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«ҚАЗАҚСТАН РЕСПУБЛИКАСЫ
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«ҚАЗАҚСТАН РЕСПУБЛИКАСЫ
ҰЛТТЫҚ ҒЫЛЫМ АКАДЕМИЯСЫ» РҚБ

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ИЗВЕСТИЯ

РОО «НАЦИОНАЛЬНОЙ
АКАДЕМИИ НАУК
РЕСПУБЛИКИ КАЗАХСТАН»

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NAS RK is pleased to announce that News of NAS RK. Series of geology and technical sciences scientific journal has been accepted for indexing in the Emerging Sources Citation Index, a new edition of Web of Science. Content in this index is under consideration by Clarivate Analytics to be accepted in the Science Citation Index Expanded, the Social Sciences Citation Index, and the Arts & Humanities Citation Index. The quality and depth of content Web of Science offers to researchers, authors, publishers, and institutions sets it apart from other research databases. The inclusion of News of NAS RK. Series of geology and technical sciences in the Emerging Sources Citation Index demonstrates our dedication to providing the most relevant and influential content of geology and engineering sciences to our community.

Қазақстан Республикасы Ұлттық ғылым академиясы «ҚР ҰҒА Хабарлары. Геология және техникалық ғылымдар сериясы» ғылыми журналының Web of Science-тің жаңаланған нұсқасы Emerging Sources Citation Index-те индекстелуге қабылданғанын хабарлайды. Бұл индекстелу барысында Clarivate Analytics компаниясы журналды одан әрі the Science Citation Index Expanded, the Social Sciences Citation Index және the Arts & Humanities Citation Index-ке қабылдау мәселесін қарастыруда. Webof Science зерттеушілер, авторлар, баспашылар мен мекемелерге контент тереңдігі мен сапасын ұсынады. ҚР ҰҒА Хабарлары. Геология және техникалық ғылымдар сериясы Emerging Sources Citation Index-ке енуі біздің қоғамдастық үшін ең өзекті және беделді геология және техникалық ғылымдар бойынша контентке адалдығымызды білдіреді.

НАН РК сообщает, что научный журнал «Известия НАН РК. Серия геологии и технических наук» был принят для индексирования в Emerging Sources Citation Index, обновленной версии Web of Science. Содержание в этом индексировании находится в стадии рассмотрения компанией Clarivate Analytics для дальнейшего принятия журнала в the Science Citation Index Expanded, the Social Sciences Citation Index и the Arts & Humanities Citation Index. Web of Science предлагает качество и глубину контента для исследователей, авторов, издателей и учреждений. Включение Известия НАН РК. Серия геологии и технических наук в Emerging Sources Citation Index демонстрирует нашу приверженность к наиболее актуальному и влиятельному контенту по геологии и техническим наукам для нашего сообщества.

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CONDITIONS FOR PREPARING THE SURFACE OF CONTACT PARTS FOR WETTIBILITY WITH MERCURY

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Abstract. The paper provides a scientific analysis of the behavior of the iron group metals and alloys on their basis in the processes of chemical preparation of surfaces prior to applying stable mercury coatings to create a new generation of switching devices, represented mainly by mercury wetted reed switches. The existing difficulties are determined by the problematic operation of reed switches with solid-state contacts, associated with the signal bounce, unstable contact resistance and wear of contact surfaces. To solve these problems, the use of liquid metal microswitches has been proposed, where a liquid-liquid contact is used, which is necessary to ensure a trouble-free operation of the devices. For these purposes, the behavior of the iron group metals and alloys on their basis in the processes of chemical preparation of their surfaces prior to applying mercury coatings has been studied. For the both stages of chemical processing - etching and polishing - the thickness of the removed layer of the material has been determined, and in case of alloys, the ratio in which the metals go into the solution has also been determined. The behavior of the iron group metals and alloys on their basis in the processes of etching and polishing indicates that during chemical treatment the surface is completely cleaned of oxides, and the correspondence of the ratio of the layer surface to their chemical composition is achieved.

Keywords: mercury, amalgamation, chemical treatment, etching, polishing, reed switch, contacts

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Аннотация. Мақалада темір тобындағы металдар мен олардың негізінде жасалған қорытпалардың тұрақты сынапты жабындарды жағуға дейінгі беттерді химиялық дайындау процестерінде негізінен сынапты геркон ажыратқыштарымен ұсынылған коммутациялық құрылғылардың жаңа буынын құруға ғылыми талдау берілген. Бар қиындықтар сигналдың серпілісімен, тұрақсыз жанасу кедергісі және жанасу беттерінің тозуымен байланысты қатты күйдегі контактілері бар геркон ажыратқыштардың проблемалық жұмысымен анықталады. Бұл мәселелерді шешу үшін жанасуда қолданылатын сұйық-сұйық микробағдарламаларын қолдану ұсынылды, бұл құрылғылардың ақаусыз жұмысын қамтамасыз ету үшін қажет. Осы мақсаттарда темір тобының металдары мен олардың негізіндегі қорытпалардың сынапты жабындарды жағу алдында беттерін химиялық дайындау процестеріндегі жағдайы зерттелді. Химиялық өңдеудің екі сатысы үшін – өңдеу және жылтырату – материалдың жойылған қабатының қалыңдығы, ал қорытпалар жағдайында металдардың ерітіндіге түсу арақатынасы да анықталады. Темір тобына жататын металдардың және олардың негізіндегі қорытпалардың өңдеу және жылтырату процестеріндегі жағдайы химиялық ою кезінде беттің оксидтерден толық тазартылып, қабат бетінің олардың химиялық құрамына қатынасына қол жеткізілетінін көрсетеді.

