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<https://doi.org/10.31643/2019/6445.08>AKBAROV M. S.¹, CHERNYSHOVA O. V.², USSOLTSEVA G. A.¹, A. AKCHIL³¹Satbayev University, Almaty, Kazakhstan, *e-mail: akbarov_meron@mail.ru²Russian Technological University, Moscow, Russia³University of Suleyman Demirel, Isparta, Turkey**ELECTROCHEMICAL PROCESSING OF TIN-CONTAINING SOLDERS WITH THE USE OF ELECTROLYTE BASED ON POTASSIUM HYDROXIDE**

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Abstract. This article is dedicated to the processing of the secondary tin-containing alloys by the electrochemical method. The provided article reviews exploratory studies regarding the anodic dissolution of the lead-tin solders, conducted by the ECC-1012 electrochemical complex in galvanostatic mode with using as an electrolyte solution of potassium hydroxide. The anodic dissolution was demonstrated to go differently depending on the concentration of alkaline electrolyte. For instance, at 50 g/dm³ concentration of potassium hydroxide, the dissolution commence was recorded at a 0.4 V capacity and the reaction goes predominantly with the HSnO₂⁻ formation, also in this case the lead dissolution is unlikely. After that, the anode passivation takes place owing to the formation of oxidation films of the metals and oxygen release. The alkali concentration increases within the electrolyte to 100 g/dm³ leads to a dramatic change in the current potential curves, corresponding to the formation of HSnO₂⁻, HPbO₂⁻, Sn₂⁺ and Pb₂⁺. In addition, an increase of alkali concentration in the solution will contribute to a more active dissolution of the metals, even at the initial stage. As the anode surface develops, it beneficiates alternately with either lead or tin. The formation of tetravalent lead and tin ions upon anodic dissolution of lead-tin alloys in the alkali solutions is unlikely. Within the process of anodic dissolution, an electrolytic sludge was obtained, which contained not only a tin but also the copper, lead, antimony, aluminum and iron have been found.

Keywords: lead-tin solders, alkaline electrolyte, anodic dissolution, electrochemical polarization.

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КҮЙДІРГІШ КАЛИЙ НЕГІЗІНДЕГІ ЭЛЕКТРОЛИТТІ ҚОЛДАНУ АРҚЫЛЫ ҚАЛАЙЫЛЫ ҚОРЫТПАЛАРДЫ ЭЛЕКТРОХИМИЯЛЫҚ ҚАЙТА ӨНДЕУІ

Түйіндемe. Бұл мақалада қалайы қорытпалардың электрохимиялық әдісімен қайта өңдеу зерттемелері қарастырылған. Мақалада каустикалық калийінің электролит ерітіндісі ретінде гальваностатикалық режимде ЕНК-1012 электрохимиялық комплексі арқылы өткізілген қорғасын қалаушылардың анодтық еруі туралы зерттеу жұмыстары қарастырылған. Сілтілік электролиттің концентрациясына байланысты анодтық ерігіштігі басқаша көрсетіледі. Осылайша, 50 г/дм³ каустикалық калий концентрациясында ерітіндінің басталуы 0,4 В әлеуетінде тіркелді, реакция көбінесе HSnO₂⁻ қалыптасуымен жүреді, бұл жағдайда қорғасынның еруі екіталай емес. Содан кейін анодты пассивациялау тотықты металдардың қабатының пайда болуымен және оттегін шығаруына байланысты өтеді. Электролит құрамында 100 г/дм³ дейін сілтіліктің концентрациясын жоғарылату HSnO₂⁻, HPbO₂⁻, Sn₂⁺ және Pb₂⁺ түзілуіне сәйкес келетін поляризация қисықтардың түрлерінің күрт өзгеруіне әкеледі. Сонымен қатар, ерітіндідегі сілтілі концентрацияның ұлғаюы бастапқы сатысында да металдардың ерітілуіне әсер

етеді. Анод беті дамыған сайын қорғасын немесе қалайы бар байытылады. Сілтілік ерітінділердегі қорғасынның қорытпаларын анодтық ерітіп алуда тетравалентальді қорғасын мен қалайы иондарының пайда болуы екіталай емес. Анодтық еру кезінде электролит шламы алынды, онда қалайы, мыс, қорғасын, сурьма, алюминий мен темір табылды.

Түйін сөздер: Қорғасынды-қалайылы қорытпа, сілтілі электролит, анодты еріту, электрохимиялық поляризация.

