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MODERN METHODS OF AMALGAMATION OF LOW SOLUBE METALS AND ALLOYS: CONTRIBUTION TO SUSTAINABLE DEVELOPMENT AND ENVIRONMENTAL PROTECTION (SDG 12)

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Abstract. In connection with the task of accelerated implementation of the latest achievements of science and technology in the national economy, it is necessary to develop new types of electrical devices and to improve the existing ones. In turn, this requires the modernization of the known magnetically controlled contacts (MCC), and, accordingly, the implementation of the works, directed to the search for new structural materials, studying the behavior of various metals and alloys under the operating conditions of devices, etc. When developing new technical solutions for switching equipment for automation and control, special attention has been paid to the possibility of miniaturization of the relay elements, based on mercury MCC. This work also focuses on sustainable development and environmental protection, which corresponds to the UN Sustainable Development

Goals (SDGs), in particular, SDG 12 (Responsible Consumption and Production). The work focuses on the amalgamation of metals and alloys, which are low soluble in mercury, and on assessing the stability of the resulting mercury coatings, which are planned to be used in production, which emphasizes the importance of studying the stability of the result of the amalgamation process. It has been established that the application of the developed method of amalgamation of metals and alloys, which are low soluble in mercury, allows a high-quality, stable mercury coating to be obtained on the materials, which are promising for the manufacture of contact parts for reed switches, beds of nails, as well as mercury-film indicator microelectrodes, etc.

Keywords: mercury film, amalgamation, contact parts, reed switches, microelectrodes, electrolysis

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ЕРІМЕЙТІН МЕТАЛДАР МЕН ҚОРЫТПАЛАРДЫ АМАЛЬГАМАЦИЯЛАУДЫҢ ЗАМАНАУИ ӘДІСТЕРІ: ТҰРАҚТЫ ДАМУ МЕН ҚОРШАҒАН ОРТАНЫ ҚОРҒАУҒА ҚОСҚАН ҮЛЕСІ (ТДМ- 12)

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Аннотация. Ғылым мен техниканың соңғы жетістіктерін халық шаруашылығына жедел енгізу міндетіне байланысты электр аппараттарының жаңа түрлерін дамыту және қолданыстағыларын жетілдіру қажет. Өз кезегінде, бұл белгілі магнитті контактілерді (МК) жаңартуды және сәйкесінше жаңа құрылымдық материалдарды іздеу, аспаптарды пайдалану жағдайында әртүрлі металдар мен қорытпалардың әрекетін зерттеу және т.б. жұмыстарды орындауды талап етеді. Автоматика мен басқарудың коммутациялық аппаратурасының жаңа техникалық шешімдерін әзірлеу кезінде сынап МК негізінде релелік элементтерді миниатюризациялау мүмкіндігіне ерекше назар аударылды. Бұл жұмыста БҰҰ-ның Тұрақты даму мақсаттарына (ТДМ), атап айтқанда ТДМ 12 жауапты тұтыну және өндіріс бағытына сәйкес келетін тұрақты даму мен қоршаған ортаны қорғауға баса назар аударылады. Жұмыста сынапта ерімейтін металдар мен қорытпаларды амальгамациялауға және өндірісте пайдалану жоспарланған сынап жабындарының тұрақтылығын бағалауға баса назар аударылады, бұл амальгамация процесінің нәтижесінің тұрақтылығын зерттеудің маңыздылығын көрсетеді. Сынапта еруі қиын металдар мен қорытпаларды амальгамациялаудың әзірленген әдісін қолдану геркондар, контактрондар, мифистерлер, сондай-ақ сынап-пленкалы индикаторлық микроэлектродтар және т.б. үшін байланыс бөлшектерін өндіруде перспективалы материалдарда сапалы, тұрақты сынап жабынын алуға мүмкіндік беретіні анықталды.

Түйін сөздер: сынап пленкасы, амальгамация, байланыс бөлшектері, геркондар, микроэлектродтар, электролиз

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СОВРЕМЕННЫЕ МЕТОДЫ АМАЛЬГАМИРОВАНИЯ ТРУДНОРАСТВОРИМЫХ МЕТАЛЛОВ И СПЛАВОВ: ВКЛАД В ДОСТИЖЕНИЕ ЦЕЛЕЙ УСТОЙЧИВОГО РАЗВИТИЯ И ЗАЩИТУ ОКРУЖАЮЩЕЙ СРЕДЫ (ЦУР 12)

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Аннотация. Современное развитие электрохимических технологий требует создания новых методов обработки труднорастворимых металлов и сплавов, обеспечивающих повышение эксплуатационных характеристик контактных материалов. Одним из перспективных направлений является амальгамирование, позволяющее формировать устойчивые ртутные покрытия на поверхностях металлов. В работе рассмотрены современные подходы к амальгамированию труднорастворимых в ртути металлов и сплавов, а также оценена устойчивость полученных ртутных покрытий, предназначенных для использования при производстве контактных элементов реле, сенсоров и микроэлектродов. Особое внимание уделено вопросам устойчивого развития и охраны окружающей среды в контексте Цели устойчивого развития № 12 «Ответственное потребление и производство». Применение разработанных методов амальгамирования обеспечивает получение качественных и стабильных ртутных покрытий, что способствует снижению потерь металлов и уменьшению экологической нагрузки при производстве. Полученные результаты могут быть использованы в области материаловедения и химической технологии для разработки экологически безопасных технологий нанесения покрытий.

Ключевые слова: ртутная пленка, амальгамирование, контакт-детали, герконы, микроэлектроды, электролиз

Introduction. Nowadays, the development of effective methods for amalgamation of the surface of metals and alloys, which are low soluble in mercury is relevant. As already mentioned, the amalgamation process is always preceded by wetting their surface with mercury, which is especially difficult to implement for the metals, which are low soluble in mercury and have a high surface energy under the normal conditions (Jaycock et al, 1984; Adam et al, 1947). It has been found that mercury on molybdenum or stainless steel, forms island drops of metallic mercury. The structure of electrodeposited mercury on the surface of the difficult-to-amalgam metals has been determined by the thermal evaporation of mercury. Unfortunately, the method itself is an environmental hazard, since it promotes the evaporation of mercury into the environment and does not guarantee the formation of a stable mercury film on the surface of the refractory metals (Zenko et al, 2006). Currently, the UN Sustainable Development Goals (SDGs), in particular SDG 12, place particular emphasis on environmental protection. In order to solve the problems of ecologically rational use of the proposed modern methods of forming mercury-film coatings on the surface of the difficult-to-amalgam metals and alloys, in accordance with the agreed international principles, it is necessary to significantly reduce the release of toxic heavy metals into the air, water and soil in order to minimize their negative impact on human health and the environment (Baitasheva et al, 2024).