Түйін сөздер: сынап, амальгамирование, химиялық өңдеу, ою, жылтырату, геркон, байланыстар

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УСЛОВИЯ ПОДГОТОВКИ ПОВЕРХНОСТИ КОНТАКТ-ДЕТАЛЕЙ К СМАЧИВАЕМОСТЬЮ РТУТЬЮ

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Аннотация. В статье проведен научный анализ поведения металлов группы железа и сплавов на их основе в процессах химической подготовки поверхностей перед нанесением устойчивых ртутных покрытий для создания нового поколения коммутирующих приборов, представленных в основном ртутными герконами. Существующие трудности определяются проблематичностью работы герконов с твердотельными контактами, связанные дребезгом сигнала, нестабильным контактным сопротивлением и износом контактных поверхностей. Для решения этих проблем предложено использование жидкометаллических микропереключателей, где используется контакт жидкость-жидкость, необходимый для обеспечения безотказного срабатывания приборов. В этих целях изучено поведение металлов группы железа и сплавов на их основе в процессах химической подготовки их поверхностей перед нанесением ртутных покрытий. Для обеих стадий химической обработки – травления и полирования – определены толщина снимаемого слоя материала, а в случае сплавов – также соотношение, в котором металлы переходят в раствор. Поведение металлов группы железа и сплавов на их основе в процессах травления и полирования свидетельствуют о том, что при химической обработке происходит полная очистка поверхности от оксидов и достигается соответствие соотношения поверхности слоя с их химическим составом.

Ключевые слова: ртуть, амальгамирование, химическая обработка, травление, полирование, геркон, контакты

Introduction

Currently, in the world and domestic technology, switching microdevices with liquid metal contacts are mainly represented by mercury wetted reed switches. The existing mechanical devices, using solid-state contacts, have the problems associated with unreliability of their operation, including unstable contact resistance, signal bounce, general loss

of performance during use, and limited service life, caused by mechanical fatigue and wear of the contact surfaces. To solve these problems, the use of liquid metal microswitches, using a liquid-liquid contact, has been proposed (Kondoh, 2005).

Upon manufacturing of such reed switches, considerable attention is paid to the development of effective amalgamation methods to obtain stable mercury coatings on the working surface of the contacts, where a contact (contacting and separation) is carried out due to the mercury film in contact with the liquid metal conductive medium (mercury) (Mussina et al., 2019). Taking into account the rapid pace of discovery and growth of new proposals in this field, the up-to-date presentations of the latest advances in methodologies, sensors, detectors, and microchips are necessary (Wang, Joseph, 2006).

Along with this, when choosing contact materials, it is necessary to have information on the corrosion resistance in mercury of metals and alloys, used in the manufacture of contact parts and to identify the nature of the processes, occurring on their surface upon contact with the liquid metal medium.

Particular attention is paid to the selection of new materials as substrates for mercury coating and their formation processes, as well as new developments, associated with the creation of new modifications of devices (Economou, Anastasios, Fielden 2003).

Only the works, devoted to the formation of a mercury film during the manufacture of electrodes on metallic silver and silver-coated glassy carbon substrates, related to the materials, wetted by mercury, are known (Lexa, Jiří, 1985).

One of the few works also includes a research on the creation of micromechanical relays, switched by moving a mercury microdrop, which has proposed the use of mercury to eliminate the common problems of solid contact switches. Although microrelays have been repeatedly used in MEMS, they are all based on solid-state contacts with their characteristic negative performance (Simon, Jonathan, 1997).

As for difficult-to-amalgam metals and alloys, there is virtually no information on reliable methods of amalgamation, i.e. creating stable mercury coatings on their surface, which would open the way for recommendations for use in the contact technology, where mercury and amalgams are used as a conductive medium.

It should be especially noted that a serious problem in the development of new modifications of mercury-containing switching devices, design and manufacture is the creation of conditions for wetting and the formation of a stable mercury film on the surface of the contact parts made of metals, which are sparingly soluble in mercury. The known amalgamation methods are not effective enough. Besides, there is practically no reliable information on their behavior in mercury. Such data are extremely necessary to clarify the nature of the processes, occurring on the surface of metals and alloys upon the contact with mercury. They will make it possible to make the correct choice of the contact parts when designing devices and selecting optimal operating conditions. The reliability of contact devices is largely related to the process of amalgamation of the contacting material, and the quality of the resulting mercury film.

