

The Obtaining of Re-S Thin Films from Thiocarbamide Electrolytes and Influence of Various Factors on Alloy Composition

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Authors' contributions

This work was carried out in collaboration between all authors. Author EAS designed the study, performed the statistical analysis, wrote the protocol and wrote the first draft of the manuscript. Authors AMA and KFI managed the analyses of the study. All authors read and approved the final manuscript.

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ABSTRACT

The joint electrodeposition of rhenium and sulphur from thiocarbamide electrolyte was investigated in this study. By measuring the cycling volt-ampere curves the field of potentials, in the presence of which the joint electrodeposition of rhenium with sulphur takes place has been determined. The joint deposition of rhenium and sulphur was associated with depolarization; additionally, depolarization is powered by the energy produced in the formation of ReS₂ compounds. We examined the influence of current density, thiurea concentration, temperature, and acidity on the composition and quality of cathode sediments. We found that with increasing current density and temperature, the electrolyte concentration of rhenium in the alloy increases; for thiocarbamide, the opposite was observed.

Keywords: *Electrochemistry; electrodeposition; rhenium; sulphur.*

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1. INTRODUCTION

The catalytic properties of rhenium have generated significant interest in the petrochemical industry, in which rhenium has become an important technological tool. The use of rhenium as catalyst in combination with platinum during the oil reforming process resulted in a 5-fold increase in catalyst lifetime and a 60% increase in effectiveness. Alloys containing rhenium are currently used in electronic, electro-measurement, and atomic applications, as well as in the production of manufactured coatings [1-5].

Rhenium alloys containing platinum can be used in the production of electric contacts with high operational properties. The resistance to electro-corrosion that prevents the burning of contacts is a distinctive property of rhenium.

In oil refinement, a combination of rhenium and platinum is used as a catalyst in the production of high-octane benzenes. In addition to the increases in catalyst lifetime and efficiency described above, the number of high-octane benzene molecules increased to 100–105. Thin coatings of rhenium disulphide have been used to catalyze alcohol dehydrogenisation. Interestingly, recent studies have applied rhenium and its alloys in the air and rocket industries. Additionally, rhenium has been used in nuclear studies [7]. The increasing demand for rhenium has resulted in a need for new sources of raw, effective methods of rhenium refinement the development of new methods for the production of rhenium and its alloys.

Rhenium is used in welding processes in the chemical industry as a catalyst. For example, in the US it is recommended to use more than 50% of rhenium as a catalyst [7]. Rhenium shows strong resistance to corrosion in wet environments, is not diluted at room temperature in solutions of hydrochloric, sulphuric and hydrofluoric acids. Hot sulphuric acid reacts with rhenium, transforming it to HReO_4 . Rhenium is diluted in brominated water at slightly elevated temperatures. Rhenium vigorously reacts with halogens to form simple and complex halogenides and oxyhalogenides. Because of its volatility, rhenium halogenides are of interest to the technology and analytical chemistry in industries. The purification of metallic rhenium from other substances after its chlorination is based primarily on the volatility of rhenium chlorides.

Post-processing, e.g. machining of rhenium, may also be difficult because of the high work hardening coefficient of the methods used to obtain rhenium shining metallic sediments as described in previous studies [1-10].

Alloys of rhenium and sulphur are used as photo-sensitive materials in the form of thin coatings in semi-conducting techniques [6]. Additionally, alloys of rhenium with sulphur are used as catalysts in the process of alcohol dehydrogenisation [7].

Various methods are available for obtaining thin films of semi-conductive rhenium alloys. However, an analysis of the main known methods showed that the electrochemical method is the most effective and economically feasible. In previous studies, semi-conducting coatings of Re-Te, Re-S and Re-Se were obtained [11-19].

The use of semi-conducting materials is important in modern techniques. In most semiconductor devices, rhenium alloys are used in thin film form. In the present study, we obtained semi-conducting alloys of the Re-S using the electrochemical method on the

various bases. Cathode process kinetic regularities and the definition of the obtained coverings with semi-conducting properties were of particular interest in this study.

