a promising material in different fields of a modern technology. In this regard, there is a growing interest in electrochemical production of nano films. Electrochemical method has its own positive features. It is possible to perform electrolysis process with accuracy and regulate the thickness of the coating using electrochemical method. To produce these substances there is a wide range of methods that electrochemical method ranks among the highest. Rhenium has a high melting temperature, mechanical durability, hardness and high resistance. Its corrosion resistance and high thermoelectric properties opens wide opportunities for its application in electrical engineering and radio engineering [4-12].It is used in electronics – for the preparation of elements of electron lapms, thermopairs, electrocontacts in electrical engineering. It is also used in the preparation of rocket body, some elements of reactors in aerospace engineering. Therefore, the development of new methods of producing rhenium nano films is of great interest [13-15].One the main issues in the production of rhenium is to obtain high-purity rhenium. Electrochemical method is the most effective method for this purpose. The main aim of the research work is to select optimum mode and electrolyte to produce thin rhenium films from chloride-sulfate solutions.

II. METHODS

The following reagents were used to perform the research work: $KReO_4$ (chemically pure), H_2SO_4 (chemically pure), HCL (chemically pure), Polarization measurements were recorded using platinum cathode with a surface are of 0,15 sm^2. In the experiments silver - chlorine electrode was used as a reference elctrode, but platinum wire was used as an auxiliary electrode. Polarization measurements were recorded using platinum cathode with a surface area of 0,15 sm^2. The kinetics of deposition of rhenium was studied using cyclic voltammetric methods and IVIUMSTAT electrochemical analyzer – potentiostat supplied with a computer. Each experiment was performed twice for the accuracy of the results. The temperature was regulated with ± 0,1°C of accuracy using U-10 thermostat. An electrolyzer supplied with a special glass and burette to collect gas released during electrolysis, was used to determine current yield of hydrogen. Current yield was determined by weight method using copper coulombmeter and calculated with regard to the composition of the deposit. Phase composition of a surface structure of rhenium cathode deposits was determined using Tesla BS-301 electron microscope and «Cotece» M-46 microprobe.

III. EXPERIMENTAL PART

Reduction of rhenium from acid solution was studied by many scientists and it was determined that reduction of rhenium goes in stages [8 - 12], during electrolysis rhenium oxides are formed and hydrogen is released. In our previous research works we have provided extensive information on the deposition of rhenium from sulfate, chloride, chloride-borate, chloride-sulfate and alkali solutions [8-12]. The main aim of the research work is to select optimum mode, electolyte and to study anodic processes in detail to produce thin rhenium films from chloride-sulfate solutions. Little information is available in literature on rhenium deposition from chloride-sulfate solutions [1-5]. Figure 1 shows the polarization curves of the rhenium deposition in various concentrations from chloride-sulfate solutions. In polarization curves of rhenium two different waves are observed: one is at + 0.45 V potential and the second is at + 0.30 V levels. The existence of these waves can be explained by gradual mechanism of reduction of perrhenate ion and as a result the formation of rhenium oxides as an intermediate product is observed. In our view in cathodic process the formation of ReO_3 and ReO_2 is explained by gradual mechanism of reduction process of perrhenate ion till rhenium and confirmed by the formation of red and blue deposits.