As is known, wetting of a solid with mercury occurs under the conditions, which provide the possibility of creating a liquid film on the solid surface. A significant role is played by such factors as the magnitude of the surface energy of the solid metal and its affinity for mercury (Goryunov, 1972). The films widely used in technology are usually applied to a metallic or nonmetallic base. The resulting connection between the film and the base, i.e. adhesion, affects the quality and properties of the product, in our case, devices and parts

for them, which come into contact with mercury. To ensure good wetting of a solid surface with a liquid, it often becomes necessary to thoroughly clean it first. The work discusses the basic techniques, used in practice for these purposes. In this case, the main attention is paid to obtaining mercury coatings with the formation of stable films and methods of determination (Mussina et al., 2019).

Materials and basic methods

As is known, a proper preparation of the surface of metals and alloys prior to applying galvanic coatings, in our case mercury, ensures the strongest adhesion to the base metal. One of the mandatory operations after mechanical grinding, degreasing in various ways (rinsing in solvents, alkaline solutions, emulsion cleaning, electrolytic treatment) is a special surface treatment, which further ensures reliable adhesion of the mercury coating to the base - metal or alloy (Griliches, 1961; Griliches, 1977; Innes, 1987).

This kind of surface preparation of materials is carried out mainly by the chemical and electrochemical methods.

The study has been devoted mainly to the issues of the chemical preparation of the surface of metals and alloys for the application of mercury coatings, taking into account the assessment of their strength, and determination of the corrosion resistance of materials in mercury. Accordingly, the selection and development of the appropriate experimental techniques have been established.

The objects of the study have been metals of the iron group - Ni, Fe and alloys on their basis - 29NK, 52N, 47ND, Monel NM-40A, nickel silver, steel 13X, used as the contact parts in the manufacture of liquid metal switching devices, the composition of which is presented in Table 1.

Table 1 - Chemical composition of the studied alloys.

Material	Content of the components, wt.%							
	Fe	Ni	Co	Cu	Cu	Mn	Zn	Mo
29HK, kovar	53	29	18	18	-	-	-	-
52N, permalloy	48	52	-	-	-	-	-	-
47ND	48	47	-	-	5	-	-	-
Steel I3X	87	-	-	-	-	-	-	-
Monel NM – 40A	2	68	-	-	28	2	-	0.05
Nickel silver	0.5	15	-	-	65	0.3	20	-

Preparation of the samples, sealed in ampoules

The samples of the materials under study in the form of wire (0.4 - 1 mm, l = 2.0 - 4.0 cm) or plates (S = 5-6 cm^2, l = 3-4 cm) were sealed in glass ampoules (15 mm); then, after degreasing, they were etched and polished.

The methodology for assessing the quality of the surface treatment of the material samples at the stages of chemical preparation was carried out on the basis of the data of an analysis of the etching and polishing solutions after the treatment and visual observation of the surface condition.

The solution after each treatment was analyzed for the content of alloy components, using the atomic absorption method. By the amount of the found metals, their ratio when entering the solution and the thickness of the removed layer were calculated. The observation of the state of the surface of the samples was carried out, using a MIM-7 (x140) microscope,

while simultaneously fixing it on the photographic film. To detrermine the effect of the sealing operation on the state of the surface of the materials, the determinations were carried out on the samples both sealed into ampoules and unsealed.

Experimental part

When processing metals and alloys at the stages of chemical preparation of the surfaces, prior to applying metal, in particular, mercury coatings, a change of the composition and micro-relief of the surface layer of the samples occurs. The chemical methods consist of treating the surface with the aggressive solutions of various compositions, under the influence of which the processes of etching and polishing proceed.

When etching, the surfaces of metals and alloys are freed from oxides, grease residues and other foreign substances. In this case, the crystalline structure of the material is exposed with some micro-leveling of the surface, reaching roughness class 9.10. At the polishing stage, a finer leveling of the micro-relief occurs and smoothing of the surface roughness. At the same time, the processing class increases accordingly to 12–13.

There is no such information in the literature for the contact materials, which we have studied. In this regard, it is very relevant to study their behavior in the chemical preparation processes in order to clarify the nature and depth of the corrosive effect of the etching and polishing solutions on the surfaces of the materials being processed.

This approach was used previously, when using steel as machine components or structures, which often broke prematurely due to corrosion. To overcome these problems, the HDG method was applied, beginning with a pre-treatment process, which included polishing, degreasing, rinsing I, etching, rinsing II, fluxing and drying (Gapsari, Femiana, 2019). But for the formation of stable mercury coatings, this approach was unacceptable. Therefore, there was a need to select special methods for preparing the surface of difficult-to-amalgamate materials to ensure ideal wetting conditions.

We selected the optimal compositions of the solutions for etching and polishing the iron group metals and alloys on their basis prior to electrolytically applying a mercury coating. The photographs are presented in Figures 1-6.

The main indicator of the quality of chemical processing of the materials should be the final result - the stability of the mercury film, as a result of which the effectiveness of the performed operations should be assessed mainly according to this criterion. In our case, the three compositions of the etching solutions, consisting of a mixture of acids, were used to carry out etching:

1) HNO_3:H_2SO_4 = 1:1 at 303K;
2) H_2SO_4:HCl:H_2O = 2:1:2 at 293K;
3) HCl (100 g/l) at 293K.

