ISSN 2518-170X (Online),
ISSN 2224-5278 (Print)

ҚАЗАҚСТАН РЕСПУБЛИКАСЫ
ҰЛТТЫҚ ҒЫЛЫМ АКАДЕМИЯСЫ
Satbayev University

ХАБАРЛАРЫ

ИЗВЕСТИЯ
НАЦИОНАЛЬНОЙ АКАДЕМИИ НАУК
РЕСПУБЛИКИ КАЗАХСТАН
Satbayev University

NEWS
OF THE ACADEMY OF SCIENCES
OF THE REPUBLIC OF KAZAKHSTAN
Satbayev University

SERIES
OF GEOLOGY AND TECHNICAL SCIENCES

1 (451)

JANUARY – FEBRUARY 2022

THE JOURNAL WAS FOUNDED IN 1940

PUBLISHED 6 TIMES A YEAR

ALMATY, NAS RK

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«ҚР ҰҒА Хабарлары. Геология және техникалық ғылымдар сериясы».
ISSN 2518-170X (Online),
ISSN 2224-5278 (Print)
Меншіктеуші: «Қазақстан Республикасының Ұлттық ғылым академиясы» РҚБ (Алматы қ.).
Қазақстан Республикасының Ақпарат және қоғамдық даму министрлігінің Ақпарат комитетінде 29.07.2020 ж. берілген **№ KZ39VPY00025420** мерзімдік басылым тіркеуіне қойылу туралы куәлік.
Тақырыптық бағыты: *геология, мұнай және газды өңдеудің химиялық технологиялары, мұнай химиясы, металдарды алу және олардың қосындыларының технологиясы.*
Мерзімділігі: жылына 6 рет.
Тиражы: 300 дана.
Редакцияның мекен-жайы: 050010, Алматы қ., Шевченко көш., 28, 219 бөл., тел.: 272-13-19
http://www.geolog-technical.kz/index.php/en/

Типографияның мекен-жайы: «Аруна» ЖК, Алматы қ., Мұратбаев көш., 75.

«Известия НАН РК. Серия геологии и технических наук».
ISSN 2518-170X (Online),
ISSN 2224-5278 (Print)

Собственник: Республиканское общественное объединение «Национальная академия наук Республики Казахстан» (г. Алматы).

Свидетельство о постановке на учет периодического печатного издания в Комитете информации Министерства информации и общественного развития Республики Казахстан **№ KZ39VPY00025420,** выданное 29.07.2020 г.

Тематическая направленность: *геология, химические технологии переработки нефти и газа, нефтехимия, технологии извлечения металлов и их соеденений.*

Периодичность: 6 раз в год.

Тираж: 300 экземпляров.

Адрес редакции: 050010, г. Алматы, ул. Шевченко, 28, оф. 219, тел.: 272-13-19

http://www.geolog-technical.kz/index.php/en/

Адрес типографии: ИП «Аруна», г. Алматы, ул. Муратбаева, 75.

News of the National Academy of Sciences of the Republic of Kazakhstan. Series of geology and technology sciences.
ISSN 2518-170X (Online),
ISSN 2224-5278 (Print)
Owner: RPA «National Academy of Sciences of the Republic of Kazakhstan» (Almaty).
The certificate of registration of a periodical printed publication in the Committee of information of the Ministry of Information and Social Development of the Republic of Kazakhstan **No. KZ39VPY00025420,** issued 29.07.2020.
Thematic scope: *geology, chemical technologies for oil and gas processing, petrochemistry, technologies for extracting metals and their connections.*
Periodicity: 6 times a year.
Circulation: 300 copies.
Editorial address: 28, Shevchenko str., of. 219, Almaty, 050010, tel. 272-13-19
http://www.geolog-technical.kz/index.php/en/

Address of printing house: ST «Aruna», 75, Muratbayev str, Almaty.

NEWS
OF THE NATIONAL ACADEMY OF SCIENCES OF THE REPUBLIC OF KAZAKHSTAN
SERIES OF GEOLOGY AND TECHNICAL SCIENCES
ISSN 2224-5278
Volume 1, Number 451 (2022), 91-98 ***https://doi.org/10.32014/2022.2518-170X.145***

UDC 661.961.1.662.796. 2. 542.76

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ALLOYS FOR THE PRODUCTION OF HYDROGEN AND ACTIVE ALUMINUM OXIDE

Abstract. The influence of an oxidizing environment (water, hydrogen peroxide, sodium hydroxide, sulfuric and hydrochloric acid) on the yield and rate of hydrogen release, as well as the composition of the resulting oxidation products of an activated aluminum alloy containing activator metals: gallium, indium, tin (5% by weight, Rau-85 alloy), depending on temperature, as well as the dispersion of alloy particles has been studied.