All the described methods are quite complex and environmentally hazardous. In this regard, their use for amalgamation of the metals and alloys, which are low soluble in mercury, is not desirable. The amalgamation of metals with high surface energy presents significant difficulties. Such metals include transition metals (Fe, Co, Ni, Zr, Hf, Th, V, Nd, Ta, Cr, Mo, W, Mn, Tc, Re, Ti) and alloys based on them. To carry out their amalgamation, it is necessary to create special conditions for the surface treatment, such as bright annealing, ion bombardment, heating in a vacuum up to 773 K, etc. (Lang et al, 2013). The efficiency of the amalgamation of such metals and alloys can be improved by using alkali metal amalgams instead of mercury, most often sodium (Kunin et al, 1955). Various metals have been considered as potential materials as a substrate for the formation of a mercury film. It has been found that only iridium has the desired properties. A complete procedure for the formation of a stable Hg film on iridium has been described (Kounaves et al, 1986). The process of preliminary preparation of the iridium substrate surface can be applied only to this particular metal. Other metals have been also considered as potential electrode substrates, but without result. Various metals have been considered as potential electrode substrates, but it has been found that only iridium has the desired properties as a mercury film substrate (Kounaves et al, 1986). Only after several, rather complex, preliminary preparation procedures of the iridium surface it has been recommended to optimize the conditions for the formation of mercury films in 0.1 M HCIO as an electrolyte. When amalgamating transition metals, especially the iron family metals and their alloys, the main obstacle to the formation of a mercury film is the presence of a dense layer of oxides, adsorbed gases or other contaminants on the surface of the material. Therefore, the preliminary preparation of the material surface is required before the amalgamation (Adamson et al, 1979), i.e. special treatment before a direct contact with mercury. In some cases, the both stages - preparation and the amalgamation process itself - can be combined. For the metals, which are difficult to amalgam, a preliminary deposition of a thin layer of metal, which is well wetted by mercury, or its intermetallic compound with mercury is often used on their surface. The metals such as Sn, Au, Cu are usually used as a coating (Semenchenko et al, 1967). There are known cases of using palladium for these purposes (Kalnin et al, 1968). Metals are applied to the surface of the samples by electrolysis or cementation (Jaycock et al, 1984; Deryagin et al, 1973).

Metal amalgamation can also be carried out, using cathodic polarization. In this process, hydrogen released at the cathode reduces the oxide film, and the surface, cleaned in this way, is better amalgamed upon the subsequent contact with mercury (Semenchenko et al, 1967). The method in which the stages of the surface preparation and amalgamation occur simultaneously is recommended for iron, nickel and alloys based on them (Kalnin et al, 1968). The surface of the sample is treated with mercury in a hydrochloric acid solution (10%), to which metallic magnesium is also added (Mg: Hg = 1:3000). In this case, the reaction of magnesium with the acid actually performs the function of cathodic polarization. The hydrogen, formed as a result of the reaction, promotes the reduction of oxides on the metal surface, which is immediately well wetted by mercury (Jaycock et al, 1984; Deryagin et al, 1973). This method has been subsequently adapted for the amalgamation of manganese, chromium, tungsten, molybdenum and their alloys (Ainbinder et al, 1962). The amalgamation conditions have remained basically the same, but ferric chloride (~0.2%) has been added to the solution to enhance the process. However, these methods do not allow one to achieve stable results on all of the above materials, since low resistance of the coating in air and insufficient uniformity of mercury distribution over the surface of the samples are observed.

Metals with high inertness to mercury, the solubility of which is within thousandths, ten-thousandths and lower fractions of a percent, have turned out to be interesting for science and technology. The physicochemical studies of these metals due to their low solubility in mercury over a wide temperature range have not attracted sufficient attention from the researchers. Nevertheless, the metals inert to mercury are indispensable materials when liquid metal comes into contact with the equipment in the technological processes where mercury is used. It is known that mercury has found application as a heat carrier in the chemical, oil industry and nuclear power engineering.

Mercury has also found wide application as a conductive material in various designs of devices with electrical contacts. The contacts made of mercury-resistant metals and alloys are an integral part of the automatic control and regulation systems in the creation of telephone exchanges, in computing and measuring devices, satellite communications, without which the development of scientific and technological progress is unthinkable (Semenchenko et al, 1967). Currently, in modern technology, switching microdevices with liquid metal contacts are mainly represented by mercury reed switches. When manufacturing reed switches, much attention is paid to the choice of an effective amalgamation method to obtain a high-quality mercury film uniformly distributed over the entire working surface of the contact. It is the high quality of the resulting mercury film that has a positive effect on increasing the service life of the device, ensuring its stable operation under various conditions. Another important characteristic in the manufacture of reed switches is the correct choice of metals used in the devices. This requires reliable data on the solubility of metals in mercury in the temperature mode of the operation of the device, including the high temperature range. To obtain a high-quality mercury film applied to the surface of the metal, it is necessary to pre-clean their surface with acids (etching, polishing, etc.).

A clean metal surface ensures high-quality mercury film, thereby extending the life of the device. The surface cleanliness is also very important when determining the solubility of metals in mercury, using the heterogeneous equilibrium method by holding the metal in pure mercury for a long time at a given temperature. Chemical cleaning of the metal, removing the oxide from its surface, prevents it from getting into the mercury, thereby preventing distortion of the data on the solubility of the metal in mercury. Another equally important condition is the duration of holding the metal in mercury. Under the conditions of a long-term contact of the metal with mercury, true equilibrium is achieved between the solid and dissolved metal in mercury, which ensures reliable data on the solubility of metals in mercury. The researchers involved in determining the solubility of metals with low solubility in mercury have not paid due attention to these conditions (Bukhman et al, 2010).

