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Morphology of Thin Films Obtained in Re-Te-Cu System by Electrochemical Method

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Abstract-- The formation of triple alloys Re-Te-Cu on the platinum electrode for volt amperemetric cycling has been studied. This research has been carried out from chloride acidic solution containing tellurium acid, potassium perrhenate, chloride copper. Kinetic of the processes has been controlled for measurements assistance by volt amperemetric cycling method on the device UVIUMSTAT. For content and structure analyses have been used XRD methods and estimation of films morphology on the platinum and copper substrates has been carried out by SEM (scanning electronic microscope)

Keywords-- *Electrodeposition, triple alloys, rhenium chalcogenides, thin films, cyclic voltampermetry.*

I. INTRODUCTION

Production of nanoparticles and study of their physical and chemical properties allow using these substances as a perspective material in various fields of modern technology.

Rhenium and its alloys are one of the substances which have wide range of application areas. Rhenium chalcogen (S, Se and Te) films are used in different fields of electronics [1-5]. Therefore, production of semi-conductive rhenium ternary alloys is of great interest. There is a growing interest in electrochemical obtaining of nanofilms. Electrochemical obtaining of thin and nano films of high photoeffective semi-conductors in visible region, study of their properties are more relevant today. In our previous works we studied electrochemical obtaining of rhenium chalcogenide thin films [6-10]. The research work is aimed at electrochemical production of thin films of rhenium chalcogenide ternary alloys and studying morpological structure of these thin films.

Recently studying the electrolytic co-deposition of rhenium with tellurium has been a scientifically and practically important problem. The literature data show that morphological structure of electrochemically produced thin films depends on some parameters. These are density of components in electrolyte, current density, temperature, pH, electrolysis time and potential region of the process.

When studying the production of rhenium-tellurium-copper alloy thin films by electrolysis, it was detected that the main factors influencing on appearance and structure of an alloy are current density, temperature and density of components in electrolyte.

II. RESEARCH METHODS

To determine chemical, phase composition and micro structure of Re-Te-Cu alloys we used platinum plate with 4 sm^2 surface, but for plotting polarization curves we used platinum cathode with 0,15 sm^2 surface, as an anode we used platinum plate. IVIUMSTAT electrochemical analyzer – potentiostate was used To study electrochemical preparation of Re-Te-Cu thin films. Silver/silver chloride electrode was used as a reference electrode. The temperature ± $0,1^0$C with accuracy was regulated using U-10 thermostate. Current yield was determined by copper coulombmeter using weight method and calculated according to the deposit composition.

X-ray phase analysis of Re-Te-Cu alloy thin films was performed on "Bruker" powdery "D2 Phazer" diffractometer in CuK_α – radiation. Micro structure (morphology) of alloys produced in Re-Te-Cu system was studied using SEM microscope.

III. EXPERIMENTAL PART

We used the following electrolyte for electrochemical production of Re–Te–Cu alloy thin films (mol/l): $7\cdot10^{-3}$ $KReO_4$+$1,25\cdot10^{-2}$$TeO_2$+$1,2\cdot10^{-3}$$CuCl_2$+2HCl; current density i_k= 1–15 mA/cm^2, t= 75 – 80^0C, anode -platinum, cathode-platinum, copper, nickel.

Microstructure of Re–Te–Cu alloy thin films produced in different current densities was studied, and it was determined that the most suitable temperature to produce qualitative Re–Te–Cu alloy thin films during studying electrolysis process was found to be 75–80^0C. At this temperature black greyish, thick, lustrous small crystalline Re–Te–Cu alloy thin films are produced on cathode. The quality and structure of a deposit changes as the temperature of an electrolyte increases.

More qualitative deposits are formed at relatively low current densities and the quality of cathode deposit deteriorates as current density increases. When studying the morphology of Re–Te–Cu thin films, it was detected that black greyish lustrous small crystalline film is produced on a cathode (5 mA/cm^2) at low current density. When current density is higher than 15 mA/cm^2 black deposit is produced which is easily removed from brittle metal.