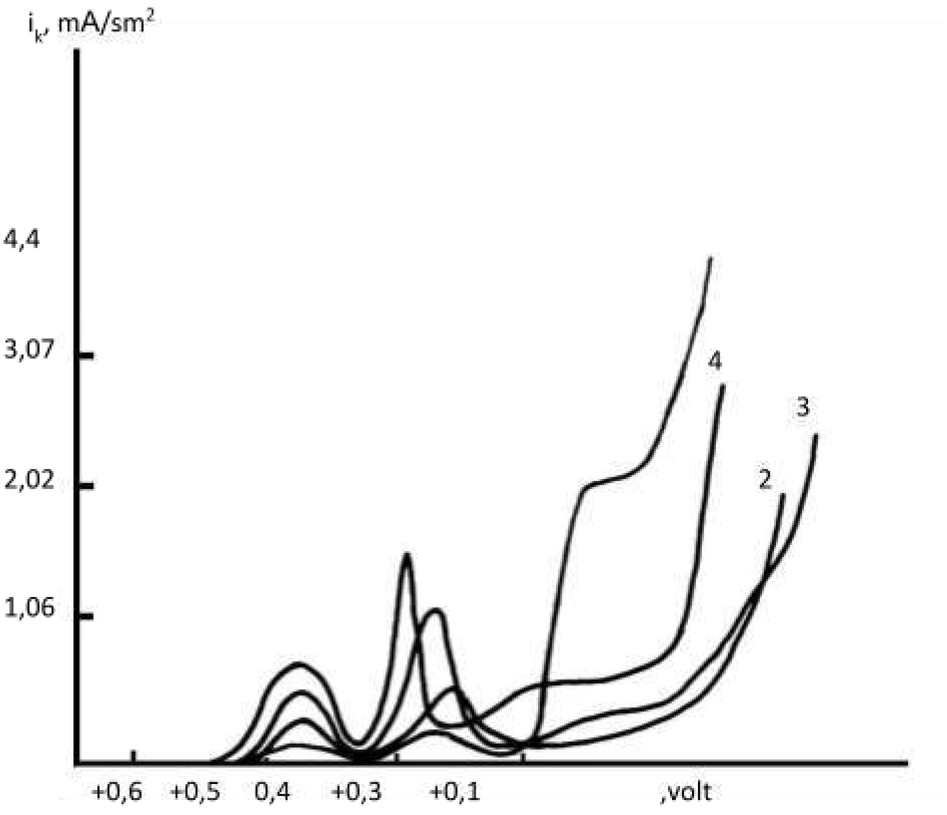

Fig.1: Cathodic polarization curves of platinum electrode in solutions containing 1,5 H_2SO_4 and 1,5 HCl various concentrations of ammonium perrhenate by temperature (°C) 75, concentration of electrolyte $KReO_4$. (mol/l): 1—0,05, 2 – 0,06, 3 – 0,07; 4 – 0,08

It is supposed that reduction of perrhenate ions for the first wave the cathodic process occurs by the following reaction:

$$ReO_4^- + 2H^+ + e \rightarrow ReO_3 + H_2O \quad \text{(I stage)}$$

$$E_1 = E_1^0 + 0{,}058 \lg a_H \cdot a_H^2 \qquad E_1^0 = +0{,}4 \text{ b}$$

The formation of the second wave can be explained by the reduction reaction of ReO_3 to ReO_2.

$$ReO_3 + 2H^+ + 2e \rightarrow ReO_2 + H_2O \quad \text{(II stage)}$$

$$E_2 = E' + \frac{0{,}058}{2} \lg a_H^2 \qquad E = +0{,}6 \text{ B}$$

The last stage is the reduction of ReO_2 to elementary rhenium and expressed by the following reaction:

$$ReO_2 + 4H^+ + 4e \rightarrow Re + 2H_2O \quad \text{(III stage)}$$

$$E_4 = E_4^0 + \frac{0{,}058}{4} \lg {}^{4}_{H} \qquad E_4 = +0{,}252 \text{ b}$$

Sharp increase in a current density observed at a constant potential (+ 0.1 V) is due to hydrogen reduction:

$$E_\Sigma = E_\Sigma^0 + \frac{0{,}058}{7} \lg a_M^2 \cdot a_H^8 \qquad E = +0{,}36 \text{ B}$$

Due to essential sorption of hydrogen by rhenium this shows the properties of hydrogen electrode. Very low tension of hydrogen [1 - 5] on rhenium is due to significant sorption of hydrogen atom by rhenium. As it is seen hydrogen which saturates rhenium, activates it due to the formation of non-constant hydride which is easily converted first to rhenium oxide, and to higher type oxides and then due to the its oxidation.