A u nit has been designed to measure the surface potential of the adsorbed vapors on mercury. It has been shown that the potential change is 0.055 V with a phase change associated with a change in the orientation of the toluene molecules, adsorbed on the mercury surface. Carbon tetrachloride, hexachloroethane and chloroform vapors have reacted with mercury and the reaction rate has been determined by the accompanying change in the surface potential, which in the case of carbon tetrachloride has been more than 1 V. It has turned out that methyl iodide vapors react with mercury only when illuminated by a mercury lamp. It has been assumed that methyl iodide dissociates into the radicals, which in turn react with mercury (Kemball et al, 1950). In our case, this method is unacceptable, since it is assumed that illumination by a mercury lamp is used and the reaction rate depends on the square root of the light intensity, which inevitably leads to the evaporation of mercury into the environment. In connection with the need to implement the latest achievements in science and technology in the national economy, it is necessary to develop new types of electrical devices, as well as to improve existing ones (Mussina et al, 2018). These tasks can be accomplished by performing work on searching for new structural materials, studying the behavior of various metals and alloys in mercury under the operating conditions of devices, etc. At the same time, the latter, in the presence of a liquid component filling most of the volume of the MCC, provide increased durability, improved vibration resistance and noise immunity, as well as the possibility of convenient arrangement in the structure of the micromodules.

Research materials and methods.

Materials under study:

– steel of various grades: 13Х36НХТ10, 12ХН10Т, Р-6, 79НМ, 67КН5Б;

– alloys 47НД, monel 40A, nickel silver.

Method of amalgamation of alloys based on iron, nickel and cobalt

The surface of alloys subject to amalgamation contains various types of contamination (oxides, sulphides, organic impurities). To clean the contact surface, a chemical treatment method is used – etching. The metal surface is preliminarily degreased in carbon tetrachloride for 10-15 minutes, dried in air, degreased in an alkaline solution, containing 10-30 g/l of caustic soda and 50-70 g/l of trisodium phosphate at the temperature of 70°C for 30 minutes, and washed with distilled water. Then the contacts are etched in acidic solutions: pre-treated in solution 1 (composition H_2SO_4 d=1.84 g/cm^3 two parts, HCl d=1.19 g/cm^3 one part and

H_2O two parts) at the temperature of 30-40°C for 90 seconds, then in solution 2 (composition HNO_3 d=1.42 g/cm^3 one part and H_2SO_4 d=1.84 g/cm^3 one part) at the temperature of 20°C and t = 15 sec; and in solution 3 - (HCl d=1.19 g/cm^3), treatment time 45 sec at t = 20°C.

An electrolyte of the known composition has been used to deposit mercury on the surface of the contact materials (Certificate of Authorship 840206 USSR et al, 1981). A typical electrochemical analytical system mainly consists of the following three parts: an electrochemical detection device, an electrochemical detection instrument and an electrolyte. An electrochemical detection device usually consists of the following three electrodes: a working electrode (WE), a reference electrode (RE) and a counter electrode (CE).

Figure 1 shows a diagram of the electrolytic deposition of a mercury film on the surface of the difficult-to-amalgam metals and alloys.

1. Electrochemical cell:
2. Reference electrode;
3. Intermediate vessel;
4. Stabilizer;
5. Autotransformer;
6. Rectifier;
7. Ammeter;
8. Switch;
9. Potentiometer;
10. Potentiostat (P-5848, P-5827) In this case, the positions - 4,5,6,7 are excluded;
11. Self-recording potentiometer.

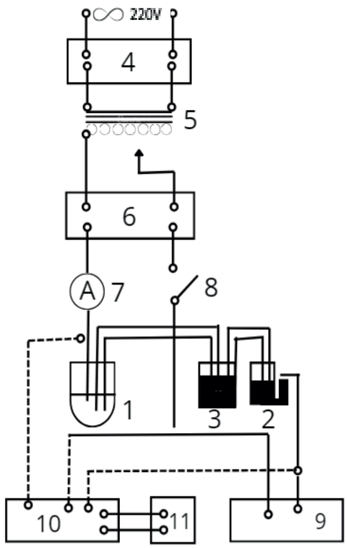


Figure 1 - Schematic diagram of the amalgamation unit

A similar representative illustration of the sample preparation is also shown in the work, where the main subsequent stages are shown (*Lu Y* et al, 2018). In our case, special attention is paid to the nature of the electrode used, which can significantly affect the sensitivity and selectivity of the analytical procedure.

Since the nature of the amalgamated material is of virtually no importance in determining the role of the geometric parameters of electrolysis (the ratio of the cathode area to the electrolyte volume, the distance between the electrodes, etc.), new materials promising for the manufacture of the contact parts with a mercury-film coating have been used in the experiments. The electrolysis conditions have been tested on the hard-to-amalgam samples of the materials under study.

Before electrolysis, the samples have been degreased and chemically prepared (etched, polished). Mercury electrodeposition has been carried out in an electrolyte of a certain volume (V_{el}) on a sample of area (S_c), using an anode in the form of a Pt-wire of area (Sa), while maintaining a stable distance between the cathode and anode (l_{a-c}) and certain ratios S_c:S_q and S_c:V_{el}.

Methodology for determining the quality of the mercury coating on the samples. The mercury coating deposited on the contact material by the electrolytic method has been assessed mainly by two parameters – the thickness of the mercury layer (δ), its stability in air under the mechanical action on the sample, the continuity of the film and the presence of metallic luster characteristic of liquid mercury.

To increase the sensitivity and selectivity of the detection of heavy metal ions and to study their toxicity both for the environment and for human health, we combine the electrochemical methods with certain modifiers, which facilitate the detection process. Due to their characteristic properties, electrode modifiers have been used in combination with the electroanalytical methods. The need for alternative electrode modifiers has increased as attention has increasingly turned to green chemistry, which aims to reduce the use and production of hazardous substances such as mercury (*Armenta* et al, 2008), and as some European regulations prohibit the export and storage of metallic mercury.

The creation of mercury-film contact parts and microelectrodes with a mercury-film coating makes it possible to work under the conditions with a minimum mercury content, i.e. transfer the properties of mercury to the film, which is well bonded to the surface of the substrate material.

