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ELECTROCHEMICAL STUDY OF RHENIUM-TELLURIUM-COPPER SYSTEM

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ABSTRACT

The formation of the triple alloys Re-Te-Cu on the platinum electrode at volt amperemetric cycling has been studied. The investigation was carried out from chloride acidic solution containing tellurium acid, potassium perrhenate, chloride copper. The kinetics of the processes was controlled using the measurements by the method of cyclic volt-amperometry on the device İVİUMSTAT. For the analysis of composition and structure the methods of XRD (X-ray diffraction analysis) were used, and the investigation of films' morphology on platinum and copper supports was conducted on scanning electron microscope (SEM). To depict the formation of the systems the corresponding mechanism of electrodeposition was suggested. It is shown that the process proceeds through several stages

KEYWORDS: electrodeposition, triple alloys, rhenium chalcogenides, thin films, cyclic voltampermetry.

I. INTRODUCTION

Beginning from 1990s a new technology – nanotechnology based on achievements of physics, chemistry and engineering has developed intensively and gained the status of XXI technology. Nanotechnology is not only the technology of nanoparticles, materials and products. This technology is a delicate technology which involves many fields of chemistry and materials, food industry and biotechnology, electronics and materials industry, science and technology. Achivements of nanotechnology is widely used and considered in all fields of human activity. Nanotechnology has great opportunities. Thus, amount and types of products based on this technology are being grown rapidly. In nanotechnology researches are conducted on the basis of physics and chemistry. By using methods and achievements of these sciences and manipulating atom and molecules small, highly-active materials are obtained. In traditional industrial technology materials and devices with relevant structure are prepared from any raw material by purifying from unwanted matters. Due to high development of science and technology production of high-efficiency materials from high quality materials by using less material is one of the topical problem of modern life.

For this purpose recently production of nanoparticles of several substances, their physical-chemical properties has allowed us to use them as a promising material in different fields of modern technology. From this point the interest to electrochemical obtaining of nano films grows.[1-4].

Electrochemical obtaining of high photo effective semi-conducting thin and nanofilms in visible region of solar spectrum and study of their properties is a topical problem. Demands for producing several semi-conducting materials due to rapid development of science and technology grow.

Production of nano particles, studying their physical-chemical properties allows us to use them as a promising material in different fields of modern technology. Rhenium and its alloys have wide application areas. Rhenium chalcogenide (S, Se and Te) films are widely used in various areas of electronics [1-7]. Thus, obtaining of semi-conducting triple rhenium alloys is of great interest. From this point demand for electrochemical obtaining of nano films grows. Electrochemical obtaining of high photo effective semi-conducting thin and nanofilms in visible region of solar spectrum and study of their properties is a topical problem. In our previous works we studied electrochemical obtaining process of thin films of rhenium chalcogenide alloys [6-12].

The aim of research work is the electrochemical obtaining of thin films of rhenium chalcogenide alloys.

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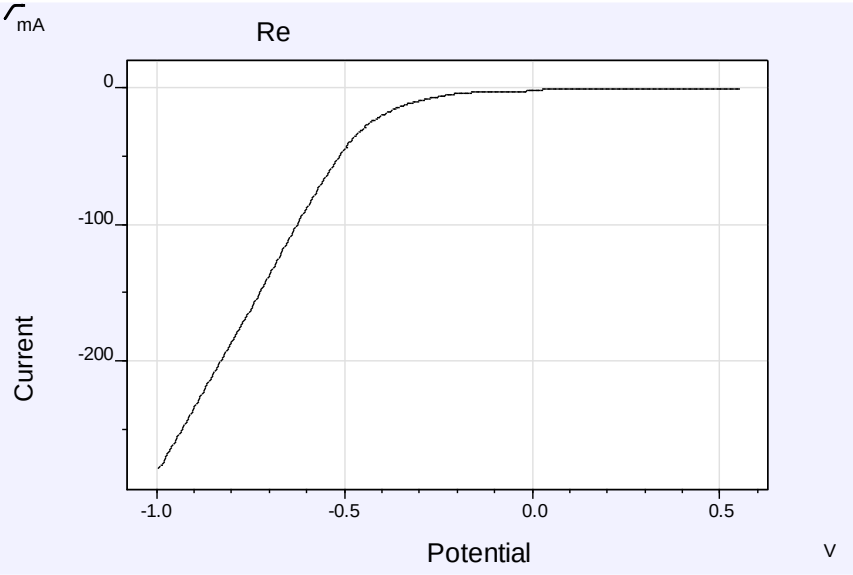
II. RESEARCH METHODS