The impact of current density on appearance and structure of Re–Te–Cu alloys is explained as follows: as ionic diffusion on electrode surface does not go slowly in current densities lower than limiting current, crystallization rate is turned to be in the right quantity. After the formation of limiting current, approxmation of ions to electrode surface is complicated, and this decreases crystallization rate. Number of ions reduced on a cathode decreases, some parts of a cathode surface become passive, crystallization takes place only in active parts of the surface. Therefore, the produced deposit is brittle, unevenly distributed and tightly connected to a surface. The quality and structure of deposit changes as the temperature of electrolyte increases.

Micro structure (morphology) of thin films electrochemically produced in system Re–Te–Cu was studied using SEM microscope. Morphology of thin films were studied by different electrodes: copper, platinum, and nickel electrodes. Sizes of thin films are different depending on the electrode material. Thus, films produced on various electrodes from the same electrolyte and in the same current density vary in sizes and compositions. Sizes of substances produced on platinum electrode vary between 200 - 350 nm. Research results are

shown in Figure 1 (morphology of films) and Figure 2 (elemental analysis of composition of films). Figure 1 shows SEM analysis of samples of thin films which were produced electrochemically in Re–Te–Cu system on platinum electrode at 343 K, within 1800 sec at +0.5V potential.

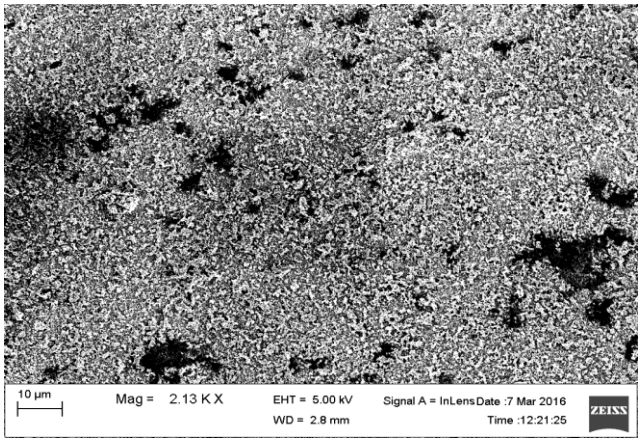
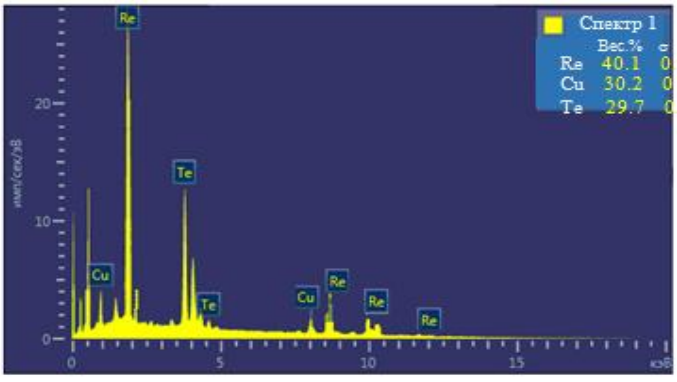


Figure 1: Analysis SEM for obtained thin films in the system Re-Te-Cu during 1800 seconds on the platinum electrode at +0,5V potential and t=343ºK

Chemical analysis of all elements in the composition of the samples of the electrolyte was performed and the results of this analysis were given in Figure 2. A sample was taken from three parts of a film produced on platinum electrode and its spectrum was taken. Results of elemental analysis of samples of thin films produced in Re-Te-Cu system on platinum electrode within 1800 sec at +0.5V potential at 343K: Re – 40,1% , Te – 29,7% , Cu – 30,2%, Re – 40,6% , Te – 30,7% , Cu – 28,7% , Re – 45,8% , Te – 28,0% , Cu – 26,2%

As seen from the results of elemental analysis films are homogeneous.