The amalgamed materials have been centrifuged, using a T-23 centrifuge. Three centrifugal acceleration modes have been used: A1 = 580 m/sec^2, A2 = 3560 m/sec^2 and A3 = 9950 m/sec^2 (at the corresponding number of revolutions: 600, 1500, 2500 rpm and radius R = 0.145 m) (Geirovskiy et al, 1965). The thickness of the mercury layer on the surface of the sample has been calculated based on the weight of the coating and the area of the amalgamed contact. The weight of the sample was determined after each of the stages - amalgamation and centrifugation. The stability of mercury coatings has been assessed based on the visual observations of the condition of the samples under the conditions of exposure to air for 30-60 minutes.

In addition, observations have been made of the condition of the contact surface at equal stages of their processing, using a MIM-7 microscope (magnification 140-120 times) with the fixation on a photographic film at the specified magnification.

Experimental part. In connection with the task of the SDG-12, it is necessary to qualitatively develop new types of electrical devices and to improve the existing ones. In turn, this requires the creation of new magnetically controlled contacts (MCC), and, accordingly, the performance of the work on the search for the structural materials, the study of the behavior of various metals and alloys under the operating conditions of the devices in production. When developing new technical solutions for the switching equipment for automation and control, special attention is drawn to the possibilities of miniaturization of the relay elements, based on mercury ball and membrane MCCs.

These tasks can be accomplished by performing the work for searching for new structural materials, studying the behavior of various metals and alloys in mercury under the operating conditions of the devices, etc. At the same time, the latter, in the presence of a liquid component filling most of the MCC volume, provide increased durability, improved vibration resistance and noise immunity, as well as the possibility of convenient arrangement in the structure of micromodules. In this regard, of interest are the studies of the processes of interaction with mercury of new alloys, assumed as the contact materials for the devices, a more detailed study of the conditions of amalgamation of the metal surfaces by the electrolytic method. At the first stage of the study, the possibility of forming mercury coatings on materials during cathodic reduction from the solutions of various mercury salts - nitrates, chlorides, and sulfates – has been determined. As is known, nitric acid solutions (sat. $Hg(NO_3)_2$); 0.1M HNO_3 + 5.10-4M $Hg(NO_3)_2$ are used to reduce mercury on platinum, where the deposition process begins at the potentials of +0.4 + 0.5V (N.H.E.), respectively, and starting from E = 0.58V is accompanied by the release of hydrogen. The mercury coating, formed in this case, is of sufficient quality, but quickly deteriorates after the current is switched off upon the contact with the working electrolyte due to the oxidation by the divalent mercury ions. We have tested this method for obtaining a mercury film on the iron group metals. Mercury deposition on nickel, alloy 29NK in the solution: 0.2-0.3M $Hg(NO_3)_2$ + 1M KNO_3 + 0.013 – 0.625M HNO_3 has resulted in only partial amalgamation, since the formation of metallic mercury has been accompanied by the release of a gray precipitate, which has shielded the surface of the samples.

In the chloride solutions (0.05M $HgCl_2$ + 1M HCl + 1M NaCl), the mercury coating is not formed, the reduction product precipitates into the solution in the form of suspensions of a poorly soluble salt of monovalent mercury. In this regard, the choice has been made in favor of the sulfuric acid medium, as a less aggressive one, and the electrolyte composition has been selected based on the solubility of the mercury salt, as well as ensuring the required electrical conductivity and optimal acidity of the solution:

$0.054M\ HgSO_4 + 0.5–1M\ H_2SO_4 + 1M\ Na_2SO_4$.

One of the main objectives of the work has been to develop conditions for the implementation of a stable electrolysis process during mercury deposition on the surface of the contact materials - parts of the magnetically controlled devices. In this regard, it has been necessary to determine the parameters of the optimal process, the degree of depletion of the electrolyte during the period of operation when obtaining sufficiently stable mercury coatings, as well as the possibility of cleaning waste solutions and industrial waters. The proposed material covers these issues.

For the first time, the characteristics of mercury coatings have been studied on the samples of cobalt alloys, Х18Н10Т, 12ХН10Т, 40КХНМ, 67КН5Б, 79НМ steels, when their surface has been only etched before the electrolytic deposition of mercury. When processing metals and alloys at the stages of chemical preparation of their surfaces before applying metal, in particular, mercury coatings, the composition and microrelief of the surface layer of the studied samples changes (Mussina et al, 2024).

Table 1 shows the parameters of mercury coatings (film thickness and stability in air) obtained on the samples of the specified materials, with different electrolysis parameters. The ratio S_c:V_{el} at l_{a-c} = const and S_c:S_a = const is significant during electrolysis.

First of all, it should be noted, as previously established, that at $l_{a-c} < 8$ cm, a stable mercury coating is not formed on the samples. Probably, at low l_{a-c}, due to strong heating of the electrolyte in the working area, strong adhesion of the deposited mercury to the surface of the material is not ensured, and mercury in the liquid form rolls off the cathode. It follows from this that only at l_{a-c} of at least 3 cm is a high dissipating capacity of the electrolyte achieved. Maintaining l_{a-c} = 3 cm and the optimal ratio S_c:S_q = 1:1, we carried out electrolysis with a change in the S_c : V_{el} from 1:30 to 1:120, monitoring the temperature in the working area. As can be seen from Table 1, the most optimal S_c:V_{el} > 100, since this does not cause undesirable heating of the electrolyte in the working area, the temperature is stably maintained at 25-30°C and it is possible to repeatedly use the original volume of the solution. It has been found that the working current density on the cathode is not the same for different materials. The values of τ, mA/cm^2, given in Table 1 have been selected as optimal: setting higher τ, on the one hand, is not advisable due to the increase in the voltage on the bath (I), and on the other hand, this leads to obtaining coatings, which are less stable in air. Probably, too intense hydrogen evolution contributes to the saturation of materials with a gaseous product of electrolysis. The current efficiency of metallic mercury is low. Since the working current densities of 200 - 600 mA/cm^2 cause the establishment of a very negative potential >-1.6 V, mercury is released at a maximum current of about 2.5-4 mA/cm^2, respectively, VT 2-4%. The main part of the current is related to the process of hydrogen evolution, which activates the surface of the material during cathodic polarization in Table 1..