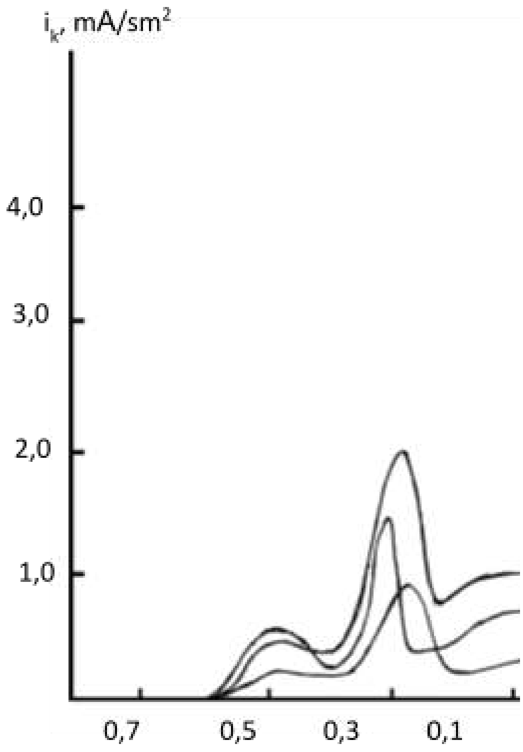

Fig.2: Cathodic polarization curves rhenium on platinum electrode in solutions containing electrolyte (mol/l): 0,05 KReO4+ 1,5 H_2SO_4 + 1,5 HCl + 0,01 $(NH_4)_2SO_4$by different temperature (°C) 1-25; 2 – 45; 3 – 80.

Stationary potential of rhenium in chloride-sulfate solutions is + 0.3 V. To study the kinetics of rhenium deposition potentiostatic and cyclic voltammetric methods were used and polarization curves were plotted. Electrodeposition of rhenium from chloride-sulfate solutions at different concentrations was studied and silvery-gray, crystalline rhenium coating with a thickness of 2-6 mkm which covers the cathode evenly is obtained on the electrode. Deposition mechanism of rhenium depends on the composition of an electrolyte, electrolysis condition, surface state of an electrode, temperature, current density and acidity of a solution. Electrodeposition of rhenium from chloride-sulfate solutions and form and quality of films depends on the temperature. For this purpose, electrodeposition of rhenium at different temperatures and polarization curves were plotted.Figure 2 shows cyclic voltammetric polarization curves of dependence of electrodeposition of rhenium from chloride-sulfate solutions on temperature. During electrolysis temperature ranged between 20-90°C. As figure shows at a higher temperature rhenium is easily deposited, the length of a wave on rhenium curve rises. According to researches it was determined that higher quality and thin rhenium films are obtained at 75°C. To clarify electrodeposition mechanism of rhenium from chloride-sulfate solutions temperature-kinetic method was used. Polarization nature in cathode process was determined by using this method and $\log_к$ – 1/T diagram at different constant values of cathode potential was plotted.

According to straight line dependence at a constant potential of cathode effective activation energy ($A_{eff.}$) of electrode process was calculated using the following equation

$$\log_{к} = cons. - \frac{A_{eff.}}{2{,}303RT}$$

here $i_к$ –current density, R – gas constant, T – absolute temperature, $A_{eff.}$ – effective activation energy.

The results show that if the dependence of logarithm of current density of cathode potential on 1/T is linear, the dependence of an effective activation energy on cathode potential will be accompanied by polarization and the rate of cathode process is characterized by ion diffusion on cathode surface. It was determined that the increase in the temperature from 20°C to 80°C rises cathodic polarization of rhenium, when electrode potential shifts to a negative side activation energy decreases sharply, then it remains constant.

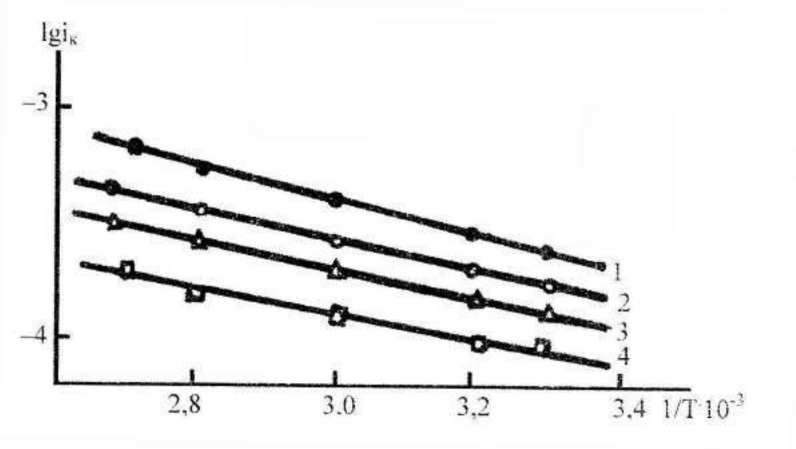

Fig.3: Dependence of lg i_k from 1/T at Pt electrode by potentials (1)-0,65, (2)-. Elektrolyte (mol/l): 0,5 NH_4 ReO_4 + 1,5 HCl + 1,5 H_2SO_4.+ 0,01 $(NH_4)_2SO_4$ by potenceals (v): 1 –(+0,50); 2 – (+0,54); 3 – (+0,56); 4 – (+0,59)

The results show that electrodeposition of rhenium from chloride-sulfate solutions occurs first with electrochemical polarization, then with mixed kinetics, and transforms to concentration polarization. The dependence of current yield of rhenium in electrolyte on temperature, current density, acidity of a solution was studied.