To obtain Re-Cu-Te alloy we used solutions of ammonium perrhenate, tellurium oxide, cupric sulfate in chloride acid as an electrolyte. For obtaining of an alloy we used platinum electrode with $4sm^2$ area as a cathode, but for polarization measurements we used platinum electrode with $0.15sm^2$ area. As a comparative electrodue we used silver-chlorine electrode, as an auxiliary electrode we used platinum plate. Potentiostatic curves were plotted in P-5827M potentiostat with KSP-4 potentiometer, cyclic voltampermetric curves were plotted with IVIUMSTAT potentiostat with 50ml glass electrolizer covered with glass coat. Temperature was controlled at ±0.1ºC accuracy by U-10 thermostat. pH of solution was measured with Az86551 device.

III. EXPERIMENTAL PART

To study co-electrolytic process of rhenium tellurium and copper the deposition process of these metals from separate electrolyte must be studied. For this purpose we studied electrolytic deposition of rhenium, tellurium and copper from chloride solutions.

We studied electrolytic deposition of rhenium from chloride solutions and it was determined that in chloride electrolyte rhenium is in the form of perrhenate ion (ReO^-_4) and reduction of rhenium goes in stages [7-10]. We studied the effect of density, temperature of rhenium, acidity of solution in electrolyte, conversion rate of potential on polarization curves. (Fig. 1). Reduction of rhenium goes in several separate process and loadlessness of ReO_4^- ion goes in stages and results in formation of intermediate products.



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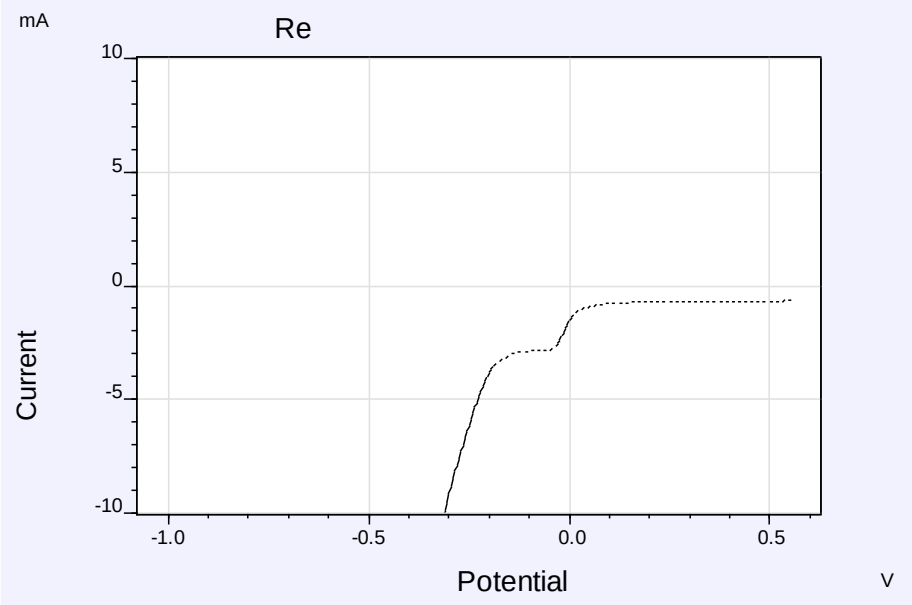

Fig.1. Volt-ampere cyclic polarizing curves of rhenium from chlorid solutions on the platinum electrode in the electrolyte contest, mol/l: 3,5·10^{-3}KReO$_4$·2HCl at t=75°; V=0,005V/S; pH=0,4

Thus, reduction of rhenium from strong acidic electrolytes results in formation of intermediate oxide layers and this is confirmed by existence of oxide layers and red and blue color in deposite. By studying effects of density of rhenium, chloride acid, temperature, conversion rate of potential we selected optimum condition and necessary electrolyte to obtain high-quality rhenium deposites from chloride electrolyte. Electrolytic deposition process of tellurium from chloride solutions was studied. The most useful electrolyte is chloride electrolyte for deposition of tellurium from acidic medium (Fig.2.)

We studied the influence ofelectrolytic deposition of tellurium from chloride acid on polarization curves, density of tellurium in electrolyte, temperature, conversion rate of potential.

Reduction of tellurium ion goes according to the following way: the first stage of electrode process is determined by formation of elementary tellurium. In the second stage divalent tellurium is formed. In the third stage of electrode process we observed the formation of hydrogen. In reduction of tellurium ion we studied the effect of temperature on electrode process in stable value on cathode potential to determine the polarization.nature of cathode.