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ЭЛЕКТРОХИМИЧЕСКАЯ ПЕРЕРАБОТКА ОЛОВСОДЕРЖАЩИХ ПРИПОЕВ С ИСПОЛЬЗОВАНИЕМ ЭЛЕКТРОЛИТА НА ОСНОВЕ ЕДКОГО КАЛИЯ

Резюме. Настоящая статья посвящена вопросам переработки вторичных оловосодержащих сплавов электрохимическим методом. В статье рассматриваются поисковые исследования по анодному растворению свинцово-оловянных припоев, проводимые с использованием электрохимического комплекса ЭХК-1012 в гальваностатическом режиме с использованием в качестве электролита раствора едкого калия. Показано, что в зависимости от концентрации щелочного электролита анодное растворение протекает по-разному. Так, при концентрации едкого калия 50 г/дм³ начало растворения зафиксировано при потенциале 0,4 В с преимущественным протеканием реакции с образованием HSnO₂⁻, растворение свинца при этом маловероятно. Затем происходит пассивация анода из-за образования оксидных пленок металлов и выделение кислорода. Увеличение концентрации щелочи в составе электролита до 100 г/дм³ приводит к резкому изменению вида поляризационных кривых, соответствующих образованию HSnO₂⁻, HPbO₂⁻, Sn²⁺ и Pb²⁺. Кроме того, рост концентрации щелочи в растворе будет способствовать более активному растворению металлов даже на начальной стадии. По мере развития анодной поверхности она обогащается попеременно либо свинцом, либо оловом. Образование четырехвалентных ионов свинца и олова при анодном растворении свинцово-оловянных сплавов в растворах щелочей маловероятно. В ходе анодного растворения был получен электролитический шлак, в составе которого помимо олова обнаружены медь, свинец, сурьма, алюминий и железо.

Ключевые слова: свинцово-оловянный припой, щелочной электролит, анодное растворение, электрохимическая поляризация.

Introduction. The dry and hydrometallurgy, electrochemical dissolution methods, and their combination may be applied to process the secondary alloys of non-ferrous metals, including tin-containing ones [1-5]. A general shortcoming of the dry metallurgy methods are the significant energy costs of the primary operations and the need for the hermetic equipment to trap volatile components released during high-temperature processing, especially in the case of lead-tin alloys melting. Despite the volatility of the tin-containing components of scrap and solders, their treatment to produce another tin-containing product (solders, bronze, pure tin) is frequently carried out by remelting [6-8].

An acid leaching, extraction, ion exchange and chemical precipitation are the main frequent stages at implementing of hydrometallurgical processing of the secondary alloys of non-ferrous metals [9]. The electrochemical methods are allow significantly to simplify the process flow sheet and instrumentation, to reduce the processing time of the secondary waste and improve the environmental performances under the high rates of valuable components extraction [10-12]. Thus, their application may be of interest and more environmentally friendly

for the processing of the secondary lead-tin alloys, especially since, under the modern industrial development, more stringent requirements are imposed on the industrial emissions.

The electrolytic method is known to be used in the lead and tin technology, but alkaline, hydrofluorosilic and hydrofluoboric electrolytes are better preferred. For instance, when applying hydrofluorosilic and hydrofluoboric electrolytes an electrolysis is carried out in the following conditions: current density is 200-250 A/m², the cathode sediment increasing time is 80-96 hours when the electrolyte circulation arrangement. At the same time, an alloy with a Pb content of 60-65 % and Sn of 34-39 % is obtained on the cathode, which is suitable as an addition alloy in the solders manufacture [11-19].

Based on the above data, the electrochemical processing of the secondary tin-containing alloys is an up-to-date and practically significant task.

The results of these exploratory studies are aimed to establish the nature of the process of tin-containing solders anodic dissolution using alkaline electrolytes based on potassium hydroxide.

Experiments and results and discussion.

The secondary tin-containing solders with the tin content of 45 and 60 % are provided as a research target, potassium hydroxide with a concentration of 50 and 100 g/dm³ were used as the electrolyte solutions.

An ICP mass spectrometer for isotope and elemental analysis with the inductively coupled plasma ELAN DRC-e (Perkin Elmer, Canada) at the Kazakh National Research Technical University named after K.I. Satpayev and Russian University of Technology was used to perform an elemental analysis of secondary alloys samples and the products of their processing.