Unfortunately, the use of these solutions for etching did not give positive results. In this regard, the possibility of using a more concentrated HCl = 1:1 for etching was investigated. The obtained results showed that such processing of the material resulted in a higher-quality mercury coating, however, the data on the stability of mercury films after centrifugation were not always reproduced. This can be explained by the fact that when the used solutions were at the room temperature, the oxide films obviously were not completely removed from the treated surfaces, and therefore their complete wetting with mercury was not ensured. Taking into account the above, the temperature regime for carrying out the etching process in a solution of the composition recommended in (Technical Guides of the Ministry 1968): H_2SO_4:HCl:H_2O = 2:1:2 was used, but at a higher temperature of 343K.

Processing time was two minutes. As a result of this treatment, the surface of the alloy materials became rough, which could be easily eliminated by subsequent polishing.

In our case, to level the micro-relief of the surface of the etched samples, we used the process of chemical polishing according to the known methods of processing the iron group metals and alloys on their basis (Technical Guides of the Ministry 1968, Reference Guide to Electroplating 1972, Nagai Yasusu, Wachi Hiroshi 1984, Kubo Hidefumi, Nakami Hajime 1985, Scientific technical report 1987).

The optimal composition of the polishing solution was established:

$HCl:HAc:HNO_3$, = 1:70:30.

The treatment in this solution was carried out at the temperature of 343–353 K for 4-6 seconds. As a result of the use of such processing, it was possible to obtain a high-quality stable mercury film, which evenly covers the surface of the samples made of various materials (Table 1).

Thus, the method of chemical preparation of the surface of the material, in addition to degreasing, includes two successive stages - etching with a solution of H_2SO_4:HCl:H_2O = 2:1:2 and polishing in a solution: $HCl:HAc:HNO_3$ = 1:70:30. As the observations under a microscope showed, the state of the surface of the materials at the etching stage was deeply worked with the formation of roughness in the form of wide grooves. Subsequent polishing ensured the alignment of the micro-relief and composition of the surface layer of the processed material. The mercury coating applied to the materials prepared in this way was stable, well adhered to the amalgamated surface. The used techniques should be considered as the most appropriate when preparing the surface of contact materials from the iron group metals and alloys on the basis for amalgamation.

Results and discussions

Available information on the effects of hard substrate surface preparation: industrial alkaline degreasing, acid etching and mechanical preparation (polishing) has been limited to mild steel, the most widely used steel in the world for industrial and domestic applications, but not relevant to switchgear where, as it has been noted earlier, it is necessary to maintain ideal surface cleanliness (Atmani, Poelman, 2010).

In this work, the quality of the surface of material samples at the stages of chemical preparation has been judged on the basis of the data of an analysis of the etching and polishing solutions. Tables 2 and 3 for all studied materials have shown the results of determining the thickness of the layer being removed and the ratio of the metals, which have gone into the solution at the both stages of chemical treatment. The values have been calculated on the basis of the data of an analysis of the etching and polishing solutions after the processing operations on the material samples.

When etching nickel and iron samples, sealed into glass ampoules, a layer of metal with a thickness of δ is removed: Ni = $1 \cdot 10^{-4}$ cm, Fe = $(2\text{-}3) \cdot 10^{-5}$ cm (Table 2, 3).

Table 2 - Thickness of the removed layer after the chemical treatment of the Ni samples, sealed into ampoules (q=0.56 mm).

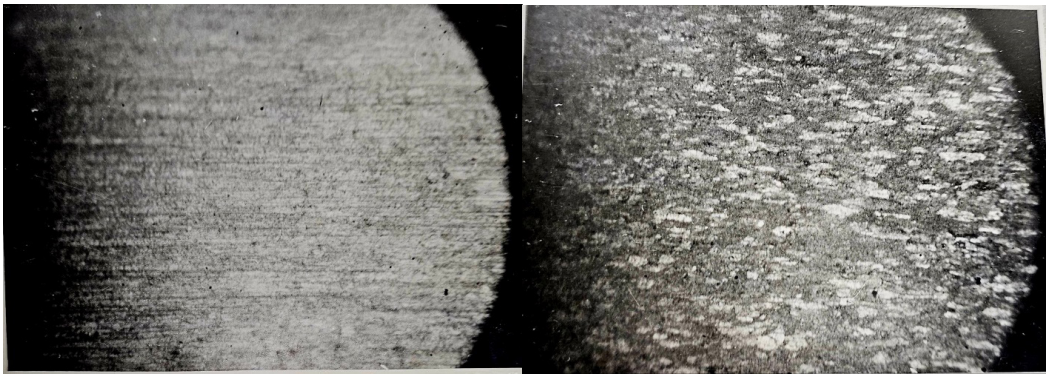
Experiment No.	Sample area S, cm^2	Thickness of the removed layer	
		Upon etching $\delta \cdot 10^{-4}$ cm	Upon polishing $\delta \cdot 10^{-4}$ cm
1	0.512	1.03	2.73
2	0.530	0.64	1.87
3	0.530	0.91	3.19

Table 3 - Thickness of the removed layer after the chemical treatment of the Fe and 13X steel samples in the form of plates