In this study, we attempted to obtain thin Re-S films using electrochemical processes. Electrodeposition offers the advantages of simple equipment, relative ease and low cost and, in some cases, allows of the production of films with controlled composition by adjusting the electrolyte composition and electrolysis conditions.

The influences of pH, electrolyte, concentration of components in solution, velocities of reamer potential and temperature on cathode polarization under electrode position of the Re-S alloys were studied. Electrolytic alloys Re-S were obtained from the electrolytes of the following compositions: $(0.2 \times 10^{-3} - 1.5 \times 10^{-3}) \text{ NH}_4\text{ReO}_4 + (1.23 \times 10^{-3} - 1.5 \times 10^{-3}) \text{ H}_2\text{SO}_4 + (1.5 \times 10^{-3}) (\text{NH}_2)_2\text{CS}$; pH 1–3.5 and current density of 25–45 mA/sm².

2. MATERIALS AND METHODS

Polarization curves were measured using the potentiodynamic method with the use of the potentiometer P-5827 and the regulator PDP4-002. Platinum (Pt) wire and a 0.15 sm² Pt platelet were utilized as the anode and cathode, respectively. For comparison, we used a silver-chlorine anode as an electrode. A constant electrolyte temperature was maintained using a thermostat. The cathode deposit was analysed for Re and S. The deposit was dissolved in concentrated HNO₃ (10 mL) during heating. After boiling in a water bath to one hour, 5N H₃PO₄ was added to the solution, which was then diluted to 50 mL in a volumetric flask. Next, rhenium was separated from sulphide by selective extraction with isoamyl alcohol. Rhenium and sulphide were determined by photometric analysis (FEK-56 M instrument) of rodanide and complexes [2]. Re-S films were characterized by X-ray diffraction (XRD) using a 57.3-mm Debye-Scherrer camera (CuK_α radiation, URS-55 X-ray generator).

3. RESULTS AND DISCUSSION

In order to examine the mechanism of Re-S alloy formation, cycling volt-ampere curves were measured to determine the composition of electrolytes and chemical and x-ray diffractive analysis of cathode sediments was conducted at certain potentials.

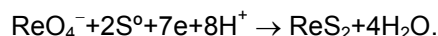
For detailed investigation of the process of obtaining the thin electrolytic coverings of Re-S alloys, information regarding the kinetics of deposition of separate components and alloys is required. Polar curves of Re-S alloys extracts as well as of separate components were determined.

It was found that the cathodic reduction of rhenium began at a potential of +0.28 V; a wave was observed on the polar curves, indicating that the regeneration of rhenium occurs gradually, stage-by-stage. Presumably, the rhenium reduction occurs with the formation of rhenium oxides, which are deoxidized into the metal [5].

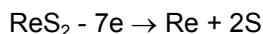
Cycling polarization curves of sulphur on platinum from the thiocarbamide–sulphuric acid electrolyte were formed at pH 1.5. The observed cathode and anode limited currents, corresponding to deoxidation of hydrogen and extraction of oxygen, were indicative of hydrogen sulphide formation.

During concurrent reduction of rhenium by sulphur, the polarization curve for concurrent reduction was shifted to more positive potentials compared to the curves for the separate components, indicating their joint deposition. Additionally, according to the theory of the joint deposition, this type of movement in a polarization curve indicates depolarization of both ions during joint electrodeposition. Depolarization itself is an immediate indicator that at the joint deposition between the components, a chemical interaction occurs with formation of ReS_2 at the cathode.

We hypothesized that the main reason for the shifted rhenium potential in the positive direction was that its joint deposition is related to depolarization, which is caused by alloy formation. Simplifying the reduction of metals during alloy formation was associated with a change in free partial energy of the alloy components. Additionally, the equilibrium potential of components was shifted in the positive direction. According to films analysis data, the process primarily proceeds with the following total reaction:



The characteristics of the anodic cycles also demonstrate the formation of rhenium sulphide. We observed two waves on the polarization curve. Rhenium dissolution and the wave at 0.7–1.2 V shows ReS_2 dissolution according to reaction:



Analysis of cycling voltamperograms provided insight into the mechanism of film deposition. Joint deposition depends on the existence of colloid sulphur in the electrolytes, which stimulates regeneration of rhenium at more positive potentials. When colloid sulphur is present in the solution, its particles are adsorbed on the cathode and they are screened, moving them closer to ReO_4^- to the surface of electrode. The sulphur, adsorbed onto the cathode, interacts with rhenium, creating ReS_2 . Chemical and X-ray phase analysis showed that the composition of a film corresponds to the chemical compound ReS_2 .