A KFK-3KM spectrophotometer was used to determine the tin content in aqueous solutions by the photometric method at the Kazakh National Research Technical University named after K.I. Satpayev using certified methods.

A Phenom XL scanning electron microscope from the Phenom-World company (Thermo ScientificTM, the Netherlands) was used for the primary analysis of a sample of sludge obtained during anodic dissolution in potassium hydrogen solution under FOV: 50.7 μm, Mode: 15kV - Point , Detector: BSD Full during a seminar conducted by the representatives of the Melitek international company.

Electrochemical dissolution was performed by electrochemical technological complex ECC-1012 (developed by FE Tetran LLC, RF), using a non-reparation method for the potential survey.

The electrochemical cell consisted of a 200 ml fluoroplast container, the electrodes were: the anode - tin-containing solder in the form of a plate with an area of 5.6 cm², the cathode - titanium (VT1-0) plate with an area of 5 cm².

The whole survey of the electrode potentials were carried out relative to the silver chloride reference electrode.

Results and discussion. The anodic dissolution of lead-tin alloys can proceed in the alkaline solutions, by the reactions provided in Table 1.

Inview of the provided reactions, the dissolution of the alloy in the alkaline solutions may be assumed to primary begin with the tin dissolution according to (4) reaction, that is, the formation of HSnO₂- and lead ions by (6) reaction is preferable. In weakly alkaline solutions, tin can preferentially dissolve by (4) reaction; dissolution of lead is unlikely.

Table 1 - Probable electrochemical reactions occurring at the anode, made of secondary lead-tin alloys [15]

	Reaction	Standard Electrode Potential (E°), B
1	Sn+3OH ⁻ -2e=HSnO ₂ ⁻ +H ₂ O	-0.91
2	Sn-2e=Sn ²⁺	-0.14
3	Sn-4e=Sn ⁴⁺	0.01
4	Pb+2H ₂ O-2e=HSnO ₂ ⁻ +3H ⁺	0.33
5	Pb-2e=Pb ²⁺	-0.125
6	Pb-4e=Pb ⁴⁺	0.77
7	Pb+3OH ⁻ -2e=HPbO ₂ ⁻ +H ₂ O	-0.502
8	Pb+6OH ⁻ -4e=PbO ₂ ²⁻ +3H ₂ O	-0.127
9	Pb+2H ₂ O-2e=HPbO ₂ ⁻ +H ⁺	0.702

Our assumptions were confirmed after taking the current potential curves (Figure 1) with anodic dissolution of lead-tin alloy with a tin content of about 60 % in a potassium hydroxide solution of 50 g/dm³ concentration, the beginning of dissolution was recorded at about 0.4 V potential. Then the anode passivation takes place, which may be due to the low solubility of its oxide films on the alloy surface, and the release of the oxygen.

The same alloy was subjected to repeated take of polarization curves (Figure 2), on the basis of which it can be assumed that the alloy dissolution follows by two preferred reactions, (4) and (3), further dissolution of the alloy is accompanied by the active oxygen emission, which was recorded visually.

An increase in the alkali concentration in the electrolyte composition to 100 g/dm³ leads to a dramatic change in the type of polarization curves both during the primary (Figure 3) and when the curves are taken again (Figure 4).

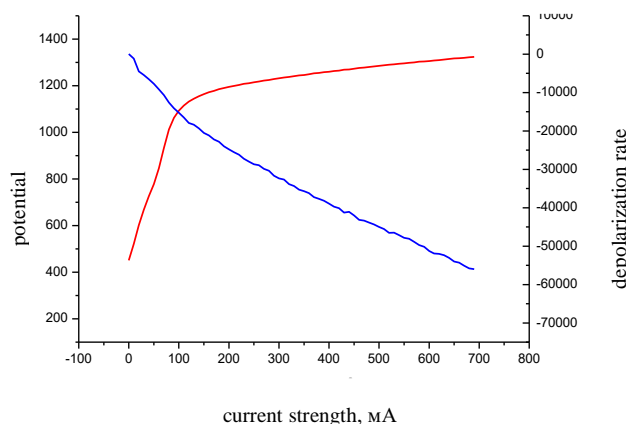


Figure 1 - — Polarizing and — Depolarizing dissolution curves of the lead-tin alloy (60.0 % wt.) in a KOH solution with a concentration of 50 g/dm³