The X-ray spectral analysis of the alloy was carried out on the spectrometer X-Ray of Innov-X systems. The microstructure of the alloy and its oxidation products was studied using a scanning electron microscope with energy dispersive X-ray radiation (SEM/EDXs) using an INCA ENERGY spectrometer from OXFORD INSTRUMENTS installed on a Superprobe 733 electron probe microanalyzer from JEOL at an accelerating voltage of 25 kV and a probe current of 25 nA. Based on the microstructure analysis, the phase components of alloys at the grain boundaries of Al have been identified. X-ray diffractometric analysis of the reaction products of the Rau-85 alloy formed in various oxidizing media was carried out on an automated diffractometer DRON-3 with CuKa radiation, a β-filter. Diffractogram shooting conditions: U=35 kV; I=20 mA; shooting θ-2θ; detector 2 deg/min.

X-ray diffraction analysis on a semi-quantitative basis was carried out using diffraction patterns of powder samples using the method of equal weights and artificial mixtures. Quantitative ratios of crystalline phases were determined.

Key words: hydrogen, alloys, aluminum, activating additives, oxidation, microstructure, X-ray diffraction analysis.

Introduction. The need to transition to a renewable energy based on a clean, sustainable and decarbonized energy system is driven by the declining availability of fossil fuels, the environmental concerns of air, water and soil pollution, and the challenges of combating climate change. More than 94% of CO_2 emissions are the result of the production and consumption of gas, oil and coal [1-3].

Hydrogen is the most successful replacement for hydrocarbon fuel. The combustion products of hydrogen fuel are environmentally friendly, the combustion of H_2 in pure oxygen does not lead to the formation of greenhouse gases, which determines the importance of hydrogen energy for the environment [1]. It is proposed to use aluminum and its activated alloys (AAA) as an alternative energy carrier that allows obtaining hydrogen and storing energy. To activate aluminum in order to obtain H_2 from water, aluminum alloying with low-melting metals Ga, In, Sn, Zn, Bi, Cd, Ga-based alloys including In, Sn and other metals is proposed [1-16].

According to the authors of [5, 8], the process of aluminum activation is associated with the destruction of the oxide layer by the liquid Ga-In alloy and with a subsequent radical increase in the reaction surface. The reason for the sharp decrease in the strength of the alloy is the formation of a large number of micro galvanic cells. During diffusion, gallium is incorporated into the aluminum lattice, and indium is released from the alloy. Further intergranular and bulk diffusion of the liquid metal alloy makes aluminum brittle under mechanical stress and reactive when it interacts with water.

The purpose of this research is to study the role of the oxidizing environment during the interaction of a multicomponent aluminum alloy activated with Ga, In, Sn metals (Rau-85 alloy) on the yield and rate of hydrogen evolution, as well as the composition of the resulting oxidation products depending on temperature, the nature of the oxidizing medium, dispersion alloy particles.

Methods and materials. Aluminum in granules State Standard 295-98 was purchased from JSC “Kazakhstan Electrolysis Plant”, the only aluminum producer in Kazakhstan, is part of the ERG group of enterprises (“Eurasian Group”).

Gallium State Standard 12797-77, in the form of cylindrical ingots. Weighing from 900 to 1000 g (OKP17 code 6832 0103 09) purchased from JSC “Kazakhstan Electrolysis Plant”, melting point 156.59°C, density: 7.362 (+20°C, g/cm^3).

Indium State Standard 10297-94 in the form of cylindrical ingots. Weighing from 0.05 to 1000g (brand In00) purchased from JSC “Kazakhstan Electrolysis Plant”, melting point 29.80°C, density: 5,904 (+20°C, g/cm^3).

Tin State Standard 860-75 model O_2 in the form of ingot weighing from 22-26kg.

Hydrochloric acid p.a., temperature of the azeotropic mixture (20.22% by weight) is 108.6°C, ρ = 1.16g/cm^3 (35%), used without additional purification.

Sodium hydroxide p.a., used without additional purification.

Sulfuric acid temperature of the azeotropic mixture of 98.3% H_2SO_4 and 1.7% H_2O is 338.8^{o}C, ρ = 1.83g/cm^3.

Hydrogen peroxide in the form of a substance - a solution of hydrogen peroxide 3%.

Activated aluminum alloy Rau-85 containing activator metals: gallium, indium, tin (5% by weight) was obtained by melting in a dispersed form with particle sizes of 80-1250 μm [6,7]. Aluminum was melted in an inert gas atmosphere in an alund crucible in a muffle furnace at a temperature of 850^{o}C. Activating additives were added into the molten aluminum and mixed to achieve homogeneity of the melt. The duration of the heating – exposure – cooling cycle was 1 hour, then the melt was poured into manufactured steel molds with an outer diameter of 60 mm, an inner diameter of 40 mm and a height of 40 mm to 80 mm. A powder with specified particle sizes of 80-1250 μm was made from the ingot by changing the lumen between the crusher cheeks (mm): 1, 2, 5 and 10.

Aluminum alloy Rau-85 was characterized using a scanning electron microscope with energy dispersive X-ray radiation (SEM/EDX). X-ray spectral analysis of the alloy was carried out on the X-Ray fluorescence spectrometer X-Ray Innov-X systems.