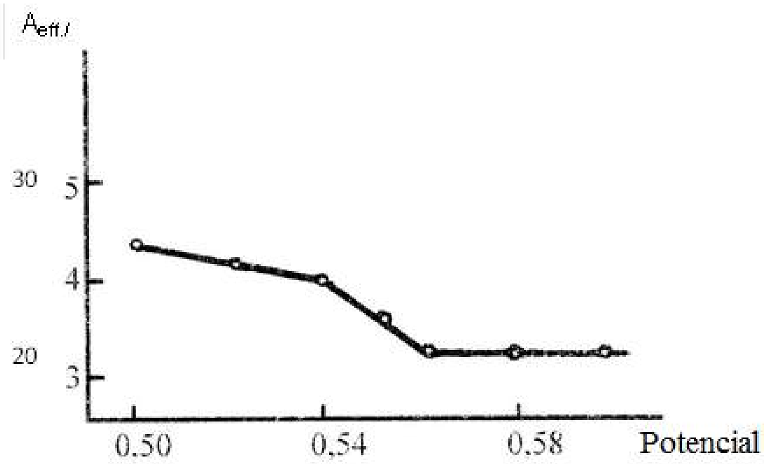

Fig. 4: De[endence of activation effective energy ($A_{eff.}$) from value of potential on the Pt electrode in electrolyte (mol/l) : 0,5 NH_4 ReO_4 + 5 HCl + 5 H_2SO_4.+ 0,01 $(NH_4)_2SO_4$

Table 1 shows the dependence of current yield of rhenium on the acidity of solution. When acidity increases, current yield of rhenium first increases, then at 2,0 mA/sm² current density it decreases and high-quality rhenium films are obtained in pH=1,5 and current yield is found to be 48 %. Thus, as the concentration of HCl in the solution increases, the process of rhenium reduction becomes easier as rhenium dissolves. When the concentration of HCl acidity increases, rhenium forms more stable complex compound in the solution. Depending on acidity effect the properties and form of rhenium films change. As the table shows when the concentration of HCl acid in solution ranges between 1 mol/l to 4 mol/l, the current yield of rhenium varies from 59% to 86%. When the concentration of HCl is 3mol/l, current yield of rhenium increases from 56% to 70% due to the increase in current density. When current density is 10 mA/sm², and concentration of HCl is 1,5 mol/l, dark gray, lustrous, evenly distributed on the surface rhenium film with the thickness of 5 mkm can be produced. At the same current density current yield of rhenium is found to be 75% and with further increase in current density the form and quality of rhenium film becomes worse. Therefore, further experiments were performed in the solution containing 3mol/l of HCl.

Table 1: Dependence of current yield of rhenium on the acidity of solution. Electrolyte content (mol/l): 0,05 $KReO_4$ + 1,5H_2SO_4 , t=75°C.

Concentration of HCl, mol/l	i_k, mA/sm²	Current yield, Re %	Form of coatings
1	10	59	Grey, uneven, congeneric
1,5	10	70	Dark-grey, smooth, congeneric
2	10	75	Dark-grey, shiny, congeneric,
2,5	10	86	Grey, uneven, shiny