Table 1 – The indicators of mercury coatings on various materials, obtained by electrolytic method in an electrolyte of the composition: 0.054M $HgSO_4$ + IM Na_2SO_4 +0.55M H_2SO_4

Cathode area, S cm^2	мA/ cm^2	S_{cat} ; V_{el}	S_{cat} ; S_{an}	Repeated use of the electrolyte	Thickness of the mercury film δ .10^3cm		Air stability
					after electrolysis	after centrifugation, 1500 rpm	
1	2	3	4	5	6	7	8
1. Mercury precipitation conditions: V_{el}=50ml. τ=1hour. l_{a-c} =3cm., S_{an}=0.43 cm^2 t=40-50°C. U=1.5-1.8 B.							
36HXT10							
0.43	256	1:70	1:1	1-0	2.3	0.23	60min.
0.60	1,83	1:50	1:0.7	2-0	2.4	0.22	< 2min.
1.08	102	1:28	1:0.4	1	3.7	0.62	120min.
1.04	106	1:29	1:0.4	2	5.8	1.61	< 2min.
12XH10T							
2. Mercury precipitation conditions: V_{el}=50ml. τ=1hour. l_{a-c} =3cm., S_{an}=0.28 cm^2 t=30-40°C. U=1.5-1.8 B.							
0.49	571	1:102	1:0.6	1	22.6	2.9	39 days
0.54	519	1:98	1:0.5	2	20.7	2.6	38 days
0.59	475	1:85	1:0.5	3	26.4	2.1	40 days
0.72	278	1:59	1:0.4	1	6.4	2.7	20 days
0.69	289	1:72	1:0.4	2	6.8	2.4	20 days
0.72	278	1:69	1:0.4	3	4.2	2.7	20 days
79 HM							
3. Mercury electrodeposition conditions: V_{el}=50ml. τ=1hour. l_{a-c} =3см., S_{an}=0.43 cm^2 t=25-30°C. U=1.6:1.8 B.							
1.0	300	1:60	1:0.4	1	16.8	3.7	2.5 months
0.9	333	1:67	1:0.5	2	13.7	4.9	2.5 months
1.1	273	1:55	1:0.4	3	3.9	3.4	2.5 months
40KXHM							
0.6	500	1:100	1:0.7	4	2.3	2.0	10 days
0.5	600	1:120	1:0.8	1	11.0	2.0	10 days
0.5	600	1:120	1:0.8	2	14.1	1.8	10 days
67 KH5Б							
0.5	600	1:120	1:0.9	3	5.8	3.8	2.5 months
0.5	600	1:120	1:0.9	4	5.1	3.5	2.5 months
0.5	600	1:120	1:0.9	5	3.6	3.5	2.5 months

It has been established that during electrolysis, mercury ions are mainly produced from the working electrolyte, the acidity changes insignificantly, and the content of SO_4^{2-} ions, due to the high concentration, remains virtually unchanged.

Thus, the optimal parameters for the electrodeposition of mercury on the surface of various contact samples in the solution are: τ = 200 – 300 mA/cm^2, depending on the type of the material; S_c:S_q = 1:1, S_c:V_{el} > 100 < 120, t = 20-30°C. In addition, the following should be noted:

1. Due to the fact that the electrolyte is an extremely aggressive environment, the material samples, while in it, without cathodic protection, are subject to

severe corrosion. In this case, their surface is passivated, and the components of the amalgamated material accumulate in the solution. To avoid this undesirable phenomenon, when placed in the solution, the sample should be connected to the electrodes under the voltage of at least 1V. In addition, it has been noted that it is not advisable to use the same volume of electrolyte for different materials.

2. When conducting electrolysis in an electrolyte of the composition 0.054M $HgSO_4$ + 1M Na_2SO_4 + 0.55M H_2SO_4, oxygen is released at the anode. No processes in the volume of the electrolyte during the electrolysis period take place, which follows from the general ideas on the chemical properties of the solution we use.

Thus, based on the literary data and the results of our experiments, we can conclude that the harmful impurities in obtaining high-quality mercury coatings on the surface of difficult-to-amalgam materials are the presence of anions in the solution - NO^{3-} and Cl^- and metal cations – the components of the amalgamated alloys.

Results and discussion. The working range of the electrolyte concentration for mercury ions and other components of the studied materials is considered. Due to the fact that mercury is deposited on the surface of the samples during electrolysis, the electrolyte is primarily depleted in mercury ions. The parallel process of hydrogen evolution at the cathode should contribute to a decrease in the acidity of the solutions. At the same time, the anionic composition of the solution (SO_4^{2-} - ion), as already indicated above, does not change.

To clarify the working range of the electrolyte concentrations by components, additional studies have been conducted to determine the lower concentration limit of the mercury ion solution, which ensures the obtaining of a stable mercury coating. The most difficult amalgamed material (in terms of the stability of the mercury film in air) has been chosen as nickel and an alloy with a high content of this component - 79HM. For other materials, these indicators should be the same.

By repeatedly using the same volume of electrolyte with monitoring the mercury ion content before and after electrolysis, the lower concentration limit of the working zone of the solution has been determined. As can be seen from Table 2, the upper limit of the concentration is established by the special experiments - 0.054 M $HgSO_4$ (10 mg / ml Hg^{2+}). It should be noted that during the period of the solution operation, there is no significant change in the concentration of its other components (H_2SO_4, Na_2SO_4). Based on the obtained data, the working capacity of the solution is estimated as 130-220 col./-cm^3. The lower limit of the Hg^{2+} concentration of the depleted electrolyte: 2-3 mg/g. in Table 2.

Table 2 – The change in the concentration of mercury ions during electrolysis during the amalgamation of the 79HM alloy.