Experiment No.	Name of the material	Sample area S, cm^2	Thickness of the removed layer at different stages, cm	
			etching $\delta \cdot 10^{-5}$ cm	polishing $\delta \cdot 10^{-4}$ cm
4	**Iron** Samples, sealed in ampoules	1.20	11.70	2.87
5		0.96	18.00	3.82
6		0.84	18.00	3.82
7		1.05	28.80	3.17
8		0.78	0.29	4.33
9		1.12	0.20	5.75
10		1.12	0.20	7.31
11		1.12	-	6.30
12		1.20	0.19	4.63
13[x]		1.36	2.89	4.67
14[x]		1.52	4.17	3.13
15[x]		1.25	-	3.81
16	Steel 13X	1.75	1.71	6.91
17		1.75	1.81	6.86

Note: x) at etching τ =90 min;

With an increase in the duration of etching of the Fe samples up to 90 min (according to the method τ = 30 min), the thickness of the crushed layer by an order of magnitude increases, and in this case its value approaches δ for the unsealed samples - $(2–3)\cdot 10^{-4}$ cm. At the polishing stage on the both metals, a layer of approximately the same thickness $(3–5\cdot 10^{-4}$ cm) is removed. The micro-relief of the surface of the samples reflects the degree of processing at the stages of etching and polishing (Fig. 1 a, b; Fig. 2).



а) б)

Figure 1 – The Fe area after the treatments:

а) etching, б) polishing

Figure 2 – The surface of nickel after polishing

The effect of the etching solution on steel 13X leads to the transition of only iron into the solution, while the thickness of the removed layer is of the same order as in the case of pure iron = $2 \cdot 10^{-5}$ cm (Table 3).

The authors (Bezuidenhout, Martin, 2020) have assessed the effectiveness of polishing by removing the mass and reducing the surface roughness. The samples polished in various solutions have had fewer surface crack initiation sites than in the initial state, but their stability over time has not been confirmed.

The solutions have been analyzed after chemical treatment for the content of alloy components, using the atomic absorption method. Such studies have been carried out on the example of the alloys on the basis of iron and nickel - permalloy (52N) and kovar (29NK), most widely used as the contact parts in the manufacture of reed switches, beds of nails and other switching devices (Table 1). In the chemical treatment of the metal surfaces and

alloys, along with a change in micro-relief, there can also be a change in the composition of the surface layer of the material, which is determined by the nature of the chemical processes, occurring during this process. For this purpose, the solutions have been analyzed for the alloy components after processing the materials according to the above methods. Based on the amount of metals, found in the solutions after etching and polishing, the ratio of components during their dissolution and the thickness of the removed layer have been calculated. To make some comparisons, some experiments have been carried out on the images, both sealed and not sealed in glass ampoules (V = 7–8 cm^3).

As can be seen from Table 3, when etching the 52H alloy, the ratio of the components, which have gone into solution, does not correspond to the initial composition of the alloy; a higher nickel content has been found (62.54 ± 1.6) wt.%, compared to iron (38.3 ± 2. 1) wt.%, the thickness of the removed layer corresponds to the value $\delta = (2.5\pm0.8)\cdot10^{-4}$ cm^3. This can be explained as follows. As is known, there is a layer of insoluble oxides on the surface of the alloy, and it has been noticed that upon the effect of the etching solution upon the sample, a mechanical removal of this layer occurs, apparently consisting mainly of iron oxides. In this regard, the following surface layer is correspondingly enriched in nickel. Therefore, during etching, an increased amount of this component passes into the solution as compared with its initial content in the alloy.

Table 3 - The results of the analysis of the etching solutions after the chemical surface treatment of the 25N and 29NK alloys

Material	Sample	Etching			
		The ratio of Me in the solution, wt.%			Removed layer, $\delta.10^{-4}$, cm
		Fe	Ni	Co	
52H	Not sealed in the ampoule	38.37±2.1	62.54±1.6		2.5±0.8
	Sealed in the ampoule	49.45±4.6	50.84±4.8		3.30±0.3
29HK	Not sealed in the ampoule	49.74±1.0	35.74±1.2	14.53±0.4	0.25±0.1
	Sealed in the ampoule	55.84±1.2	30.94±1.1	13.21±1.3	1.15±0.3

When processing the samples sealed in ampoules, the ratio of the alloy components in the solution approaches the original composition: Fe (49.45±4.6)%; Ni (50.84±4.8)%. It can be assumed that this is a consequence of the thermal effect to which the contact is exposed when sealed into the ampoule. In this case, a layer of oxides of the both metals is formed, which, when separated during the etching process, bares the surface of the alloy of the original composition. The thickness of the removed layer is slightly less than $\delta = (1.5\pm0.4)\cdot10^{-4}$ cm (calculated without taking into account the amount of the insoluble scale) than in case of the etching samples in the non-sealed ampoules. Thus, it is the conditions for sealing the samples that should further determine the selection of the optimal composition of the etching mode: the less the possibility of oxidation of the samples during sealing, the more effective the conditions developed by us and recommended in the literature may be.