As electrolysis occurs at acid medium, hydrogen ions are separated in solution and one part of current is consumed to the separation of hydrogen, the other part is consumed to the separation of rhenium. During deposition of rhenium certain amount of hydrogen is also separated in a cathode and partial polarization curves were plotted to study the processes on electrodes in detail. As it is known as several electrochemical reactions occur on an electrode surface, general polarization curves cannot characterize the rate of any process. Thus, it is found to be a total curve and expresses all electrochemical reactions on electrode surface totally. In this case such complex curve is divided into partial curves. The amount of any current consumed for any reaction is calculated according to the obtained substance: current consumed for the separation of hydrogen is calculated according to the gas volume released during electochemical process. To clarify the kinetics of such mixed electrochemical reactions electrolysis process is performed in a special electrolyzer. Special device is used to collect gases released on electrode. To calculate the amount of a current consumed for the separation of substance on electrode this device is connected to copper coulombmeter. As partial curves show total curve for rhenium is obtained at -0,05 V potential. But the separation of hydrogen in this solution occurs at -0.12 V potential, reduction of rhenium occurs at -0.11V potential. In electrolysis oxidation of water molecule and intensive separation of hydrogen on rhenium occurs at a separation potential of rhenium. The amount of hydrogen separated intensively depends on the composition of solution and current density. Depending on the effect of these factors the amount of hydrogen separated on eelctrode varies. The property and structure of rhenium deposit changes depending on the amount of separated hydrogen. Thus, a part of hydrogen separated during electrolysis sticks to rhenium surface, changes its property and causes defects in its structures. Therefore, to study the mechanism of electrochemical deposition of rhenium from chloride-sulfate solution the effect of all factors (electrolyte composition, current density, acidity, temperature, electrode material) which impact on electrolysis process must be considered in research work.

As it is known the form and quality of rhenium film also depends on a current density. When current density varies between 10 mA/sm² - 20 mA/sm² in solution during electrolysis, current yield of rhenium and form of rhenium film change. This dependence is presented in the table 2.

Table 2: Dependence of current yield of rhenium on current density
Composition of electrolyte (mol/l): 1,5H_2SO_4+1,5HCI

Concentration of $KReO_4$, mol/l	i_k, mA/sm²	Current yield, Re %	Tempe-rature, °C	Form of coatings
0,01	10	30	75	Greyish-blue, uneven
0,01	15	54	75	Light-grey, congeneric unsmooth
0,01	20	58	75	Light-black, matte, uneven
0,01	25	45	75	Dark-grey, unsmooth, congeneric

As the table shows at 10 mA/sm² current density a light-blackish, unlustrous, rhenium film distributed evenly on platinum electrode with thickness of 5 mkm and current yield of 30% was obtained from 0,01mol/l $KReO_4$+1,5H_2SO_4+1,5HCl solution at 75°C. temperature. It was determined that when current density increases, i.e. at 2 mA/sm² of current density, a dark-gray, uneven rhenium film distributed unevenly on a platinum electrode with a thickness of 5 mkm and current yield of 28% was obtained.

The dependence of current yield of rhenium from chloride-sulfate solutions on current density was studied. It was defined that when current density 10-25 mA/sm² changes, current yield of rhenium varies between 15-60%. When current density is 20 mA/sm², i.e. at higher current densities current yield of rhenium reduces. This is explained by hydrogen release at high current densities. It was found out that to produce thin rhenium films from chloride-sulfate solutions electrolysis must be performed at low current densities. The dependence of electrodeposition of rhenium from chloride-sulfate solutions on rhenium concentration on platinum electrode was studied using voltammetric method. Figure 3 shows these polarization curves. By performing electrolysis on a platinum electrode it was determined that anodic polarization curve characterizes anodic dissolution of rhenium on electrode and the process occurs at a positive potential.

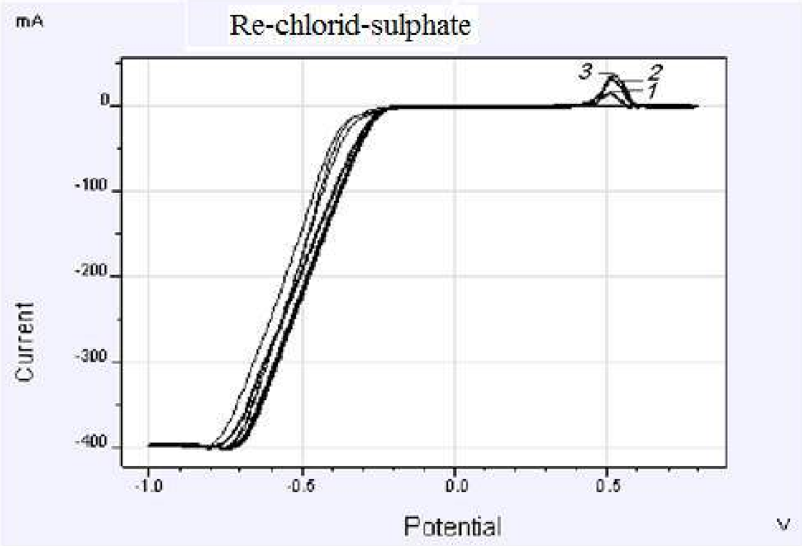

Fig. 5: Cyclic polarizing curves of rhenium on Pt electrode in the electrolyte containing (mol/l): (1) $6.9 \cdot 10^{-3}$ $KReO_4$ + 2 HCl+2H_2SO_4 ;, pH=0.5, T=75 ° (scan rate $V=0{,}005VS^{-1}$)