Differencies in phases on the border of deposited and non-deposited parts during studying samples deposited on platinum electrodes confirms the presence of various compositions. In more detailed study we obseved nanostructure like structure. Formation of nanosize areas with a certain regularity during growing on platinum sample is observed. Sizes of areas vary between 180–350 nm. Areas are in shapes of an egg, ellipse and in various form s. Morphology of thin films of Re–Te–Cu system was also studied on copper electrode. It was detected that sizes of thin films in Re–Te–Cu system vary depending on an electrode material.



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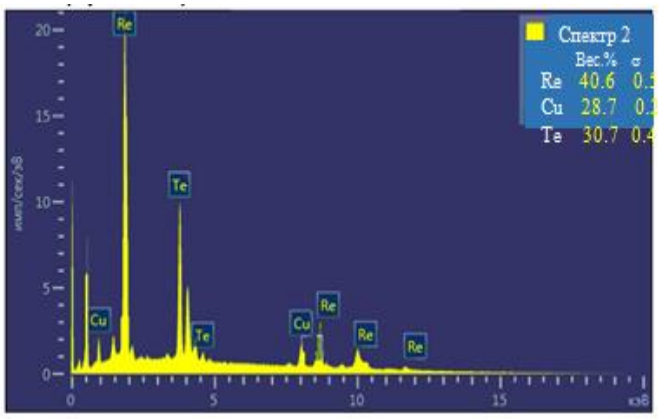

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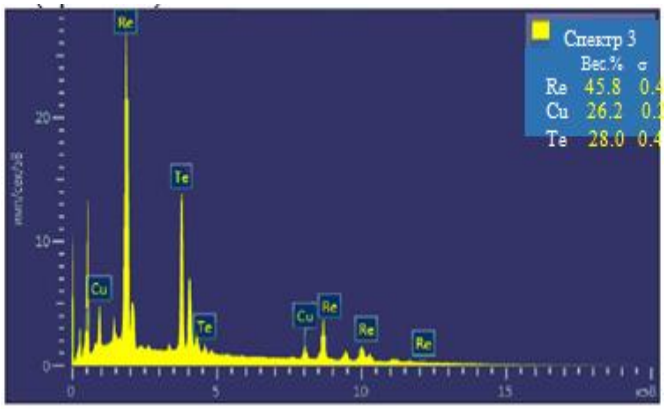


Figure 2: Elemental analysis SEM for obtained thin films in the system Re-Te-Cu during 1800 seconds on the platinum electrode at +0,5V potential and t=343ºK

Morphology of these thin films produced in Re–Te–Cu system was also studied on copper electrode. It was also determined that sizes of thin films in Re–Te–Cu system vary depending on electrode material. During SEM observation square or cube copper particles in copper samples are clearly seen. It should be noted that sizes of a deposited substance are very small and sizes of this subtance vary in the range of 50–130 nm. According to the results of SEM analysis carried out in 4 different parts of thin films Re, Te, Cu

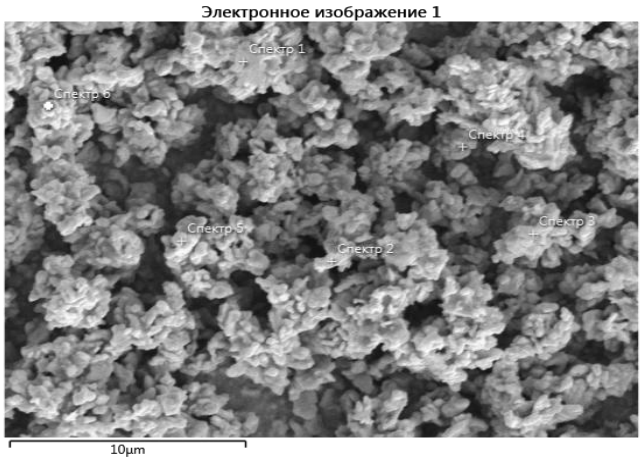


Figure 3: Analysis SEM for obtained thin films in the system Re-Te-Cu during 1800 seconds on the platinum electrode at +0,5V potential and t=343ºK

Figure 3 shows SEM analysis of the samples of thin films on copper electrode at +0.5V potential within 1800 sec at 343 K. The type of conductivity of thin films of Re-Te-Cu system was determined by hot-probe method and it was found that they have “p” type conductvity.