Duration of the electrolysis, h		Content of H_g^{+2}, mg/ml		Amount of the extracted mercury, mg	Characteristics of the mercury coatings
stages	general	starting	ending		
1.Electrolysis conditions: S_{an}=1.08 cm^2, S_c=1.08 cm^2, S_c: S_a =1:1, V_{el}=16ml, l_{a-c}=3cm., U=1.5-1.8 B, S:V_{el}=1:16, t=25-30°C, I=20-30 A/dm^2					
79HM					
5	5	10.08	1.81	131.60	shiny, durable
4	9	5.02	1.51	56.19	-“-
3	12	2.81	1.31	24.03	stable for 30 min.
4	16	1.20	0.30	7.44	-“-
2.Electrolysis conditions: S_c:V_{el}=1:100, S_c=1,1 cm^2, V_{el}=110ml, , l_c=2.3cm., φ=0.15cm, S_c: S_a =1:1, t=22-25°C, $l_{а-кс}$=3cm, U=1.5-1.8 B, I=20-30 A/dm^2					
10	10	9.08	6.02	330.99	stable in air for 2 hours
3	13	6.02	5.02	110.33	-“-
2	15	5.02	4.01	110.33	-“-
4	19	4.01	3.76	27.50	-“-
4	25	3.76	3.51	27.60	stable for 5 min.
1	26	3.51	3.22	32.00	-“-
1	27	3.22	2.0	134.20	-“-

Figure 2 shows the dependence of the concentration of the mercury ions (Hg^{2}□) on the electrolysis time during the amalgamation of the 79HM alloy.

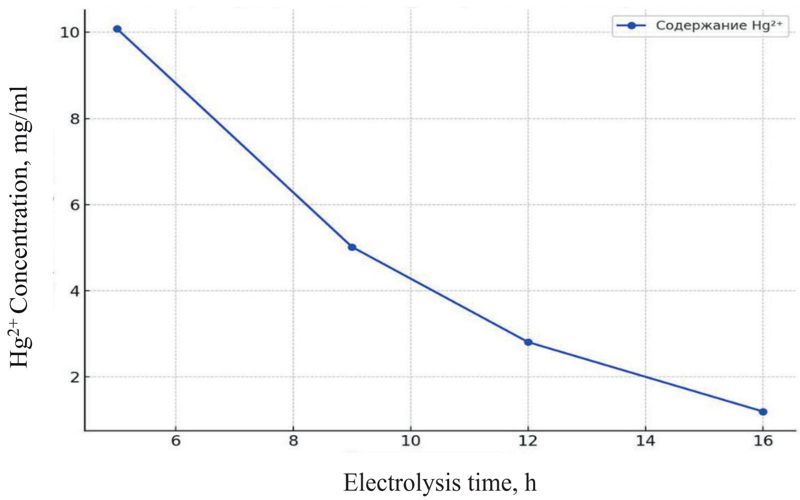


Figure 2 – The change in the concentration of the Hg^{2+} ions during the amalgamation of the 79HM alloy.

Determination of the thickness and stability of the mercury coatings on the studied materials

As indicated above, the quality of the mercury coating on the materials has been assessed based on the values of the thickness of the mercury layer on the samples and its stability during the centrifugation. The objects of study have been metallic iron, 13X steel, 47НД alloys, monel 40A and nickel silver. The results of all determinations are given in Tables 3 and 4.

During the amalgamation, a mercury film with a thickness of $\delta = (2\text{-}3).10^{-3}$ cm is formed on the iron samples, and a certain tendency is visible for the dependence of δ on the polishing time τ of the sample: at the maximum τ, the highest value of δ is recorded (Table 3). After the centrifugation of the samples, the thickness of the mercury film decreases by 3-4 times, on the average $\delta = 1.10^{-3}$ cm. In appearance, the coating is continuous and shiny.

Table 3 - Thickness of the mercury film on the amalgamed iron plates.

Polishing time, τ, sec	Sample dimensions, cm	Sample area, S cm^2	Thickness of mercury coating, $\delta .10^{-3}$ cm	
			After the amalgamation	After the centrifugation
60	15.0x3.0	0.90	2.14	0.66
60	17.5x2.4	0.84	3.38	1.10
90	17.0x2.5	0.85	3.68	1.03
90	15.5x3.0	0.93	4.87	1.09
120	15.0x2.0	0.60	3.68	0.99
120	16.0x3.0	0.96	4.69	0.82
180	17.5x2.0	0.70	4.30	1.31
300	16.0x1.9	0.61	8.06	1.43

For the other materials under study, the thickness of the mercury film immediately after the amalgamation is, on the average, becomes ($\delta .10^{-3}$ cm):

47НД – 1.24 x 10^{-3} cm; nickel silver – 1.71 x 10^{-3} cm;
13X steel – 6.02 x 10^{-3} cm; monel 40A – 0.85 x 10^{-3} cm.

After the centrifugation of the samples, the thickness of the mercury coating decreases by 2-4 times and, on the average, becomes ($\delta .10^{-3}$ cm):

47НД – 0.78 x 10^{-3} cm; nickel silver – 0.48 x 10^{-3} cm;
13X steel – 2.00 x 10^{-3} cm; monel 40A – 0.40 x 10^{-3} cm.

(Tables 3 and 4). The mercury film on the samples of the above materials is continuous, smooth and well preserved in the air. After the centrifugation, when the excess mercury is removed, a thin, shiny layer of mercury remains on the surface, simply bonded to the surface of the material. The high stability of the mercury film, especially on the alloys 47НД, monel 40A and nickel silver, is obviously explained by the fact that these alloys contain metals, which have a significant affinity for mercury, ensuring good wetting and strong adhesion of the liquid metal to the base. The condition of the mercury coatings on the samples of the 13X steel should be especially noted. After the centrifugation, after 30-60 minutes, a gray, pasty coating appears on the amalgamed surface. Moreover, the possibility of its formation increases with increasing the sample polishing time. After the mechanical removal of the coating (filter paper), the surface of the mercury film becomes shiny, smooth and remains in this form in the air for quite a long time: 10-15 days or more.

Table 4 - Thickness of the mercury film on the amalgamed (electrolytically) samples of the 47НД (q = 0.08 cm) and 13X steel (plate = 1.5x2.5).