We studied the influence of different factors on the composition and quality of rhenium sulphur. As expected, the composition and quality of alloys significantly influences current density. The dependence of Re-S alloy composition on current density at a temperature of 60°C was defined. An increase in current density from 5 to 40 mA/sm^2 increases rhenium content in the alloy from 30–80% (based on rhenium mass) (Fig. 1)

At a current density of 35 mA/sm^2 and electrolyte composition (M) of $1.5 \times 10^{-3} (\text{NH}_2)_2\text{CS} + 1.0 \times 10^{-3} \text{NH}_4\text{ReO}_4 + 1.23 \times 10^{-3} \text{H}_2\text{SO}_4$, small, crystalline, glossy, dark-grey coverings of rhenium sulphur alloys formed with a thickness of 10 μk on the cathode. Based on chemical and X-ray structural analysis, the composition of the obtained alloy corresponds to the chemical compound ReS_2 (74% according to rhenium mass). Thus, with increasing current density to 35 mA/sm^2 , cathode sediment quality was improved and the outlet by the current increases (Table 1).

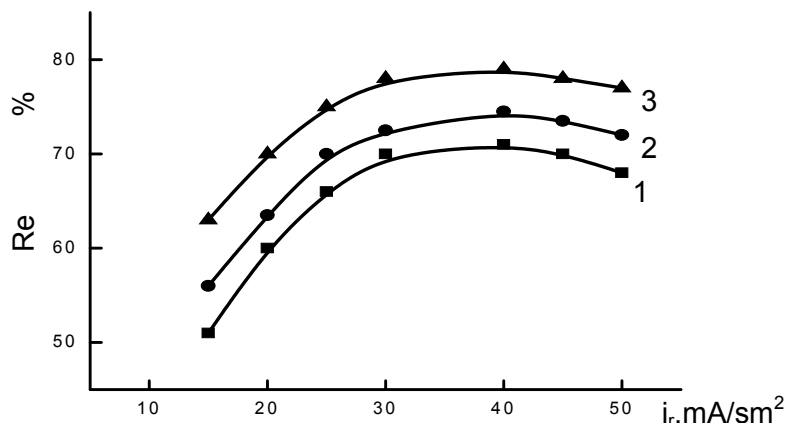


Fig. 1. Dependence of rhenium composition in the rhenium-sulphur alloy on current density at a temperature of 60°C and different concentrations of electrolyte (M): 0.2×10⁻³ NH₄ReO₄+1.23×10⁻³ H₂SO₄+1.5×10⁻³ (NH₂)₂CS; 2–1.0×10⁻³ NH₄ReO₄ + 1.23×10⁻³ H₂SO₄+1.5×10⁻³ (NH₂)₂CS; 3–1.5 × 10⁻³ NH₄ReO₄+1.23×10⁻³ H₂SO₄+1.5×10⁻³ (NH₂)₂CS

Table 1. Dependence of composition and quality of Re-S alloy on current density. Electrode: platinum. Temperature: 60°C

Electrolyte, M			i_k mA/sm ²	Alloy composition, %		Appearance of coatings
H ₂ SO ₄	(NH ₂) ₂ CS	NH ₄ ReO ₄		Re	S	
1.23×10 ⁻³	0.2×10 ⁻³	1.2×10 ⁻³	15	32	68	grey, mat
1.23×10 ⁻³	0.2×10 ⁻³	1.2×10 ⁻³	20	40	60	dark-grey, mat
1.23×10 ⁻³	0.2×10 ⁻³	1.2 ×10 ⁻³	25	48	52	blackish grey, glossy
1.23×10 ⁻³	0.2×10 ⁻³	1.2×10 ⁻³	30	50	50	black, glossy
1,23×10 ⁻³	0.2×10 ⁻³	1.2×10 ⁻³	35	52	48	black, glossy

Alloy composition and quality also depends the concentration of rhenium in the electrolyte. A higher concentration of rhenium in the electrolyte and the cathode deposit determined by the concentration of cations will result in better quality deposits (Table 2).