In case of chemical preparation of the samples from the kovar alloy during etching (Table 3), a transition into the solution of metals in a ratio slightly different from the

initial composition of the alloy is also observed: Fe (49.74 ± 10.36)%; Co(14.5210.39); Ni (35.74±1.24)%, i.e. an underestimated amount for cobalt and an overestimated amount for nickel. However, the thickness of the layer removed from the surface $\delta = (2.5\pm0.7)\cdot10^{-4}$ cm is an order of magnitude lower than when etching the alloy with permalloy.

To clarify the nature of the effect of the etching solution on the surface of the material, the behavior of the 29NK alloy in it, sealed in an ampoule, has been studied, for which the chemical treatment has been carried out in two stages. At the first stage, the sample has been kept in the solution for 20–30 seconds until the layer of oxides (scale) has been separated, and at the second stage, it has been placed in a fresh solution and etching has been continued, bringing the total duration of treatment to 2 minutes.

An analysis of the etching solution has shown the following metal content in the removed layer:

Stage 1 - Fe (64.02±0.95)%, Co (17.76±0.96), Ni (18.19±0.98)%, $\delta = (3.39\pm0.98)\cdot10^{-4}$ cm;

Stage 2 - Fe (43.49±0.95)%, Co (17.69±0.98), Ni (38.90±0.99)%, $\delta = (2.18\pm0.98)\cdot10^{-4}$ cm - without taking into account the metal located in the insoluble residue of scale (Fe_3O_4).

As can be seen, at the first stage of etching, an increased content of iron and a decreased content of nickel are found in the solution, as compared with their ratio in the original alloy. Since at this stage there has been mainly a dissolution of metals from the film oxides, this indicates that it largely consists of iron oxides. Accordingly, the subsequent layer under the scale should be enriched in nickel and somewhat depleted in iron. An analysis of the etching solution after the second stage of processing has confirmed our assumption: indeed, there is an overestimated (up to 39 %) nickel content, and a reduced iron content (43.5 %) relative to the initial composition. The thickness of the removed layer at the two stages of etching $\delta = 0.7\cdot10^{-4}$ cm is of the same order as for one-stage etching $\delta = 1.1\cdot10^{-4}$ cm. During the subsequent polishing of the kovar (Table 3), the following results have been obtained for the metal content in the removed layer, %: Fe (60.00±0.93), Co (12.94±0.77), Ni (27.05± 0.94), $\delta = (9.26\pm0.95)\cdot10^{-4}$ cm. These results are in good agreement with the data on polishing the 29NK alloy, carried out after one-stage etching. Some differences in the behavior of the 29NK and 52N alloys in the etching solution are probably explained by the structure of the alloys. According to the state diagram of the Fe-Ni system, the 52H alloy is a solid solution. In the alloy 29NK, which is a ternary Fe-Ni-Co alloy, slightly higher bond strength of the components is expected due to the influence of the superstructure of the Fe–Co binary system (Kornilov, 1951).

And when processing the samples of the both types of materials in a polishing solution (Table 4), the alloy components dissolve in a ratio close to the original: Ni (51.4±3.0)%; Fe (48.53±3.0)%, the thickness of the removed layer is of the same order as during etching $\delta = (3.39\pm0.9):10^{-4}$ cm. This is quite consistent with our ideas: when polishing, the cleaned surface of the alloys, which does not contain oxides, comes into contact with acids.

Table 4 - The results of an analysis of the polishing solutions after the chemical surface treatment of the 25N and 29NK alloys

Material	Sample	Etching			
		The ratio of Me in the solution, wt.%			Removed layer, $\delta.10^{-4}$, cm
		Fe	Ni	Co	
52H	Not sealed in the ampoule	51.00±1.2	49.06±1.4		3.39±0.9
	Sealed in the ampoule	48.50±3.0	51.47±3.0		3.30±0.3
29HK	Not sealed in the ampoule	60.57±14.2	26.68±2.1	11.45±2.1	7.80±0.9
	Sealed in the ampoule	57.92±1.9	29.57±1.5	12.51±1.0	7.88±1.8

In the contact technology, along with the indicated materials 52N and 29NK, other metals and alloys are widely used. In particular, these include Fe, Ni, alloys 47ND, Monel 40A, nickel silver, steel 13X, etc. The listed materials have been subjected to chemical processing, like kovar and permalloy in the etching and polishing solutions.

Polishing ensures smoothing of all surface roughness by removing a larger layer of material $\delta = 7 .10^{-4}$ cm (Fig. 3).

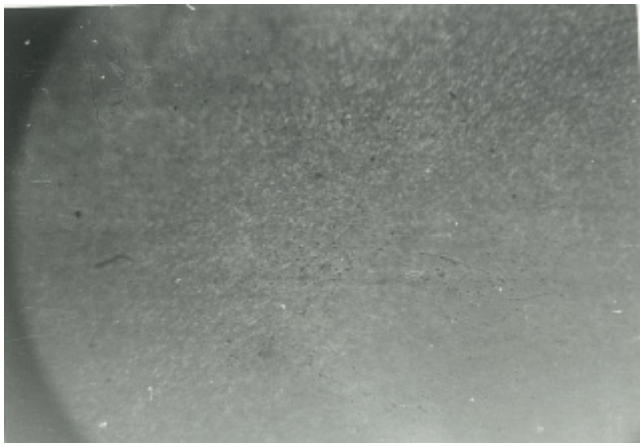


Figure 3 – Surface of 13X steel after polishing

When etching the samples of the 47ND alloy, only nickel and iron pass into the solution (Table 5).