The analysis of the elemental composition of the samples and photographing in various types of radiation were performed using a spectrometer INCA ENERGY from OXFORD INSTRUMENTS installed on a Superprobe 733 electron probe microanalyzer from JEOL at an accelerating voltage of 25 kV and a probe current of 25 nA.

X-ray diffractometric analysis of the reaction products of the Rau-85 alloy formed in various oxidizing media was carried out on an automated diffractometer DRON-3 with $Cu_{K\alpha}$ radiation, a β-filter. Diffractogram shooting conditions: U=35 kV; I=20 mA; shooting θ-2θ; detector 2 deg/min. X-ray phase analysis on a semi-quantitative basis was performed using diffractograms of powder samples using the method of equal attachments and artificial mixtures. Quantitative ratios of crystal phases were determined. The interpretation of diffractograms was carried out using data from the ICDD card file: a database of PDF-2 powder diffractometric data and diffractograms of minerals pure from impurities.

Powder X-ray diffraction patterns were recorded on a D8 ADVANCE diffractometer (Bruker), α-Cu tube voltage 40 kV, current 40 mA. Processing of the obtained data of diffraction patterns and calculation of interplanar distances were carried out using the EVA software. The interpretation of samples and the search for phases were carried out using the Search/match program using the PDF-2 powder diffraction data base.

The volume of hydrogen released during the interaction of aluminum alloy Rau-85 in various oxidizing media was measured on a drum gas meter. All experiments were repeated at least three times and were conducted at a temperature of 25, 60°C and a humidity of 60%. The water heating temperature was measured with a thermometer with an accuracy of 0.1°C.

The rate of hydrogen evolution is calculated by the formula (ml/g*min):

$$W_{\text{H}} = \frac{V}{m\Delta t} \qquad (2)$$

where V is the volume of released hydrogen, m is the weight of the alloy sample;

Δt is the time between two gas clock readings.

The theoretical volume of hydrogen was calculated based on the generation of 1.244l of hydrogen per 1g of Al under standard conditions (273 K, 1 atm).

Results and discussion. X-ray spectral analysis of the Rau-85 aluminum alloy was obtained on the X-Ray Innov-X systems spectrometer showed that the main component of the activated Rau-85 alloy is aluminum, the content of which is 85.71%. It has been established that the content of metals in the alloy differs slightly from the initial ratio of metals taken for the preparation of the alloy. It was revealed that the Rau-85 alloy contains trace amounts of metal impurities that were not used as initial components: Fe, Cd, Pb, Bi in an amount of 0.01 to 0.02%, the iron content reaches 0.09%. The content of these metals in aluminum is allowed by State Standard 295-98.

SEM - image and EDXs maps of the surface of the Rau-85 alloy sample are shown in Figure 1. The study made it possible to obtain a general picture of the alloy, a fractogram of the fracture surface, the distribution of dissolved elements in the alloy and their content in separate local areas of the sample with a microprobe with a diameter of 1 μm (Figure 1). Lighter areas in the micrograph indicate the presence of liquid eutectics of activating metals on aluminum grains [5, 8].

The results of the analysis of samples by the EDXs method of bright white grains and the aluminum alloy matrix are shown in Table 1. Based on the data in the table, it can be noted that bright white grains are heterogeneous in composition, the activating components Ga, In, Sn in the composition of the alloy participate in the formation of complex eutectics, have different distributions in the volume and on the aluminum surface.

Table 1 - Results of X-ray spectral microanalysis of Rau-85 alloy

	The content of elements, weight. %					
Spectrum	Grain	Al	Ga	In	Sn	Total
Spectrum 1	Bright white grain	3.04	5,90	69,37	21,70	100,00
Spectrum 2	Bright white grain	2,26	3,59	72,42	21,70	100,00
Spectrum 3	Matrix - gray in the photo	58,49	12,54	17,70	11,27	100,00