The influence of the concentration of NH₄ReO₄ on the cathode deposits obtained from thiocarbamide electrolyte is shown in Fig. 2. With an increasing concentration of NH₄ReO₄ and current density, rhenium content in the deposits increased. Thus, for a current density of 30 mA/sm², concentration of thiocarbamide of 0.2×10⁻³ M, and concentration of ammonium perrhenate of 1.2×10⁻³ L, the cathode deposit contains 55% rhenium. Increasing ammonium perrhenate to 2 × 10⁻³ M increased the rhenium content to 65% (by weight).

Table 2. Dependence of alloy Re-S composition on rhenium content in electrolyte. Temperature: 60°C. Cathode: platinum

Electrolyte, M			i_r , mA/sm ²	Alloy composition, %		Appearance of coatings
NH ₄ ReO ₄	(NH ₂) ₂ CS	H ₂ SO ₄		Re	S	
0.2 × 10 ⁻³	1.5 × 10 ⁻³	1.23 × 10 ⁻³	20	40	60	dark-grey, mat
0.6 × 10 ⁻³	1.5 × 10 ⁻³	1.23 × 10 ⁻³	20	47	53	dark-grey, glossy
1.0 × 10 ⁻³	1.5 × 10 ⁻³	1.23 × 10 ⁻³	20	48	52	black, glossy
1.4 × 10 ⁻³	1.5 × 10 ⁻³	1.23 × 10 ⁻³	20	50	50	black, glossy
1.5 × 10 ⁻³	1.5 × 10 ⁻³	1.23 × 10 ⁻³	20	54	46	black, glossy

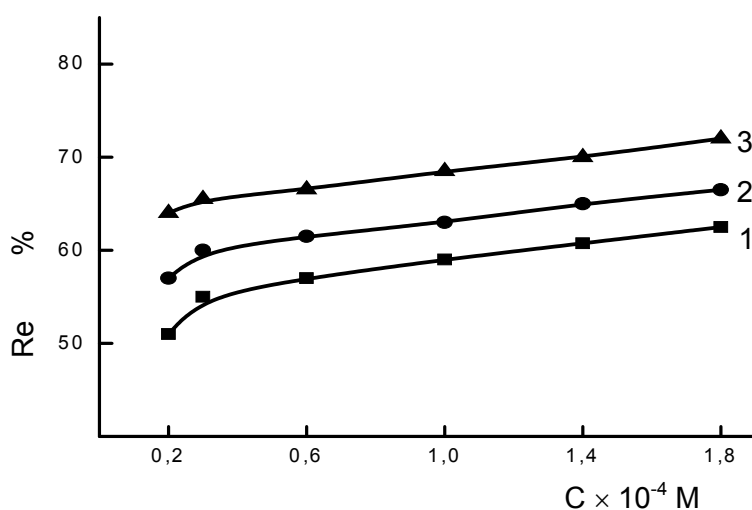
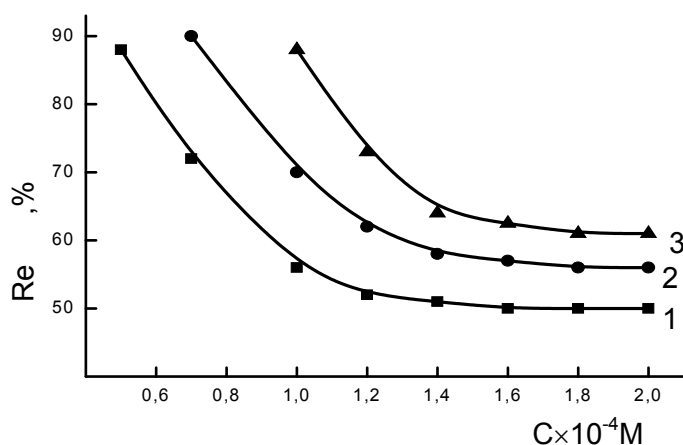