Electrodeposition of rhenium also depends on the material of an electrode. Figure 8 shows the study of rhenium deposition on different electrodes. Kinetics and mechanism of electrochemical processes depend on an electrode surface, its polishing degree, lustre and so on. Depending on an electrode material any process occurs at various stages. Electrodeposition of rhenium from chloride-sulfate solutions was studied on platinum and rhenium electrodes. In both cases there were no any changes in the form of polarization curves, and this shows that rhenium deposition on both platinum and rhenium electrode occurs by the same electrochemical reaction. By performing electrolysis both on platinum and rhenium electrodes it was determined that reduction of perrhenate ions on rhenium electrode occurs at more positive potential, i.e. electrochemical reduction of rhenium becomes easier. Electrodeposition of rhenium from chloride-borate solutions was also studied. Figure 5 presents cyclic voltammetric polarization curves of electrodeposition of rhenium from chloride-borate solutions.

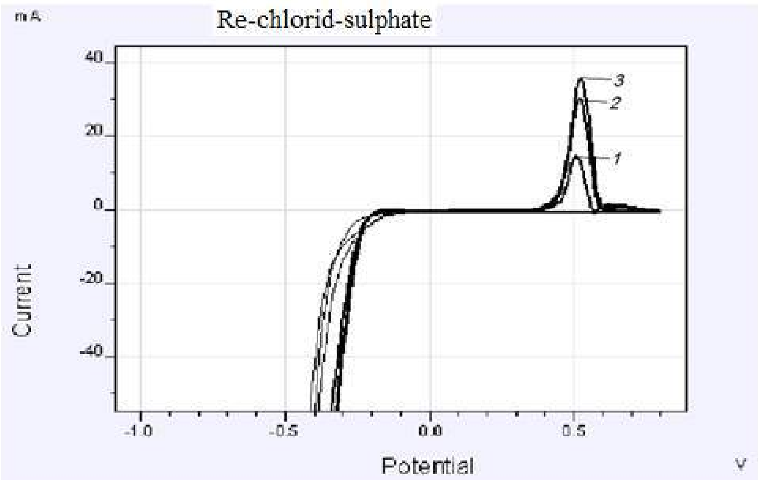

Fig. 6: Cyclic polarizing curves of rhenium on Pt electrode in the electrolyte containing (mol/l): (1) $6.9 \cdot 10^{-3}$ $KReO_4$ + 2 HCl+2H_2SO_4 ;, pH=0.5. temperatur °C; 1-25, 2-45, 3-75 .(scan rate $V=0{,}005VS^{-1}$)

As figure shows rhenium deposition from chloride-sulfate and chloride-borate solutions occurs at various potentials and there are certain differences and changes both in cathodic and anodic polarization curves.