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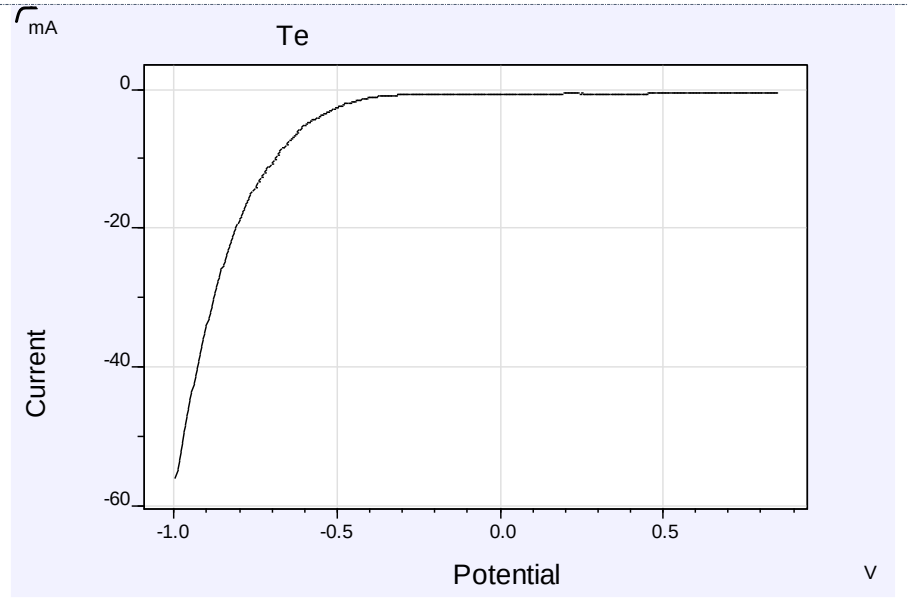

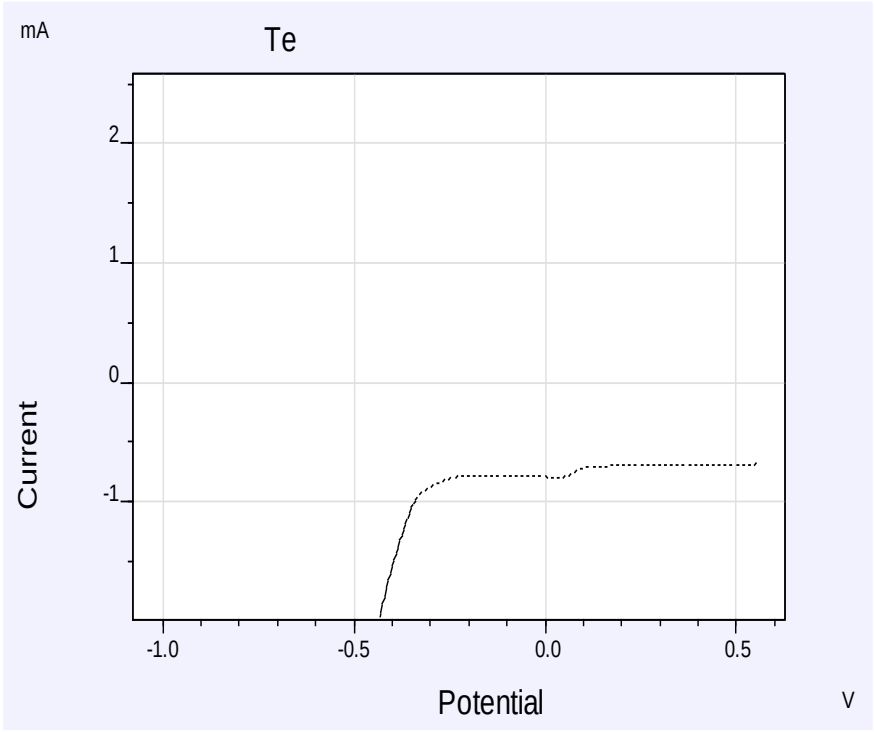

Fig.2. Volt-ampere cyclic polarizing curves of tellurium from chlorid solutions on the platinum electrode in the electrolyte contest, mol/l: 0,6·10^{-2}TeO$_2$+2HCl at t=75°; V=0,005V/S; pH=0,4

Copper and its alloys (chalcogenides) are widely studied, how it is shown in literaure. To study electrolytic deposition of copepr from chloride solutions we plotted cyclic polarization curves. For this purpose anodic curve of copper was plotted and they are given in figure 3.