Fig. 2. Dependence of rhenium composition in the alloy rhenium-sulphur on concentration NH₄ReO₄ in the electrolyte on concentration of thiocarbamide 1.5 × 10⁻³ (NH₂)₂CS and 1.23 × 10⁻³ M of H₂SO₄. Current density (mA/sm²) 1–20; 2–30; 3–40 for a temperature of 60°

The concentration of thiocarbamide in the electrolyte also influences alloy content. Increasing thiocarbamide concentration leads a reduction in rhenium content in the alloy (Fig. 3). For example, at a current density 3.0 A/dm² at the cathode, lusterless, small, crystalline thin coverings of ReS alloy were observed that were 15 μk thick and contained 50% rhenium concentration (by mass). A reduced thiocarbamide concentration in the electrolyte changed rhenium content with current (Table 3).

Table 3. Dependence of Re-S alloy on the concentration of thiocarbamide in electrolyte at current density $i_r = 30 \text{ mA/sm}^2$ and temperature of 60°C

Electrolyte, M			Alloy composition, %		OC, Re, %	Appearance of coatings
NH_4ReO_4	$(\text{NH}_2)_2\text{CS}$	H_2SO_4	Re	S		
2.2×10^{-3}	0.5×10^{-3}	1.23×10^{-3}	85.5	14.5	60	black, glossy
2.2×10^{-3}	1.0×10^{-3}	1.23×10^{-3}	68	32	56	black, glossy
2.2×10^{-3}	1.5×10^{-3}	1.23×10^{-3}	54.5	45.5	52	black, glossy
2.2×10^{-3}	2.0×10^{-3}	1.23×10^{-3}	50	50	46	dark-grey, mat
2.2×10^{-3}	2.5×10^{-3}	1.23×10^{-3}	48	52	44	grey, mat

**Fig. 3. Dependence of rhenium composition in alloy rhenium-sulphur on thiurea concentration at different current densities from electrolyte containing $1.2 \times 10^{-3} \text{ NH}_4\text{ReO}_4 \text{ M}$ and $1.23 \times 10^{-3} \text{ H}_2\text{SO}_4 \text{ M}$. Current density (mA/sm^2): 1–30; 2–35; 3–40; temperature, 60°C .**

The influence of temperature on the joint electrodeposition of rhenium and sulphur from the thiocarbamide electrolyte was determined (Fig. 4). The rise in temperature from 20 to 90°C increased rhenium concentration in the alloy from 48 to 75% (by rhenium mass). When the temperature was increased to greater than 60°C , an interaction between colloid sulphur and ammonium perrenate takes place in solution, which dilutes the electrolyte and reduces the number of perrenate molecules entering the electrode reaction for Re-S formation. Thus, the concentration of rhenium in the alloy was reduced and cathode sediment quality decreased. Electrolyte acidity influences the content and quality of alloys (Table 4).

The dependence of cathode sediment composition on the pH of electrolyte was determined. With increasing electrolyte pH, rhenium concentration in the alloy was reduced from 70 to 30% (by rhenium mass). For example, at pH 3 with an electrolyte of composition $2.2 \times 10^{-3} \text{ NH}_4\text{ReO}_4 \text{ (M)} + 1 \times 10^{-3} \text{ (NH}_2)_2\text{CS M}$, at the current density 35 mA/sm^2 and temperature 60°C , thin, lusterless, small crystalline coverings Re-S appeared on the cathode that were $10 \mu\text{m}$ thick with an Re content of 45% (according to mass). Based on our data, electrolysis should not be carried out at pH greater than 3 because cathodic sediments cannot be obtained with a content of 74% , corresponding to ReS_2 . The ReS_2 covering is black in color, has a density

of 8.27 g/sm^2 , is stable in the air and dissolves well in nitric acid with heating but is insoluble in sulphuric and chloric ones.