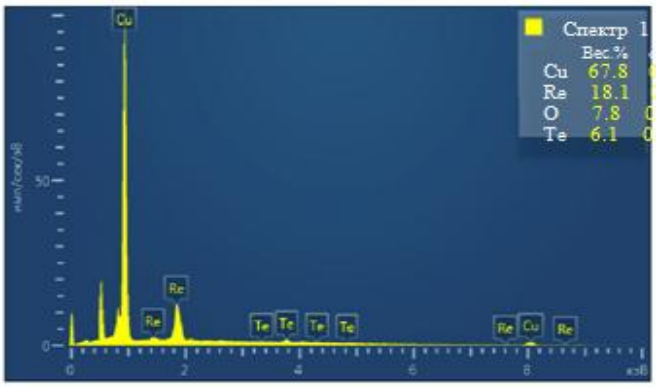
We can thus draw the conclusion that the co-deposition of rhenium with tellurium occurs with minor depolarization. The magnitude of depolarization depends on the energy liberated during the alloy formation.

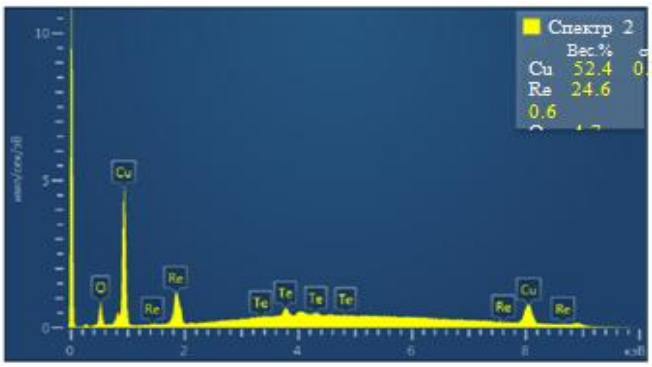
CONCLUSION

Microstructure (morphology) of thin films produced electrochemically in Re–Te–Cu system was studied using SEM microscope on different electrodes: copper, platinum and nickel. It was determined that sizes of thin films are different depending on an electrode material. Thus, these thin films produced from the same electrolyte and in the same current density differ for their sizes and compositions. Sizes of films on platinum electrode are 80–150 nm, but the sizes of thin films on copper electrode vary in the range of 200–350 nm.

ACKNOWLEDGMENT

Development Foundatoin under the Prezident of the Republic of Azerbaijan (Grant № EIF-2013-9(15)-46/19/4) films in the system Re-Te-Cu during 1800 seconds on the platinum electrode at +0,5V potential and t=343ºK





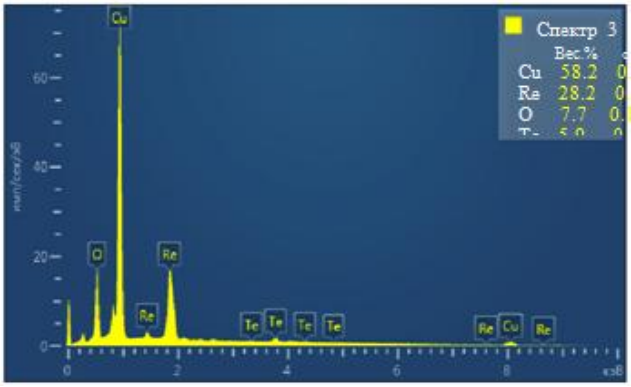


Figure 4: Elemental analysis SEM for obtained thin

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