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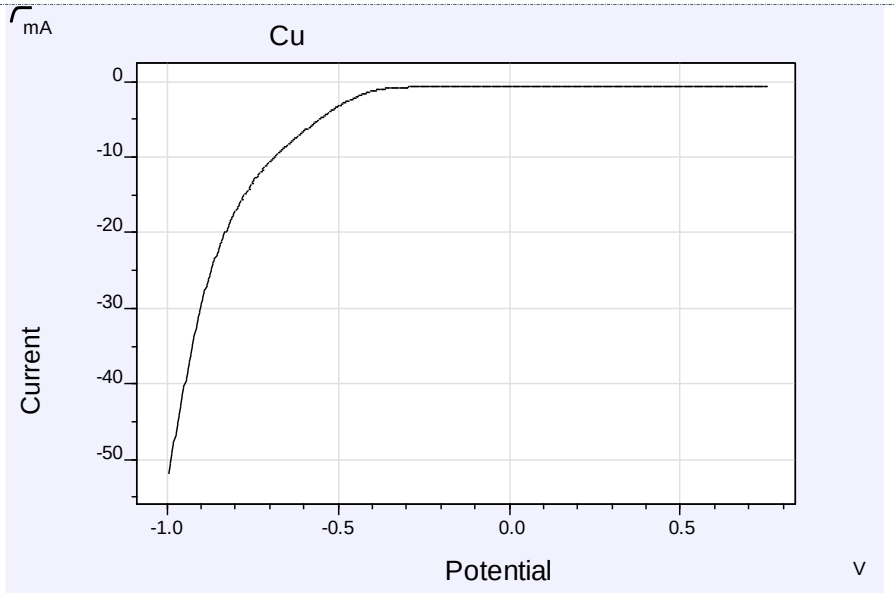

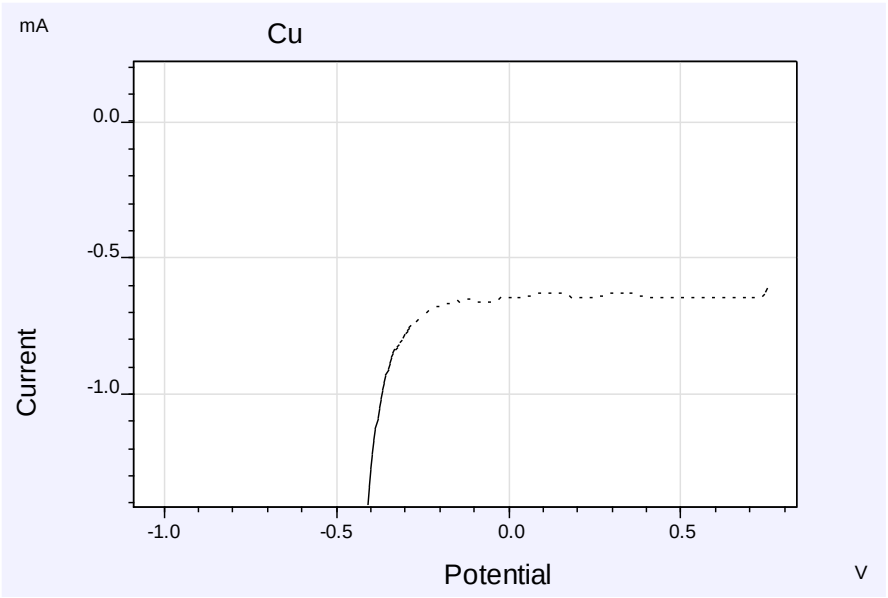

Fig.3. Volt-ampere cyclic polarizing curves of cuprum (II) from chlorid solutions on the platinum electrode in the electrolyte contest, mol/l: 1,2· $10^{-3}$$CuCl_2$+2HCl at t=75°; V=0,005V/S; pH=1,2

One wave is observed in anodic curve of copper (potential-0.05B) and this is oxidation curve of copper.

$$Cu - 2e = Cu^{2+}$$

To clarify the mechanism of electrolytic deposition process of copper from chloride solutions we plotted cyclic polarization curves and it was determined that reduction process of copper occurs in cathode (Fig. 3.). By studying the effect of density of copper, electrolyte, temperature, acidity, conversion rate we selected deposition of copper from electrolyte and optimum condition. When adding copper to tellurium solution cyclic polarization curve changes and several anodic peaks are formed and co-electrolytic deposition process of copper with tellurium occurs on electrode (Fig. 4.).