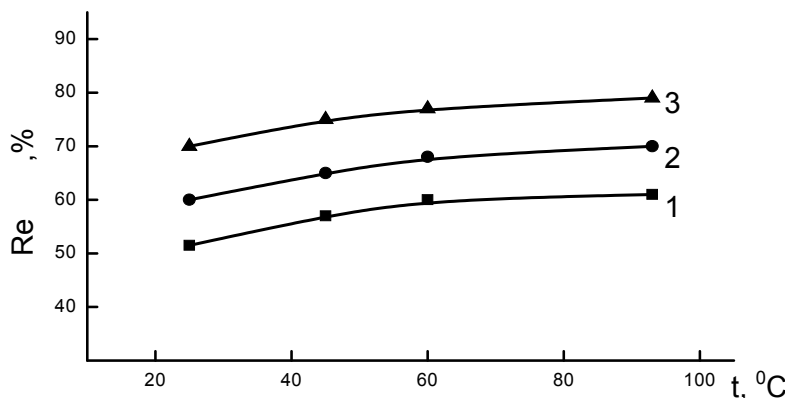


Fig. 4. Dependence of rhenium content in the alloy on the temperature of an electrolyte of composition (M) $2.2 \times 10^{-3} \text{ NH}_4\text{ReO}_4 + 1 \times 10^{-3} (\text{NH}_2)_2\text{CS} + 1.23 \times 10^{-3} \text{ H}_2\text{SO}_4$ at the different current densities (mA/sm^2): 1–25; 2–30; 3–35.

Table 4. Dependence of Re-S alloy composition on electrolyte acidity in solutions containing $2.2 \times 10^{-3} \text{ NH}_4\text{ReO}_4$ (M) $+ 1 \times 10^{-3} (\text{NH}_2)_2\text{CS}$ M at $i_k = 35 \text{ mA/sm}^2$; temperature, 60°C

Alloy composition		pH of solution	OC, Re%	OC, alloy%	Appearance of coatings
Re	S				
73	27	1.0	60	95	black, glossy
68	32	1.5	58	94	black, glossy
64	36	2.0	54	92	blackish-grey, mat
60	40	2.5	50	90	dark-grey, mat
56	44	3.0	48	90	dark-grey, mat

To examine how to prevent the formation of a crystalloid structure on the thin coverings composed of ReS_2 , X-ray graphical investigation was conducted.

According to RPA, ReS_2 complexes crystallized in a triclinic symmetry with the parameters of an elementary cell: $a = 6.455 \text{ \AA}$, $b = 6.363 \text{ \AA}$, $c = 6.401 \text{ \AA}$.

Thus, based on our experimental data, the optimum preparation method and composition of electrolyte was determined for obtaining qualified semiconducting coverings of rhenium-sulphur alloy containing 73–76% rhenium (by rhenium mass) and 24–27% sulphur, which is very similar to the ReS_2 complex. Using the thermoprobe method, it was established that all alloys of rhenium sulphide possess p-type conductivity.

We examined the microstructure of a cathodic deposit of Re-S obtained by electrodeposition from sulfate thiourea solutions containing (M): $2.2 \times 10^{-3} \text{ NH}_4\text{ReO}_4$, $1.0 \times 10^{-3} (\text{NH}_2)_2\text{CS}_2$ and $1.23 \times 10^{-3} \text{ H}_2\text{SO}_4$. Analysis of the influence of electrolysis conditions on the microstructure of obtained films confirmed the suggested mechanism of joint sedimentation of rhenium with sulphur.

Changes in electrolyte pH and current density influenced sedimentation. At low pH values, colloidal sulphur formed in the electrolyte was precipitated onto the cathode.

Depending on the conditions, at a low current density, the multiphase precipitation was a dark color containing brown, red and blue fragments, with yellow crystal dissemination (Fig. 5).

At a low current density, insignificant quantities of Re_2S_7 (brown color), ReO_2 (red color), Re_2S_5 and S elementary (yellow color) were produced during electroreduction. Formation of rhenium oxides and Re_2S_7 occurred because the low current density delivery of perrhenate ions to the surface of the cathode and their discharge was complicated.

The availability of crystal sulphur was clearly correlated with the observation that at low current density, sulphur reduction to sulphide ion did not occur.