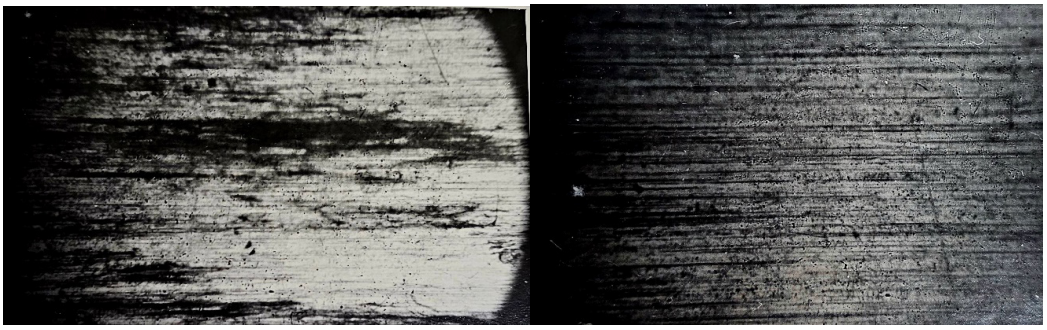
Table 5 - The results of an analysis of the solutions after the chemical preparation of the 47ND alloy for amalgamation (q = 0.8 mm)

Experiment No.	Sample area S, cm²	Etching				Polishing			
		The ratio of the metals in the solution. wt.%			Thickness of the removed layer	The ratio of the metals in the solution. wt.%			Thickness of the removed layer
		Fe	Ni	Cu	δ·10⁻⁵ cm	Fe	Ni	Cu	δ·10⁻⁸ cm
18	0.882	35.60	64.40	-	0.73	39.60	55.50	4.65	1.75
19	1.158	36.84	63.16	-	0.99	53.32	41.55	5.12	1.88
20	1.032	37.50	62.50	-	0.54	51.17	43.51	5.31	1.68
21	1.082	41.86	58.14	-	0.48	51.16	43.50	5.34	2.01
22	1.007	41.40	58.59	-	1.53	51.20	43.22	5.58	2.25
23	1.007	41.18	58.82	-	1.02	50.89	43.81	5.29	1.61
average	1.028	39.06	60.94		0.88	49.56	54.18	5.22	1.86
The samples, sealed in the ampoules									
24	0.606	61.96	38.28	-	5.15	55.10	39.49	5.41	1.58
25	0.631	52.07	39.94	7,99	5.99	46.01	49.08	4.60	1.56
26	0.710	50.53	43.01	6,45	7.80	47.71	47.09	4.83	3.44

Upon etching τ> 2 min; upon polishing τ> 13 sec

The ratio of these metals in the solution (39 % Fe and 61 % Ni) is slightly different from that in the original alloy (47 % Ni, 48% Fe, i.e. 1:1). In case of etching the samples sealed into ampoules, in addition to nickel and iron, copper is also fixed in the solution. The etching process is accompanied by the formation of small symmetrical grooves on the surface of the material being processed, the thickness of the removed layer reaches the following values: unsealed samples - $\delta = 0.9 \cdot 10^{-5}$ cm; sealed - $\delta = (5–7) \cdot 10^{-5}$ cm.

In the polishing solution after processing the both types of the samples, the ratio of the metals is close to the initial composition of the 47ND alloy. The thickness of the removed layer in this case $\delta = (1.6–2) \cdot 10^{-3}$ cm is higher than in the case of Fe, Ni and 13X steel, due to this, the deep surface treatment is ensured (Fig. 4).



а) б)

Figure 4 – The surface of the alloy 47ND after the treatments:
a) etching, b) polishing

The behavior of the alloys containing the Monel 40A copper and nickel silver

When processing the Monel 40A alloys and nickel silver by the etching solution, the main components of the alloys are released into the solution: copper and nickel in case of the Monel 40A, and in nickel silver, zinc is also fixed (Tables 6 and 7).

Table 6 - The results of an analysis of the solutions after the chemical treatment of Monel NM 40A alloy (q = 0.042 cm)

Experiment No.	Sample area S, cm^2	Etching			Polishing		
		The ratio of the metals in the solution. wt.%		Thickness of the removed layer	The ratio of the metals in the solution. wt.%		Thickness of the removed layer
		Cu	Ni	$\delta \cdot 10^{-3}$ cm	Cu	Ni	$\delta \cdot 10^{-3}$ cm
27	0.50	-	-	1.30	38.00	62.00	0.56
28	0.49	-	-	1.15	36.90	63.10	0.59
29	0.53	44.44	55.56	0.96	36.36	64.64	0.64
The samples, sealed in the ampoules							
30	0.41	41.40	53.20	5.72	29.63	70.37	0.56
31	0.43	41.18	53.03	8.64	27.93	72.07	0.72
32	0.39	39.06	55.56	5.57	29.34	70.65	0.66