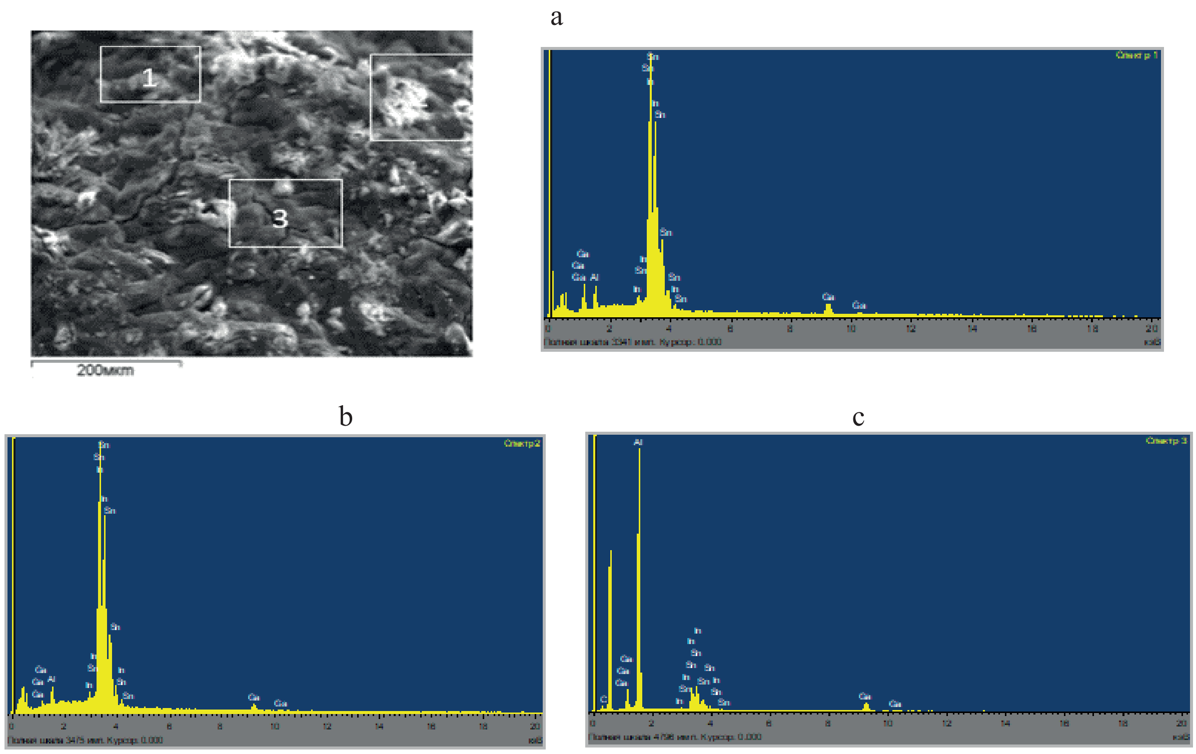


Figure 1 - SEM image of the surface of the activated Rau-85 alloy in secondary electrons and EDX maps - spectra 1(a), 2(b) bright white grain and 3(c) matrix (gray matter)

The reactivity of the activated aluminum alloy Rau-85 with respect to distilled water in terms of hydrogen evolution was studied depending on the particle size of the alloy and the reaction temperature. The particle size varied from 80 μm to 1250 μm, the process temperature was 25°C and 60°C. Comparative data on the volume of hydrogen released during the interaction of Rau-85 alloy particles of different dispersion with distilled water at a temperature of 25°C and 60°C for 60 seconds are presented in Table 2.

Table 2 - Influence of particle size of aluminum alloy Rau-85 on the yield of hydrogen when interacting with water at different temperatures

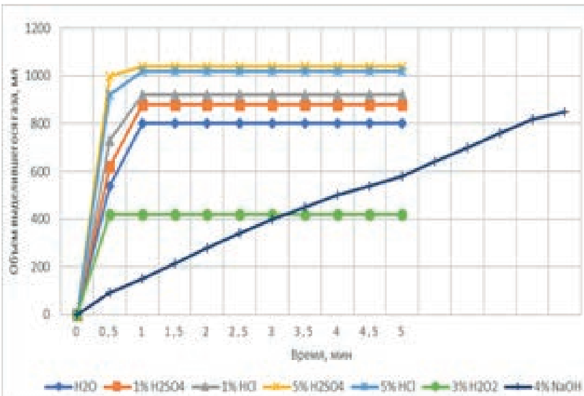
Particle sizes of Rau-85, μм	Volume of released hydrogen at 25°C, ml, reaction time 60sec	Volume of released hydrogen at 60°C, ml, reaction time 60sec
<80	980	1400
80-140	840	1300
140-630	330	1230
630-1250	110	1060
1250<	30	1000

Analysis of the data in Table 2 indicates that the size of the dispersed particles of the alloy and the temperature of the process are the key factors that control the reactivity of Al-water. The yield of hydrogen at a temperature of 25°C and a particle size of 80 μm was 980 ml in 60 seconds. Raising the reaction temperature to 60°C increases the yield of hydrogen to the theoretically possible and amounts to 1400 ml over the same period of time. An increase in the size of dispersed particles of the alloy to 1250 μm at a temperature of 25°C significantly reduces the yield of hydrogen to 30 ml. Shortly after raising the reaction temperature to 60°C for particles with a size of 1250 μm leads to an increase in the volume of released hydrogen up to 1000 ml, which is much lower than the theoretically calculated value.

The reactivity of aluminum alloys in a chemical reaction with aqueous solutions of electrolytes (NaOH, HCl, H_2SO_4, H_2O_2) was evaluated by the rate of hydrogen evolution at different temperatures. Particle size 80-140 μm.

Comparative kinetic curves of the release of H_2 during the interaction of the Rau-85 alloy over time, depending on the concentration of the studied acids and the fineness of the particles of the activated alloy, are shown in Figure 2.