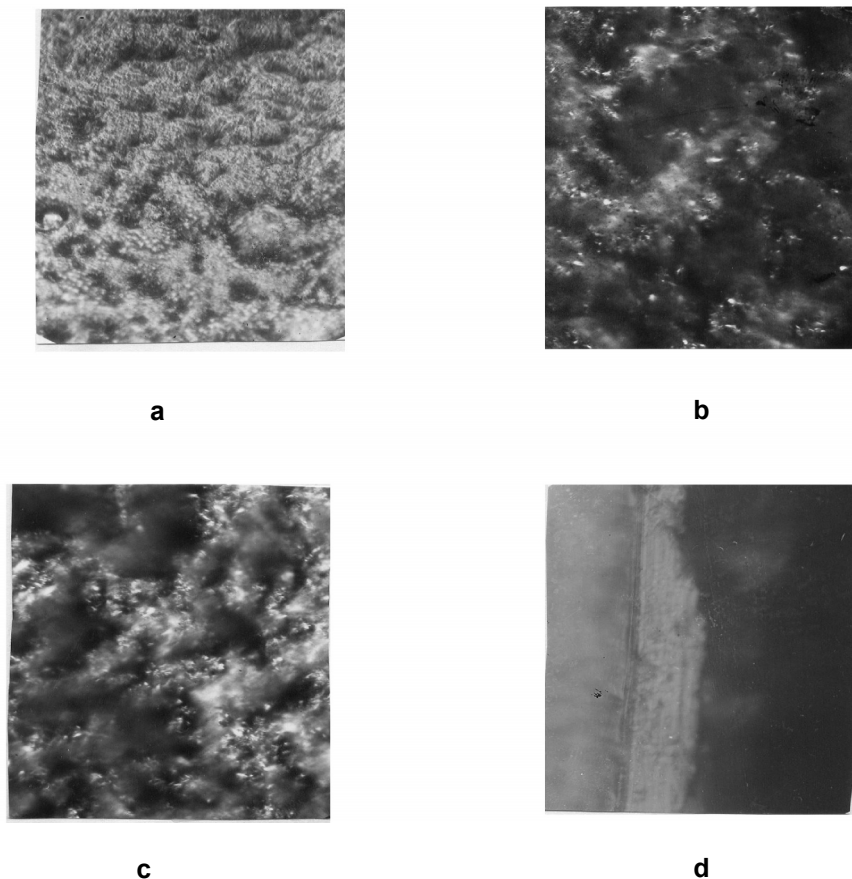


Fig. 5. Microstructure of cathodic precipitation of an ReS_2 alloy obtained at various current densities (mA/sm^2): (a) 30; (b) 35; (c) 40; (d) microphotography of cross section of the platinum cathode with an alloy of ReS_2 structure; ($\times 500$ magnification).

Increasing current density changed the characteristics of the film surface. At a current density of $30 \text{ mA}/\text{sm}^2$ (under optimum conditions), the film appeared to be a single-phase uniform micro crystallographic deposit of rhenium disulphide. Under these conditions, the

rate of ReO discharge was not limited by diffusion, and the crystallization rate was satisfactory. With a further increase in current density, the precipitates remained in a single-phase state, but in this case they were characterized by friability. Increasing thiocarbamide concentration in the electrolyte resulted in the formation of semicrystalline sulphur as yellow threads (Fig. 5) which were coiled into a ball and formed on the surface of an electrode. Increased temperature to higher than 80°C also led to decreased precipitate quality. The microphotography of a cross-section of the platinum cathode with a deposit of the following composition, wt %: Re = 74.4; S = 25.6 showed that the deposit consisted of one phase (Fig. 5) and its structure corresponded to the ReS₂ compound.

4. CONCLUSION

1. We examined the influence of current density, thiurea concentration, temperature and acidity on the composition and quality of cathode sediments. We found that with increasing current density and temperature, the electrolyte concentration of rhenium in the alloy increases; for thiocarbamide, the opposite was observed.

2. Thus, based on our experimental data, the optimum preparation method and composition of electrolyte was determined for obtaining qualified semiconducting coverings of rhenium-sulphur alloy containing 73-76% rhenium (by rhenium mass) and 24-27% sulphur, which is similar to the ReS₂ complex.

3. The microphotography of a cross-section of the platinum cathode with a deposit of the following composition, wt %: Re = 74.4; S = 25.6 showed that the deposit consisted of one phase and its structure corresponded to the ReS₂ compound.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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