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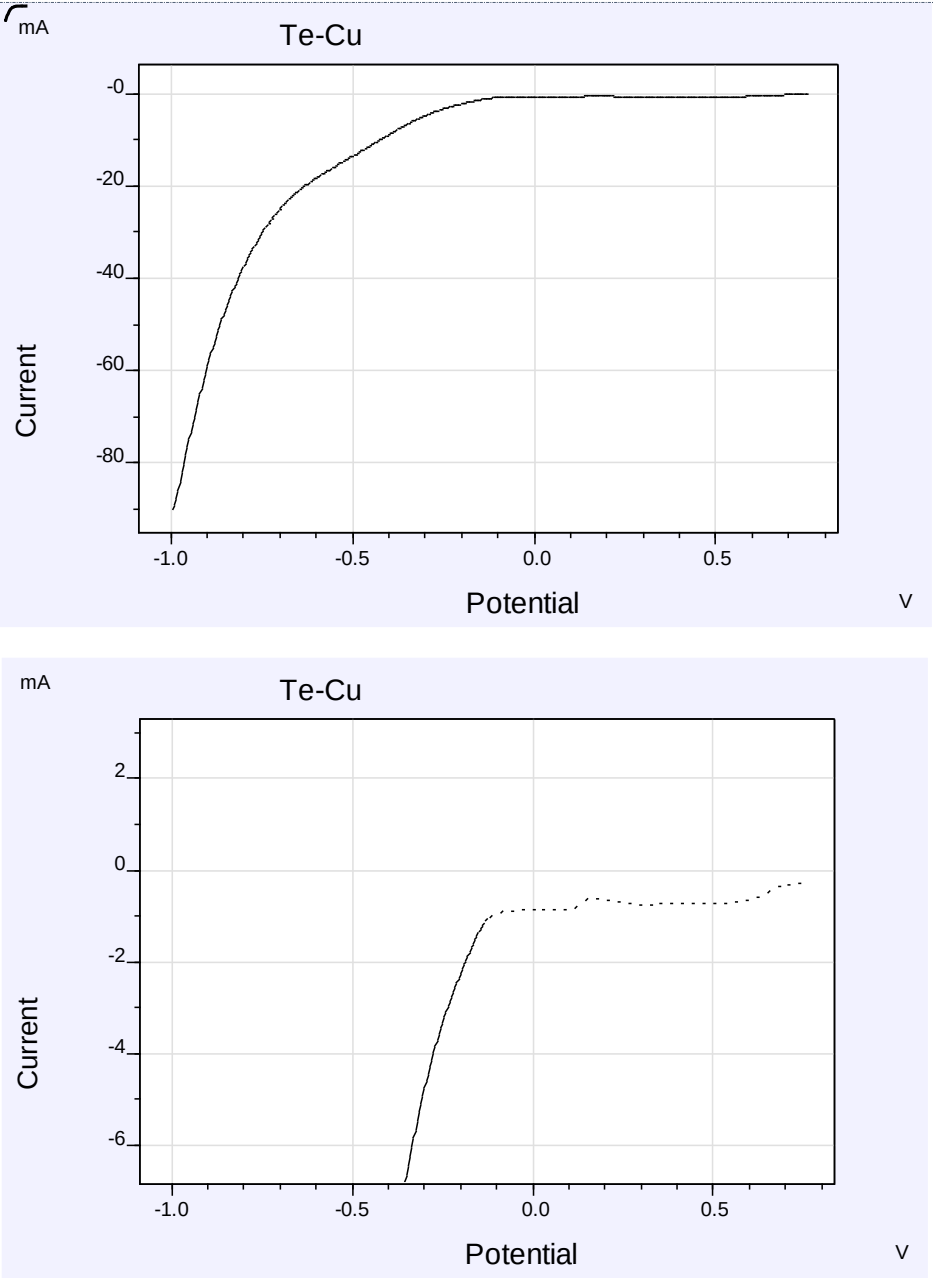

Fig.4. Volt-ampere cyclic polarizing curves of Cu-Te alloy from chlorid solutions on the platinum electrode in the electrolyte contest, mol/l: 1,25·10^{-2}TeO$_2$+1,2· 10^{-3}CuCl$_2$·2H$_2$O+2HCl at t=75°; V=0,005V/S; pH=1,2

CuTe - 2e = Cu+ Te

Cu-Te compound is formed on electrode. In the third stage of electrolysis process by adding rhenium solution into solution we studied formation of triple alloy. When observing voltamperic curves we see that deposition of Cu(II) and Te(IV) begins at +0.2V potential and in this process two metals are deposited and CuTex chemical compound is formed on electrode. Hydrogen telluride is formed by the reduction of CuTex compound with hydrogen. In anodic curve 2 maximums are observed and at +0.50 potential oxidation of CuTe chemical compound occurs. As we know, during co-electrolytic deposition of copper with tellurium CuTex, Cu_2Te or $CuTe_2$ chemical compound is formed. In our curves, polarization curves 2 waves are observed and this conforms to literature data and confirms the formation of a new chemical compound. Then we studied co-electrolytic deposition of Re with Te in chloride solutions and polarization curved were plotted.