<table>
<tr><td rowspan="2">Sample area,
S cm^2</td><td colspan="2">Mercury coating thickness
$\delta \cdot 10^{-3}$ cm</td></tr>
<tr><td>After the amalgamation</td><td>After the centrifugation</td></tr>
<tr><td colspan="3">47НД</td></tr>
<tr><td rowspan="5">0.88
0.73
0.96
0.91
0.88</td><td>1.25</td><td>0.68</td></tr>
<tr><td>0.80</td><td>0.64</td></tr>
<tr><td>1.70</td><td>0.72</td></tr>
<tr><td>1.27</td><td>0.89</td></tr>
<tr><td>1.20</td><td>0.98</td></tr>
<tr><td colspan="3">13X steel</td></tr>
<tr><td rowspan="5">0.48
0.44
0.36
0.64
0.56</td><td>6.81</td><td>1.79</td></tr>
<tr><td>6.75</td><td>1.95</td></tr>
<tr><td>6.27</td><td>2.50</td></tr>
<tr><td>5.63</td><td>1.83</td></tr>
<tr><td>4.66</td><td>1.94</td></tr>
</table>

Table 5 – Thickness of the mercury coating applied electrolytically to the nickel silver (plate) and monel 40A samples (q – 0.042 cm)

<table>
<tr><td rowspan="2">Sample area,
S cm^2</td><td colspan="2">Mercury coating thickness</td></tr>
<tr><td>After the amalgamation
$\delta \cdot 10^{-3}$cm</td><td>After the centrifugation
$\delta \cdot 10^{-4}$cm</td></tr>
<tr><td colspan="3">nickel silver</td></tr>
<tr><td rowspan="6">2.48
2.64
2.45
1.58
1.60
2.40</td><td>1.54</td><td>6.69</td></tr>
<tr><td>1.82</td><td>5.83</td></tr>
<tr><td>1.57</td><td>-</td></tr>
<tr><td>2.07</td><td>2.69</td></tr>
<tr><td>1.39</td><td>5.79</td></tr>
<tr><td>1.89</td><td>3.03</td></tr>
<tr><td colspan="3">monel 40A</td></tr>
<tr><td rowspan="7">0.20
0.16
0.18
0.24
0.44
0.50
0.49</td><td>1.15</td><td>3.50</td></tr>
<tr><td>0.94</td><td>3.75</td></tr>
<tr><td>0.57</td><td>3.43</td></tr>
<tr><td>0.86</td><td>3.68</td></tr>
<tr><td>0.95</td><td>-</td></tr>
<tr><td>0.78</td><td>4.85</td></tr>
<tr><td>0.74</td><td>5.13</td></tr>
</table>

The effect of the preliminary preparation stages of various materials on the quality of the mercury coating.

First, the characteristics of the mercury coatings have been studied on the samples of the 29НК, 52Н alloys, X18H10T steel, cobalt alloys, when their surface has been only etched before electrolytic deposition of mercury.

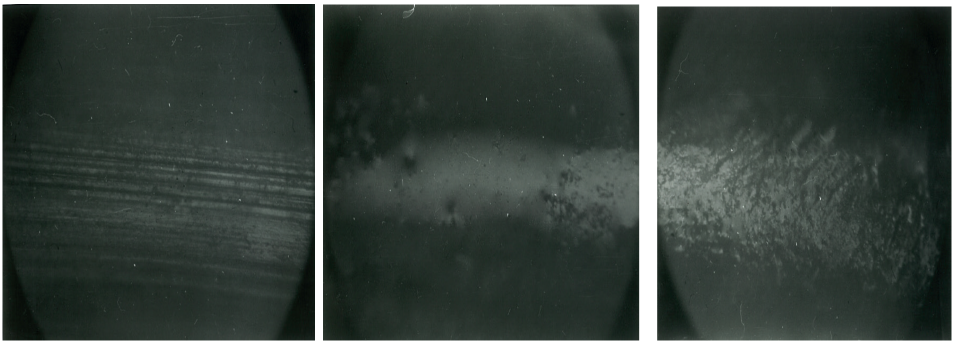
The results of the determinations are given in Table 6. As can be seen, after the amalgamation, the contact surface is covered with an even layer of mercury with the thickness of (1-3).10^{-3} cm. As a result of the centrifugation (1600 rpm), the excess mercury is removed, leaving a thin film with the thickness of (1-2).10^{-4} cm. The coating of the 29НК alloys, Х18Н10Т steel has turned out to be stable, continuous (Table 5). And the stability of the mercury coating on Co alloys is lower than on the 29НК alloy.

Table 6 – The quality of the mercury film on the amalgamed samples of the contact materials (at 20°C) subjected to etching during the preparation.

Name or composition of the alloy	Electrolysis condition		Weight of the samples after the treatment, g				Thickness of the mercury coating δ, cm			l, mm	Coating condition after the centrifugation at 1500 rpm
	τ, mA	Time, hour	Etching	Amalgamation	Centrifugation		Initial	After the centrifugation			
					1500 rpm	2500 rpm		1500 rpm $\delta \cdot 10^{-4}$	1500 rpm $\delta \cdot 10^{-4}$		
29НК	30	2	0.3569	0.3737	0.3586	0.3583	$0.51 \cdot 10^{-3}$	1.53	1.26	26	The coating is smooth
-“-	120	1	0.3599	0.3818	0.3612	0.3608	$1.46 \cdot 10^{-3}$	0.87	0.60	28	Partially has come off at 2500 rpm
Х18Н10Т steel	200	1	0.8648	0.9105	0.8697	0.8678	$5.63 \cdot 10^{-3}$	6.03	3.07	19	Continuous film
Co alloy No. 1 (Ni – 20%; Co -60%)	60	2	0.0744	0.0814	0.0758	0.0745	$1.40 \cdot 10^{-3}$	2.81	0.20	17	Continuous film
-“-	-“-	1	0.0659	0.0737	0.0664	0.0662	$2.66 \cdot 10^{-3}$	1.70	1.02	10	The coating has come off
Co Alloy No. 3 (Ni – 10%; Co -75%)	120	1	0.1015	0.0115	0.1026	0.1021	$1.48 \cdot 10^{-3}$	1.63	0.89	23	Continuous film
-“-	-“-	-“-	0.0702	0.0779	0.0711	0.0708	$1.46 \cdot 10^{-3}$	1.71	1.14	18	Partially has come off at 2500 rpm
Co Alloy No. 7 (Ni – 2%; Co -86%)	-“-	-“-	0.0754	0.0792	0.0769	0.0759	$8.07 \cdot 10^{-4}$	1.92	1.07	16	The coating has come off
Co Alloy No. 8 (Ni – 2%; Co -88%)	200	1	0.0772	0.0855	0.0775	0.0773	$2.18 \cdot 10^{-3}$	0.79	-	16	The coating has come off
Co Alloy No. 10 (Ni – 2%; Co -92%)	60	2	0.0726	0.0728	0.0742	0.0740	$1.53 \cdot 10^{-2}$	3.42	2.99	16	The coating is good