An analysis of Figure 2 made it possible to conclude that, when the process is carried out at 25°C, the volume of the released gas and the rate of its release depend on the concentration of the oxidizer and its nature. With an increase in the concentration of sulfuric acid from 1% to 5%, the rate of hydrogen evolution in 60 seconds increases from 1200 ml to 1800 ml/g·min. A similar dependence was observed when using 1% and 5% hydrochloric acid as an oxidizing medium.



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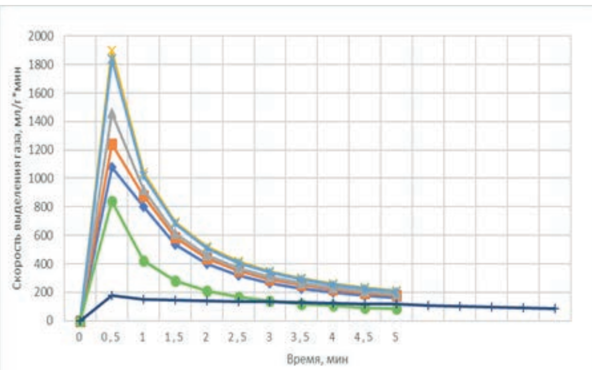

b

Figure 2 - Comparative kinetic curves of the volume of the evolved gas (a) and the rate of hydrogen evolution (b) during the interaction of the Rau-85 aluminum alloy, depending on the nature of the oxidizers at a temperature of 25°C

It has also been established that with an increase in the reaction temperature, the volume of released H_2 and the rate of its release increase, reaching the theoretically calculated value. The reaction proceeds without an induction period and ends within 1-5 minutes. It is noted that the studied acids are almost equal in oxidative activity with respect to Rau-85 aluminum alloy (Figure 2). It should be stated that with an increase in the process temperature, the difference in the oxidizing ability of aqueous solutions of acids is smoothed out. In general, an increase in the acidity of the medium leads to an increase in the rate of hydrogen evolution and its release. Conversion reaches 95-100%. It was also revealed that the nature of the oxidizing medium in which the process is carried out affects the volume of the evolved gas and the rate of interaction of the activated aluminum alloy.

The reaction proceeds at the lowest rate when a 3% solution of hydrogen peroxide and sodium hydroxide is used as an oxidizing agent. Thus, the obtained results indicate that the factors that control the reactivity of Al-water are the size of the dispersed particles of the activated aluminum alloy, temperature, and the nature of the oxidizing environment.

The microstructure and phase components of the products formed during the interaction of the Rau-85 alloy in various oxidizing media (water, H_2O_2, HCI, H_2SO_4, NaOH) depending on the temperature, nature of the oxidizing agent, and pH medium were studied by SEM with EDXs. The complex of these methods provides sufficient and objective information about the composition of the studied objects.

The results of X-ray spectral microanalysis of the reaction products of the Rau-85 alloy with distilled water (bright white grain and matrix) obtained at temperatures of 60°C and 25°C for 2 hours are represented in Figure 4 and Table 4.

Table 4 - Results of X-ray spectral microanalysis of the reaction products of the Rau-85 alloy with distilled water (bright white grain and matrix) obtained at temperatures of 25°C and 60°C for 2 hours

Spectrum	Grain	The content of elements, weight. %					
		O	Al	Ga	In	Sn	Total
25°C							
Spectrum	Bulk composition	57,82	33,16	2,65	3,55	2.82	100,00
Spectrum 1	Bright white particle	33,73	5,08	0,81	27,45	32,93	100,00
Spectrum 2	Matrix, gray matter	31.22	64,50	4,291	-	-	100,00
60°C							
Spectrum	Bulk composition from the area	54.03	42.81	2.03	0.52	0.61	100,00
Spectrum 1	Bright white particle	27.87	3.93	0.63	45.64	21.93	100,00
Spectrum 2	Bright white particle	15.11	4.64	1.21	53.99	25.05	100,00
Spectrum 3	Matrix, gray matter	48.47	48.99	2.55	-	-	100,00
The alloys are obtained by cooling the melt in vacuum. The bulk of the substance is in an amorphous state.							

Comparative results of semi-quantitative X-ray diffractometric analysis of crystalline phases of the Rau-85 alloy reaction product in various oxidizing environments at 25°C and 60°C are shown in Table 5.