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$ReO_{4^-} + 7e + Te_{2^-} + 8H^+ \rightarrow ReTe_2 + 4H_2O$

According to long-term experiments we studied electrochemical obtaining of Re0Te alloy and during deposition process obtaining of $ReTe_3$, Re_2Te_5 compound was confirmed. To confirm the obtained results and to clarify kinetics and mechanism of co-deposition process of Re with Te anodic curves were plotted.

$ReTe_2 - 7e \rightarrow Re + 2Te$

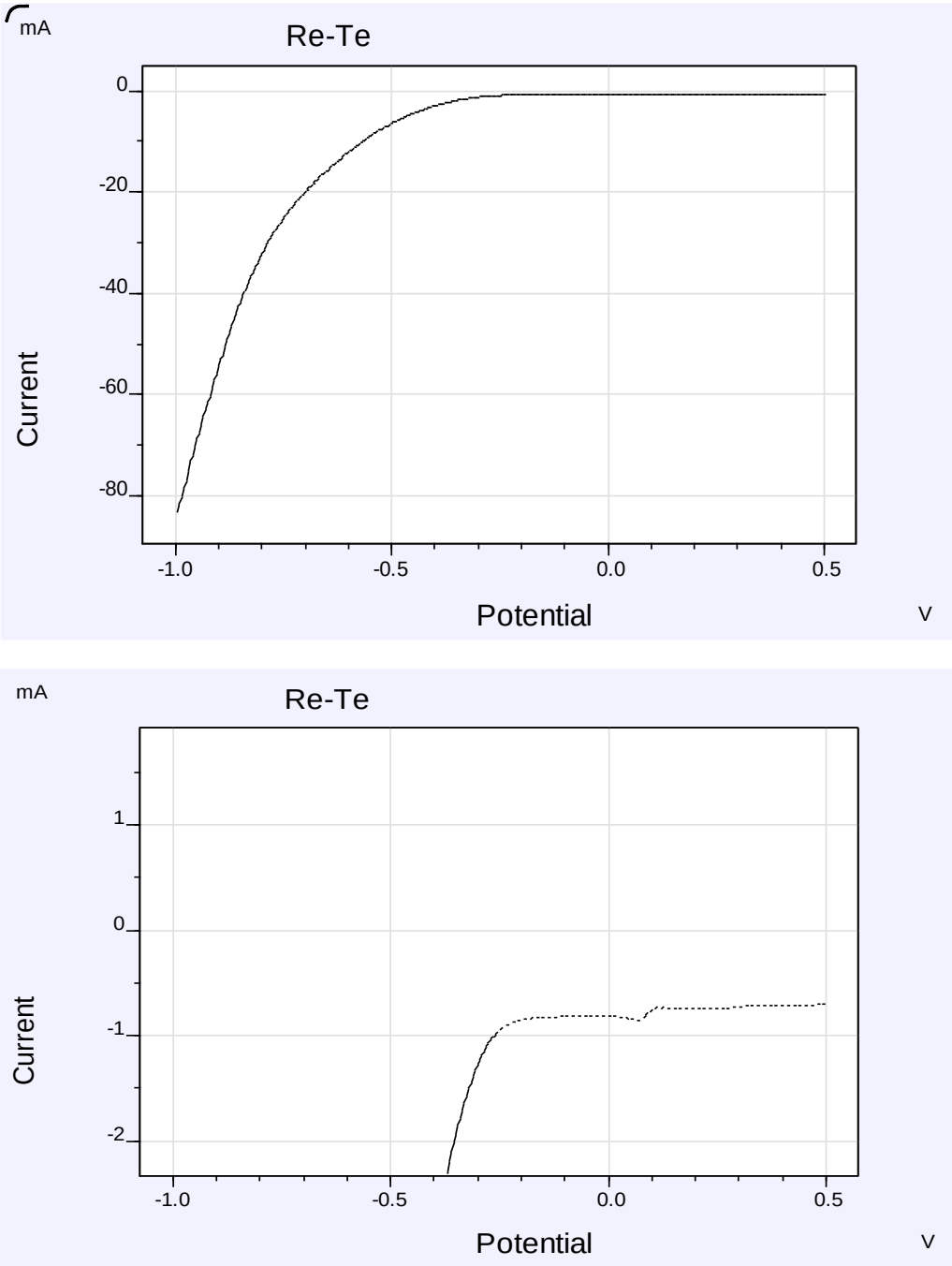

Fig.5. Volt-ampere cyclic polarizing curves of Re-Te alloy from chlorid solutions on the platinum electrode in the electrolyte contest, mol/l: $7 \cdot 10^{-3} KReO_4 + 1{,}25 \cdot 10^{-2} TeO_2$ at t=75 °; V=0,005V/S; pH=0,4

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At figure 5 cyclic polarization curves of rhenium-tellurium curve were plotted and it was determined that co-deposition of Re with Te is possible and $ReTe_2$ and Re_2Te_5 compound is formed on electrode. To study co-deposition of Re-Te-Cu components we took an electrolyte containing three components and polarization curves were plotted. In figure 6 shows voltamperic polarization curves of co-deposition process of Re-Cu-Te. For studying deposition process of Re-Te-Cu alloy we researched the effect of density of components, temperature, pH, total density. When observing voltamperic polarizarion curves of Re-Cu-Te we see 3 waves here as compared to Re-Te and Cu-Te anodic curves. I wave in anodic curve can be related to oxidation of Cu, II wave – to oxidation of tellurium, III wave – to anodic dissolution of new triple Re-Te-Cu alloy.