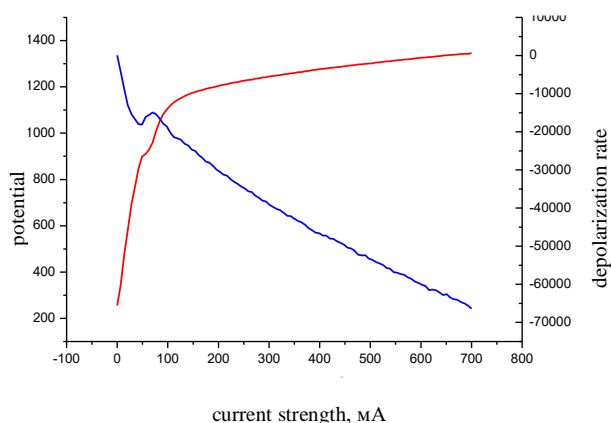


Figure 2 - — Polarizing and — Depolarizing curves of lead-tin alloy dissolution (60.0 % wt.) in a KOH solution 50 g/dm³ - repeated removal

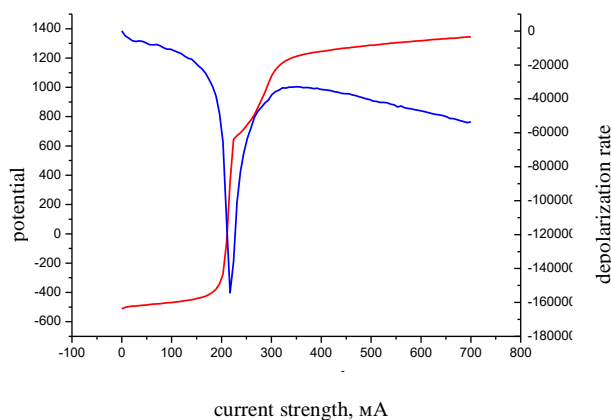


Figure 3 - — Polarizing and — Depolarizing dissolution curves of the lead-tin alloy (60.0 % wt.) in a KOH solution of 100 g/dm³

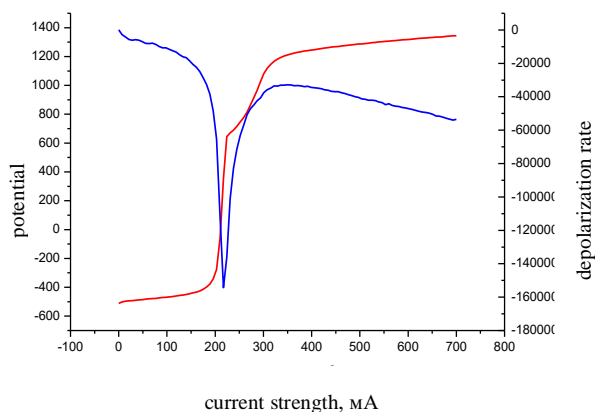


Figure 4 - — Polarizing and — Depolarizing dissolution curves of the lead-tin alloy (60.0% wt.) in the KOH solution 100 g/dm³ – taken again.

During the initial removal of the polarization curves, the preferential sequential flow of reactions (1), (7) and (2) of Table 1 may be noted. Further dissolution of the alloy is also accompanied by the oxygen emission.

As the curves are taken again (Figure 4), with the growth of the anodic surface of the dissolving alloy and the change in the surface content of the alloy components, the sequence of electrochemical reactions changes first (1), then (7), (2), (5), (8) Tables 1 and only then - the oxygen emission.

In the course of anodic dissolution of lead-tin alloy in an alkaline electrolyte, sludge was obtained, its composition was studied using a number of physicochemical research methods. An analysis of the sludge sample is carried out using a Phenom XL scanning electron microscope from Phenom-World (Thermo ScientificTM, the Netherlands), showed that the sludge is heterogeneous both in structure and composition. In addition to tin, the sludge contains copper, lead, antimony, aluminum, and iron.

Summary. Thus, it is impossible to separate lead and tin during anodic dissolution of the alloy in alkaline solutions, both metals pass into the solution. An increase in the alkali concentration in the solution will contribute to a more active dissolution of the metals even at the initial stage. As the anodic surface develops, it is enriched alternately with either lead or tin, which leads to a change in the form of polarization curves, the excesses on which correspond to the oxidation reactions of lead and tin to hydro-stannates, hydro-plumbates and even to tin (+2) cations and plumbates. The formation of the tetravalent lead and tin ions in the anodic dissolution of lead-tin alloys in alkali solutions is unlikely.

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