Table 5 - Comparative results of semi-quantitative X-ray diffractometric analysis of crystalline phases of the Rau-85 alloy reaction product in various oxidizing environments at 25°C and 60°C

№	Reaction conditions	Phase	Chemical formula	Total Percentage [%]
1	Rau-85 alloy treated with 1% H_2SO_4 solution at 25°C for 2 hours.	Aluminum Oxide	Al_2O_3	51.7
		Alumina beta	$In_{2.42}Al_{22}O_{34.8}$	22.6
		Gallium Aluminum Oxide	$Ga_2Al_{22}O_{34}/Ga_2O\cdot 11Al_2O_3$	10.9
		Aluminum	Al	5.8
		Gallium Indium Oxide	$GaInO_3$	4.9
		Berndtite-2T	SnS_2	4.1
2	Rau-85 alloy treated with 1% H_2SO_4 solution at 60°C for 2 hours.	Aluminum Oxide Aluminum	Al_2O_3 Al	85,6 14.4
3	Rau-85 alloy treated with 5% H_2SO_4 solution at 60°C for 2 hours.	Aluminum Sulfate Hydrate	$Al_2SO_4\cdot 5H_2O$	33.3
		Aluminum Sulfate Hydroxide Hydrate	$3Al_2O_3\cdot 4SO_3\cdot H_2O$	27.8
		Aluminum Sulfate Hydrate	$Al_2(SO_4)_3\cdot 14H_2O$	21.3
		Metaalunogen, syn	$Al_2(SO_4)_3\cdot 12H_2O$	17.6
4	Rau-85 alloy treated with 5% HCl solution at 60°C for 2 hours	Chloraluminite, syn	$AlCl_3\cdot 6H_2O$	93.9
		Gallium Chloride	$GaCl_3$	6.1

5	Rau-85 alloy treated with 3% H_2O_2 solution at 60℃ for 2 hours.	Aluminum Oxide	Al_2O_3	39.2
		Alumina beta	$In_{2.42}Al_{22}O_{34.8}$	16.6
		Aluminum	Al	15.1
		Boehmite	AlO(OH)	10.7
		Gallium Aluminum Oxide	$Ga_2Al_{22}O_34/Ga_2O{\cdot}11Al_2O_3$	9.0
		Tin Oxide	SnO_2	5.1
		Gallium Indium Oxide	$GaInO_3$	4.4
6	Rau-85 alloy treated with 4% NaOH solution at 60℃ for 2 hours.	Sodium Aluminum Oxide	$NaAlO_2$	100
7	Rau-85 alloy with distilled water at 25°C for 2 hours.	Aluminum Tin	Al0.975Sn0.025	77,1%
		Indium Tin	(In3Sn)0.5	9,8%
		Indium Tin	(InSn4)0.2	7,6%
		Boehmite, syn	AlO(OH)	5,5%
8	Rau-85 alloy with distilled water at 60°C for 2 hours.	Aluminum Oxide	Al_2O_3	77.4
		Tin Oxide	Sn_3O_4	10.0
		Indium	In	7.0
		Tin Oxide	SnO_2	5.6
9	Rau-85 alloy with distilled water at 60°C for 2 hours.	Aluminum	Al	49,34
		Indium Tin	(In3Sn) 0.5	23,17
		Indium Tin	In0.1818 Sn0.8182	18,34
		Aluminum Oxide Hydroxide	Al O (O H)	9,15
Sample 7, 9 was recorded on D8 Advance (Bruker), α-Cu, tube voltage 40 kV, current 40 mA. Processing of the obtained data of diffraction patterns and calculation of interplanar distances were carried out using the EVA software. The interpretation of samples and the search for phases were carried out using the Search/match program using the PDF-2 Powder Diffraction Data Base.				

According to X-ray diffractometric analysis data (Table 5), the reaction products of Rau-85 aluminum alloy with various oxidizing agents are heterogeneous in composition. The composition of the resulting phases and their total percentage depends on the temperature, the nature of the oxidizer used for the reaction, and the concentration of the acid. The diffraction patterns of the samples contain a mixture of crystalline and amorphous phases. There is evidence to indicate that sample №1 contains an X-ray amorphous phase that contains sulfur, samples №3-№6 contain an X-ray amorphous phase of hydrates of aluminum sulfate, aluminum chloride, aluminum boehmite, sodium aluminate, samples №7-№9 contain X-ray amorphous boehmite phase.

Conclusion. Based on the results obtained, it can be concluded that the reactivity of the alloy with respect to water can be controlled by changing the oxidizing environment by introducing appropriate additives to water, changing their amount, as well as varying the temperature of the experiment and the dispersion of particles.

The composition of the resulting phases and their total percentage depends on the temperature, the nature of the oxidizer used for the reaction.

The work was carried out at the expense of grant funding from the Ministry of Education and Science of the Republic of Kazakhstan IRN AP09260008.

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СУТЕГІ МЕН БЕЛСЕНДІ АЛЮМИНИЙ ОКСИДІН ӨНДІРУГЕ АРНАЛҒАН ҚОРЫТПАЛАР

Аннотация. Тотығу ортасының (су, сутегі асқын тотығы, натрий гидрототығы, күкірт және тұз қышқылы) сутектің шығуы мен шығарылу жылдамдығына әсері, сондай-ақ құрамында активатор металдар: галлий, индий, қалайы (5% масса, Rau-85 қорытпасы) бар активтендірілген алюминий қорытпасының тотығу өнімдерінің құрамы температураға, сондай-ақ қорытпа бөлшектерінің дисперсиясына байланысты зерттелді.