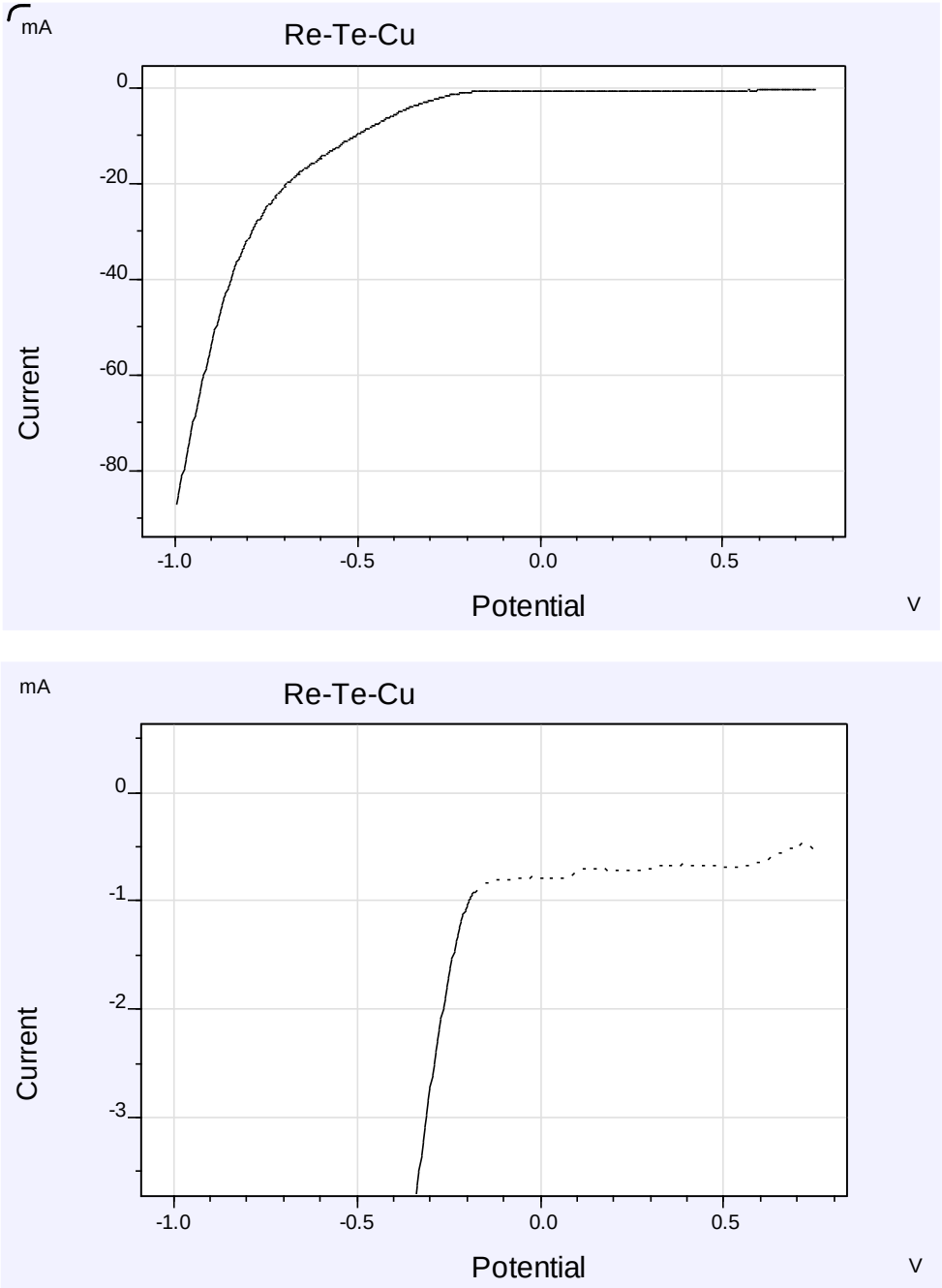

Fig.6. Volt-ampere cyclic polarizing curves of Re-Te-Cu alloy from chlorid solutions on the platinum electrode in the electrolyte contest, mol/l:7·10⁻³$KReO_4$+1,25·10⁻²TeO_2+1,2·10⁻³$CuCl_2$+2HCl at t=75°; V=0,005V/S; pH=0,4

$ReCuTe - 7e = Re^{7+} + CuTe$

This wave confirms that triple Re-Te-Cu alloy is formed in the solution at definite potential. We studied the influence of different factors on the alloy. Thus, the effect of density, temperature, pH of solution, conversion rate of potential, materials fo electrode and others on electrolysis process. It was determined that more high-quality thin coatings are obtaiend on Pt and Ni electrode at 75°C. Besides, polarization curves in different cycles at various conversion rate of potential were plotted and mechanism of deposition process was studied. X-ray structure and microstructure of electrochemically obtained alloys in Re-Te-Cu were studied by SEM microscopy. Electrochemically obtained substances (Re-Te-Cu) were studied on copper, platinum and nickel electrodes.