Table 7 - The results of an analysis of the solutions after the chemical treatment of nickel silver alloy (platinum 0.4x3) prior to amalgamation

Experiment No.	Sample area S, cm^2	Etching				Polishing			
		The ratio of the metals in the solution. wt.%			Thickness of the removed layer $\delta \cdot 10^{-5}$ cm	The ratio of the metals in the solution. wt.%			Thickness of the removed layer $\delta \cdot 10^{-3}$ cm
		Cu	Zn	Ni		Cu	Zn	Ni	
33	2.28	53.36	24.21	22.42	1.15	59.74	22.98	17.28	1.12
34	2.64	54.93	23.94	21.13	1.26	62.50	21.88	15.62	1.06
35	2.45	50.23	22.37	27.40	1.05	61.53	20.51	17.96	1.16
36	1.58	50.81	22.58	26.61	0.92	66.64	24.15	13.55	1.03
37	1.60	49.22	25.00	25.78	0.94	62.90	20.97	16.13	2.26^{x}

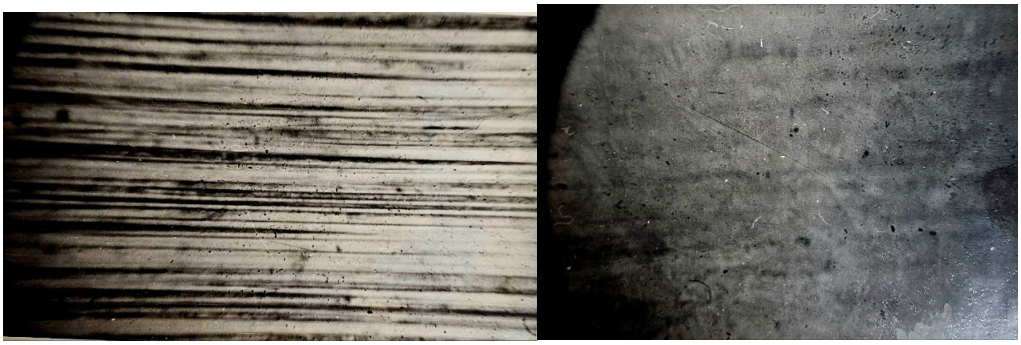
The thickness of the removed layer is of the order of $(1–1.3) \cdot 10^{-5}$ cm for the both materials. The behavior of these alloys in the polishing solution is the same. The ratio of the components, passing into the solution is close to the original composition of the materials, and the thickness of the removed layer is almost two orders of magnitude higher than during etching: Monel 40A – $\delta = (0.6\text{-}0.7) \cdot 10^{-3}$ cm; nickel silver – $\delta = (1–1.2) \cdot 10^{-3}$ cm. Processing

in the polishing solution ensures a deep surface treatment, due to which its composition and micro-relief are leveled. As can be seen from Figures 5 and 6, the surface of the samples becomes smooth, uniform and shiny.

а) б)

Figure 5 – The Monel 40A alloy surface after the treatments:

a) etching, b) polishing



а) б)

Figure 6 – The nickel silver alloy surface after the treatments:
a) etching, b) polishing

Conclusion

The behavior of the iron group metals and alloys on their basis in the processes of chemical preparation of their surfaces prior to applying mercury coatings has been studied. For the both stages of the chemical processing - etching and polishing - the thickness of the removed layer of material has been determined, and in case of alloys, also the ratio in which the metals go into the solution has been determined. The optimal conditions for polishing iron, 13X steel and nickel silver alloy have been selected. It has been established that during the etching process of Ni, Fe, 47ND a layer with a thickness of $\delta = (1–2)\cdot10^{-4}$ cm is removed, and for steel 13X $\delta = 2.10^{-5}$ cm, and during polishing a deeper development of the sample surfaces occurs. The thickness of the removed layer on Ni, Fe is $\delta = (3–5)\cdot10^{-4}$ cm, and on 13X and 47ND, respectively, $7\cdot10^{-4}$ cm and $(1.6–2)\cdot10^{-4}$ cm. In case of processing the 47ND alloy, iron and nickel pass into the solution, and during etching the Fe:Ni ratio is 1:1.6, and

during polishing it is 1:1, i.e. close to the original alloy composition.

During the chemical preparation of the copper-containing alloys — Monel 40A and nickel silver, the main components of the alloys — copper and nickel, and nickel silver also contain zinc — are transferred into the solution. The thickness of the layer, removed on the both materials during etching, is of the same order, and during polishing it is $\delta = (1–1.3)\cdot10^{-5}$ cm, i.e. two orders of magnitude higher, and the ratio of the components, which have gone into the solution is close to the original composition of the alloys. Processing in a polishing solution ensures a good preparation of the surface of the alloys, due to which its composition and micro-relief are leveled.

Thus, the data obtained on the behavior of the iron group metals and alloys on their basis in the processes of etching and polishing indicate that during the chemical treatment the surface is completely cleaned of oxides, and the ratio of the layer surface to their chemical composition is achieved.

The final conclusion about the suitability of the developed conditions for the chemical preparation of the surface of metals and alloys can be made only on the basis of assessing the quality of the mercury film during subsequent amalgamation**.**

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