Қорытпаның рентгендік спектрлік талдауы X-Ray Innov-X systems спектрометрінде жүргізілді.

Қорытпаның микроқұрылымдары мен оның тотығу өнімі Superprobe 733 электронды-зондты микроанализаторына орнатылған Oxford INSTRUMENTS фирмасының INCA ENERGY спектрометрін, 25 кВ үдеткіш кернеуі және 25 зонд тогы бар JEOL фирмасын пайдалана отырып,энергодисперсиялық рентген сәулесімен сканерлейтін электрондық микроскоп (СЭМ/ EDXs) әдістерімен зерттелді. Микроқұрылымды талдау негізінде Al түйірлері шекарасындағы қорытпалардың фазалық компоненттері анықталды. Әртүрлі тотығу орталарында түзілетін Rau-85 қорытпасының реакциясының өнімін рентгендік дифрактометриялық талдау $Cu_{K\alpha}$ электронды сәулелену, β-сүзгісі бар ДРОН – 3 автоматтандырылған дифрактометрінде жүргізілді. Дифрактограммаларды түсіру шарттары: U=35 кВ; I=20 мА; түсірілім θ-2θ; детектор 2 град/мин.

РФА жартылай сандық негізде ұнтақ сынамаларының дифрактограммалары бойынша тең аспалар мен жасанды қоспалар әдісін қолдана отырып жүзеге асырылды. Кристалдық фазалардың сандық қатынасы анықталды.

Түйінді сөздер: сутегі, қорытпалар, алюминий, белсендіретін қоспалар, тотығу, микроқұрылым, РФА.

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СПЛАВЫ ДЛЯ ПРОИЗВОДСТВА ВОДОРОДА И АКТИВНОГО ОКСИДА АЛЮМИНИЯ

Аннотация. Изучено влияние окислительной среды (вода, перекись водорода, гидроокись натрия, серная и соляная кислота) на выход и скорость выделения водорода, а также состав образующихся продуктов окисления активированного сплава алюминия, содержащего металлы-активаторы: галлий, индий, олово (по 5% масс, сплав Rau-85) в зависимости от температуры, а также дисперсности частиц сплава.

Рентгеноспектральный анализ сплава осуществляли на спектрометре X-Ray Innov-X systems. Микроструктура сплава и продуктов его окисления изучена методами сканирующего электронного микроскопа с энергодисперсионным рентгеновским излучением (СЭМ/ EDXs) с использованием спектрометра INCA ENERGY фирмы OXFORD INSTRUMENTS, установленного на электронно-зондовый микроанализатор Superprobe 733, фирмы JEOL при ускоряющем напряжении 25 кВ и токе зонда 25 нА. На основе анализа микроструктуры идентифицированы фазовые составляющие сплавов на границах зерен Al. Рентгенодифрактометрический анализ продуктов реакции сплава Rau-85, образующихся в различных окислительных средах, осуществляли на автоматизированном дифрактометре ДРОН-3 с $Cu_{K\alpha}$ – излучением, β-фильтр. Условия съемки дифрактограмм: U=35 кВ; I=20 мА; съемка θ-2θ; детектор 2 град/мин.

РФА на полуколичественной основе осуществлен по дифрактограммам порошковых проб с применением метода равных навесок и искусственных смесей. Определялись количественные соотношения кристаллических фаз.

Ключевые слова: водород, сплавы, алюминий, активирующие добавки, окисление, микроструктура, РФА.

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CONTENTS

Absametov M.K., Itemen N.M., Murtazin Ye.Zh., Zhexembayev E.Sh., Toktaganov T.Sh.
FEATURES OF THE ISOTOPIC COMPOSITION OF GROUNDWATER IN THE MANGYSTAU REGION............6

Akimbek G.A., Aliyarov B.K., Badaker V.C., Akimbekova Sh.A.
METHODOLOGY AND EXPERIMENTAL SETUP FOR THE STUDY OF RELATIVE ABRASIVENESS OF BULK SOLIDS............14

Baibolov K., Artykbaev D., Aldiyarov Zh., Karshyga G.
EXPERIMENTAL INVESTIGATIONS OF THE COARSE-GRAINED SOIL IN THE DAM OF THE PSKEM HEP............21

Bolatova A., Kuttybayev A., Кainazarov A., Hryhoriev Yu., Lutsenko S.
USE OF MINING AND METALLURGICAL WASTE AS A BACKFILL OF WORKED-OUT SPACES............33

Hajiyeva G.N., Hajiyeva A.Z., Dadashova Kh.D.
IMPACT OF URBAN LANDSCAPE POLLUTION ON HUMAN HEALTH............39

Hayitov O.G., Zokirov R.T., Agzamov O.O., Gafurov Sh.O., Umirzoqov A.A.
CLASSIFICATION OF HYDROCARBON DEPOSITS IN THE SOUTH-EASTERN PART OF THE BUKHARA-KHIVA REGION, JUSTIFICATION OF ITS METHODOLOGY AND ANALYSIS OF THE RESULTS............46