Results of experiments morphology films are shown in figure 7. It was determined that sizes of substances obtained in samples of Re-Te-Cu variy in 80-150 nm on platinum electrode, in 200-350nm on copper electrode. It was determined that during electrolysis process depending on electrolyte and current density substances with double and triple phases are formed.

IV. CONCLUSION

1. On the basis of investigating volt ampere dependences at the joint electrodeposition of ions of perrhenate (VII), tellurium (IV) and copper (II) from chlorid acidic electrolytes on Pt, (copper) electrode the conditions of potentiostatic deposition of the thin ternary coverings Re- Te-Cu have been established.
2. By the method RPhA and investigation of morphology of the films on copper support and atomic adsorption spectroscopy (AAS) method of analysis, the composition of the received ternary covering Re-Te-Cu has been confirmed and surface morphology has been studied

	%	Ato
Cl K	5.53	15.9
Cu	26.6	42.9
Te L	15.6	12.5
Re L	52.1	28.6
	100.	

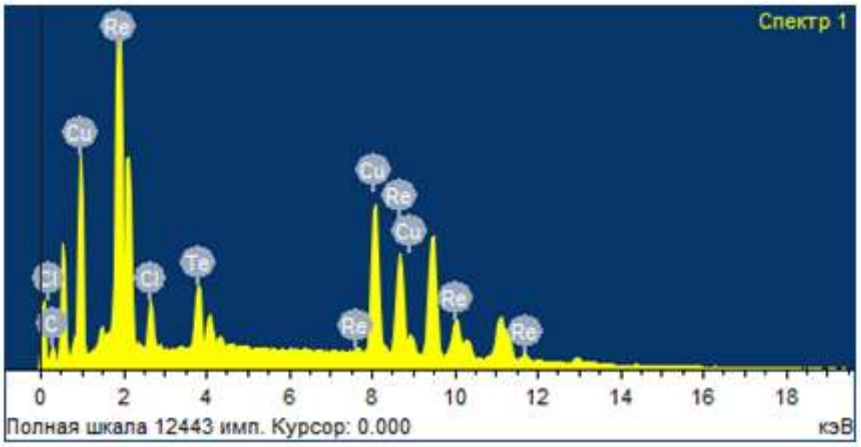

Fig.7. Analysis SEM for obtained thin films in the system Re-Te-Cu during 1800 seconds on the platinum electrode at +0,5V potential and t=343°K

V. ACKNOWLEDGMENT

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