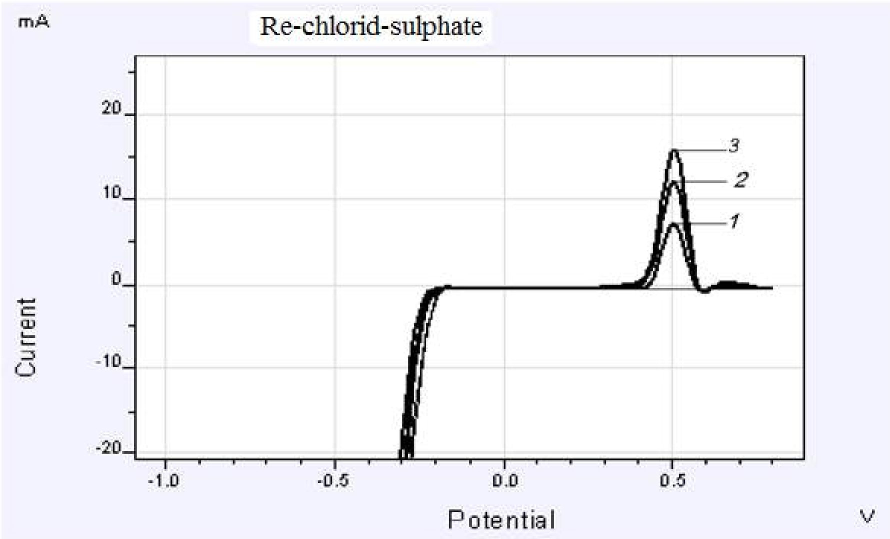

Fig. 7: Cyclic polarizing curves of rhenium on Pt electrode in the electrolyte containing (mol/l): (1) $6.9 \cdot 10^{-3}$ $KReO_4$ + 2 HCl+2H_2SO_4 ; 2-0,015$KReO_4$+2HCl +2H_2SO_4; 3-2$KReO_4$+2HCl +2H_2SO_4, pH=0.5. temperature 75 °C; (scan rate $V=0{,}005VS^{-1}$)

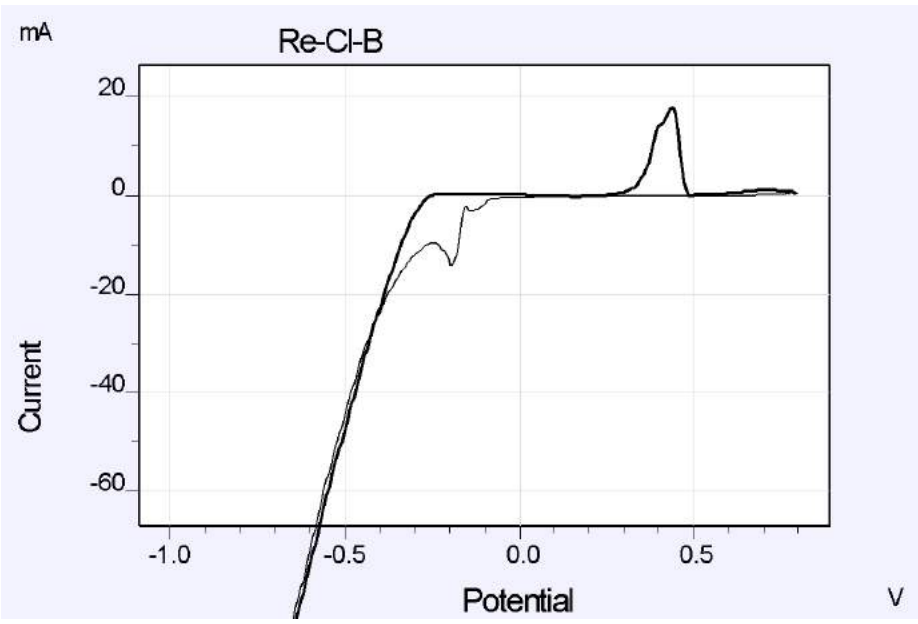

Fig. 8: Cyclic polarizing curves of rhenium on Pt electrode in the electrolyte containing (mol/l): (1) $6.9 \cdot 10^{-3}$ $KReO_4$ + 2 HCl+2 H3BO3; pH=0.5. temperatur 75°C; (scan rate $V=0{,}005VS^{-1}$)

Thus, according to the experiments the electrolyte with following composition was proposed to produce thin rhenium films from chloride-sulfate solutions. Electrolyte composition is as follows (mol/l):0,01$KReO_4$+1,5H_2SO_4+1,5HCl,i_k=1–4mA/sm², t=75°C, pH=0,5

IV. CONCLUSIONS

1. The dependence of current yield of rhenium from chloride-sulfate solutions on current density was studied. It was determined that when current density 10-25 mA/sm² changes, current yield of rhenium ranges between 15-60%. It was defined that electrolysis must be performed at lower current densities to obtain thin rhenium films from chloride-sulfate solutions.
2. Electrodeposition of rhenium at different temperatures was studied and it was defined that at higher temperatures rhenium deposition becomes easier, and wave height in rhenium curve rises. Studies have determined that high-quality and thin rhenium films are obtained at 75°C and optimum temperature was found to be 75°C.
3. Based on the experiments the electrolyte with the following compositon was proposed to obtain thin rhenium films from chloride-sulfate solutions. The composition of an eletrolyte is as follows: (mol/l): 0,01K ReO_4+2H_2SO_4+2HCl, i_k=1-15mA/sm², t=75°C, pH=0,5

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