Kabylbekov K.A., Abdrakhmanova Kh.K., Kuatbekova R.A., Makhanov T.S., Urmashev B.
COMPUTER SIMULATION OF RADIONUCLIDE ISOTOPE SEPARATION USED IN NUCLEAR ENERGY AND MEDICINE............53

Kassenov A.Zh., Abishev K.K., Absadykov B.N., Yessaulkov V.S., Bolatova A.B.
ANALYSIS AND JUSTIFICATION OF THE LAYOUT OF A MULTIPURPOSE MACHINE FOR THE DEVELOPMENT OF MINERAL DEPOSITS............63

Kaumetova D.S., Koizhanova A.K., Toktar.G., Magomedov D.R., Abdyldaev N.N.
STUDY OF THE FINELY-DISPERSED GOLD RECOVERY PARAMETERS............69

Rakhmanova S.N., Umirova G.K., Ablessenova Z.N.
STUDY OF THE GREATER KARATAU'S SOUTH-WEST BY RANGE OF GEOPHYSICAL SURVEYS IN SEARCH OF THE CRUST-KARST TYPE POLYMETALLIC MINERALISATION............76

Oitseva T.A., D′yachkov B.A., Kuzmina O.N., Bissatova A.Y., Ageyeva O.V.
LI-BEARING PEGMATITES OF THE KALBA-NARYM METALLOGENIC ZONE (EAST KAZAKHSTAN): MINERAL POTENTIAL AND EXPLORATION CRITERIA............83

Sarmurzina R.G., Boiko G.I., Lyubchenko N.P., Karabalin U.S., Demeubayeva N.S.
ALLOYS FOR THE PRODUCTION OF HYDROGEN AND ACTIVE ALUMINUM OXIDE............91

Suleyev D.K., Uzbekov N.B., Sadykova A.B.
MODERN APPROACHES TO SEISMIC HAZARD ASSESSMENT OF THE TERRITORY OF KAZAKHSTAN............99

Temirbekova M.N., Temirbekov N.M., Wojcik W., Aliyarova M.B., Elemanova A.A.
THE USE OF ORGANIC FRACTION OF SOLID HOUSEHOLD WASTE TO GENERATE ETHANOL AND BIOGAS USING A SIMULATION MODEL............105

Tulegulov A.D., Yergaliyev D.S., Bazhaev N.A., Keribayeva T.B., Akishev K.M.
METHODS FOR IMPROVING PROCESS AUTOMATION IN THE MINING INDUSTRY..................115

Tulemisova G., Abdinov R., Amangosova A., Batyrbaeva G.
STUDY OF THE BOTTOM SEDIMENTS OF RESERVOIRS OF URAL-CASPIAN BASIN................126

Turgazinov I.K. Mukanov D.B.
ANALYSIS OF FLUID FILTRATION MECHANISMS IN FRACTURED RESERVOIRS......................135

Uakhitova B., Ramatullaeva L.I., Imangazin M.K., Taizhigitova M.M., Uakhitov R.U.
ANALYSIS OF THE LEVEL OF OCCUPATIONAL INJURIES ON THE EXAMPLE
OF AN INDUSTRIAL ENTERPRISE OF A METALLURGICAL CLUSTER...145

Yurii Feshchuk, Vadym Nizhnyk, Valeriia Nekora, Oleksandr Teslenko
IMPROVING THE SYSTEM FOR RESPONDING TO FIRE IN AREAS CONTAMINATED
BY THE CHERNOBYL DISASTER..152

Sherov A.K., Myrzakhmet B., Sherov K.T., Absadykov B.N., Sikhimbayev M.R.
METHOD FOR SELECTING THE LOCATION OF THE CLEARANCE FIELDS OF THE LANDING
SURFACES OF GEAR PUMP PARTS WITH A BIAXIAL CONNECTION..159

Khamroyev J.Kh., Akmalaiuly K., Fayzullayev N.
MECHANICAL ACTIVATION OF NAVBAHORSK BENTONITE AND ITS TEXTURAL
AND ADSORPTION CHARACTERISTICS..167

Zhurinov M.Zh., Teltayev B.B., Aitbayev K.A., Loprencipe G., Tileu K.B.
MODELING OF NON-STATIONARY TEMPERATURE MODE OF A MULTI-LAYER
ROAD STRUCTURE..175

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ISSN 2518-170X (Online),
ISSN 2224-5278 (Print)

Редакторы: *М.С. Ахметова, А. Ботанқызы, Д.С. Аленов, Р.Ж. Мрзабаева*
Верстка на компьютере *Г.Д.Жадыранова*

Подписано в печать 14.02.2022.
Формат 60х881/8. Бумага офсетная. Печать – ризограф.
11,5 п.л. Тираж 300. Заказ 1.

Национальная академия наук РК
050010, Алматы, ул. Шевченко, 28, т. 272-13-19