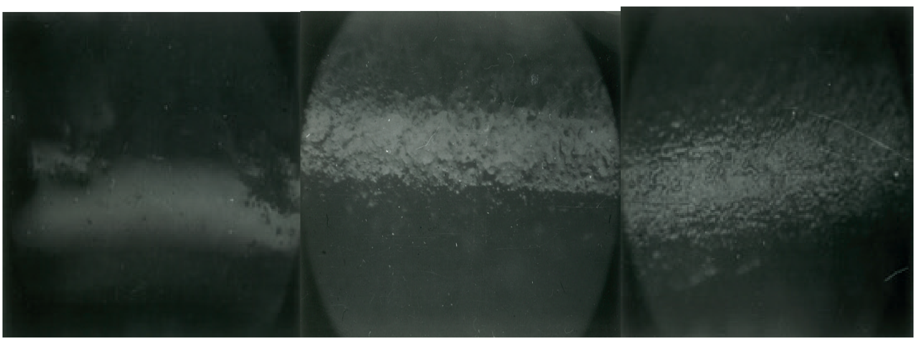
-“-	-“-	1-“-	0.0776	0.0814	0.0783	0.0780	$8.60 \cdot 10^{-4}$	0.16	-	15	Partially has come off at 2500 rpm

A certain tendency towards a decrease in film stability with increasing the Co content in the alloy has been noted. More stable mercury coatings on the alloys are obtained by treating the alloy surface not only by etching, but also by subsequent chemical polishing. Table 6 shows the results obtained during the amalgamation of 47НД, Н52ВИ, Co alloys, nickel silver and beryllium bronze, alloys subjected to the centrifugal acceleration from 580 up to 3560 m/sec^2. All the alloys studied are characterized by the formation of a stable, even mercury coating on their surface (Fig. 3, 4). Thus, the application of the amalgamation method, developed by us for metals and alloys, which are low soluble in mercury, makes it possible to obtain a high-quality, stable mercury coating on the materials, which are promising for the manufacture of the contact parts for the magnetically controlled devices and mercury-film microelectrodes.



а) б) в)

Figure 3 - Surface of the Co-alloy No. 3 after the treatments:
a) etching; b) amalgamation; c) centrifugation n = 600 rpm.



а) б в

Figure 4 – The surface of the Co-alloy No. 5
a) after the amalgamation b) after the centrifugation = 600 rpm.
c) after the centrifugation n = 1500 rpm.

If we compare this with the data obtained by the authors (Kounaves S.P et al, 1987) on the iridium samples, then in the morphology of the mercury film, the flat non-uniform mercury spots are observed. Mercury deposition occurs very slowly and with the same result. A long-term deposition up to 5 hours causes the destruction of the surface and the formation of a continuous black film on it. HClO has been the only electrolyte with which a completely flat or hemispherical film has been formed (Figure 5)

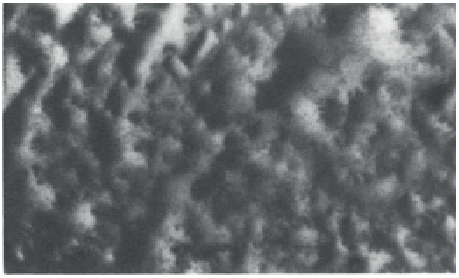


Figure 5 – Morphology of the mercury film in the solutions, containing ethylenediamine, EDTA.

In all cases, mercury deposition has occurred very slowly and with the same result. However, even in this case, as the authors note, there has been only a 50% success rate.

The remaining attempts have resulted in a coating similar to that shown in Figure 6 (22).

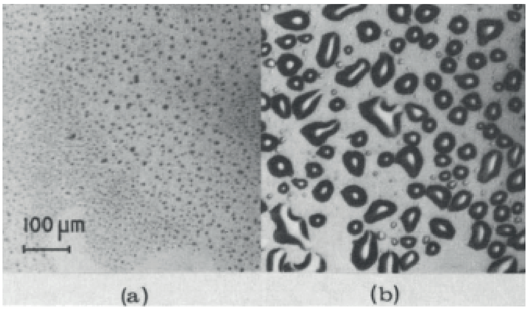


Figure 6 – Morphology of the mercury film in the solutions for both KHO (Sl) and HNO (w = 1000 rpm) in the potential range from -200 mV to -1500 mV after: (a) 10 min, (b) 60-90 min.

Thus, in the electrolyte we have used, an overvoltage is created to reduce the hydrogen ions on the contact material due to the previous release of mercury on the surface of the alloys. The mercury coating obtained in this solution on the 29НК alloy (τ = 40 mA, φ = -1.1 – 1.26) is quite smooth and fairly stable.

Under these conditions, the possibility of the amalgamation of the 48NH, 52N alloys and platinum has been also tested. It has been shown that these materials amalgam well with the formation of a stable mercury film. In addition, the effect of current density on the process of mercury ion reduction has been studied. The study has shown that the optimal conditions for the amalgamation of the 48HX, 52H alloys and platinum are a current density of 40-100 mA/cm^2, the electrolysis time of 15-20 min. In this case, the surface of the contact materials has been covered with an even layer of mercury, which had good adhesion to the base metal.

Conclusions. It has been established that the complex use of the developed methods for the formation of stable mercury-film coatings on the surface of difficult-to-amalgam metals and alloys allows us to recommend them as contact parts for reed switches, contactors, etc.

For the first time, the characteristics of mercury coatings on new cobalt-containing materials have been studied in detail, the possibility of using them as a substrate for mercury coating in the development of new technical solutions for switching equipment has been demonstrated, and it corresponds to the modern UN Sustainable Development Goals (SDGs) (Responsible Consumption and Production).

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