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Satbayev University

Х А Б А Р Л А Р Ы

ИЗВЕСТИЯ

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НАУК РЕСПУБЛИКИ
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HYDROGEN OBTAINING FROM THE SYSTEM ACTIVATED ALUMINUM – WATER

Abstract. New economically viable alloys have been developed that exclude the use of scattered non-ferrous metals and contain Wood's, Rose's and Darcet's alloys as activator metals.

The applicability of Wood's, Rose's and Darcet's eutectic alloys as activating additives for activating aluminum and obtaining efficient environmentally friendly sources of hydrogen production and storage has been theoretically substantiated and experimentally confirmed.

The parameters of interaction of aluminum alloys activated with Darcet's, Wood's, Rose's alloys with water are optimized depending on the temperature, the amount of the activating additive in the alloy, and the pH of the water.

The X-ray spectral analysis of the alloys was carried out on an X-Ray Innov-X systems spectrometer. The microstructure of the alloy and its oxidation products was studied using an energy-dispersive X-ray scanning electron microscope (SEM/EDXs using an OXFORD INSTRUMENTS INCA ENERGY spectrometer mounted on a JEOL Superprobe 733 electron probe microanalyzer at an accelerating voltage of 25 kV and a probe current of 25 nA. Based on the analysis of the microstructure, the phase components of the alloys at the Al grain boundaries were identified. X-ray diffraction analysis of the reaction products of the alloys formed in various oxidizing media was carried out on an automated DRON-3 diffractometer with Cu_K radiation, β-filter. Conditions for shooting diffraction patterns: 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: water, hydrogen, alloys, aluminum, activating additives, Wood, Rose, Darcet alloys, oxidation, microstructure, XRF.

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БЕЛСЕНДІРІЛГЕН АЛЮМИНИЙ - СУ ЖҮЙЕСІНЕН СУТЕК АЛУ

Аннотация. Шашыраңқы түсті металдарды қолдануды болдырмайтын және активтендіргіш металдар ретінде құрамында Вуд, Розе және Дарсе қорытпалары бар жаңа экономикалық тиімді қорытпалар әзірленді. Вудтың, Розе және Дарсенің эвтектикалық қорытпаларының алюминийді белсендіру және сутегін өндіру мен сақтаудың тиімді экологиялық таза көздерін алу үшін белсендіруші қоспалар ретінде қолдану мүмкіндігі теориялық тұрғыдан негізделіп, тәжірибе жүзінде расталды.

Дарсе, Вуд, Розе қорытпаларымен белсендірілген алюминий қорытпаларының сумен әрекеттесу параметрлері температураға, қорытпадағы активтендіргіш қоспаның мөлшеріне және судың pH деңгейіне байланысты оңтайландырылған.

Қорытпалардың рентгендік спектрлік талдауы X-Ray Innov-X жүйесінің спектрометрінде жүргізілді. Қорытпаның микроқұрылымы және оның тотығу өнімдері энергия-дисперсиялық рентгендік сканерлеуші электронды микроскоптың (SEM/EDXs) көмегімен JEOL Superprobe 733 электрон зондының 25 кВ жылдамдықпен кернеуі микроанализаторына орнатылған OXFORD INSTRUMENTS INCA ENERGY спектрометрі арқылы зерттелді және зонд тогы 25 нА. Микроқұрылымды талдау негізінде Al түйіршіктерінің шекарасындағы қорытпалардың фазалық компоненттері анықталды. Әртүрлі тотықтырғыш ортада түзілген қорытпасының реакция өнімдерінің рентгендік дифракциялық талдауы Cu_K сәулеленуі, β-сүзгісі бар автоматтандырылған DRON-3 дифрактометрінде жүргізілді. Дифракциялық үлгілерді түсіру шарттары: U=35 кВ; I=20 мА; ату θ-2θ; детектор 2 градус/мин.

Жартылай сандық негізде рентгендік дифракциялық талдау бірдей салмақтар мен жасанды қоспалар әдісін қолдану арқылы ұнтақ үлгілерінің дифракциялық үлгілерін қолдану арқылы жүргізілді. Кристалды фазалардың сандық қатынасы анықталды.

Түйін сөздер: су, сутегі, қорытпалар, алюминий, активтендіргіш қоспалар, Вуд, Дарсе, Розе қорытпалары, тотығу, микроқұрылым, РФА.

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ПОЛУЧЕНИЕ ВОДОРОДА ИЗ СИСТЕМЫ АКТИВИРОВАННЫЙ АЛЮМИНИЙ - ВОДА

Аннотация. Разработаны новые экономически целесообразные сплавы, исключающие применение цветных рассеянных металлов и содержащие в качестве металлов – активаторов сплавы Вуда, Розе и Дарсе.

Теоретически обоснована и экспериментально подтверждена применимость эвтектических сплавов Вуда, Розе и Дарсе в качестве активирующих добавок для активации алюминия и получения эффективных экологически чистых источников получения и хранения водорода.

Оптимизированы параметры взаимодействия сплавов алюминия, активированных сплавами Дарсе, Вуда, Розе с водой, в зависимости от температуры, количества активирующей добавки в сплаве, pH воды.

Рентгеноспектральный анализ сплавов 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 сплавов, образующихся в различных окислительных средах, осуществляли на автоматизированном дифрактометре ДРОН-3 с Cu_K – 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: вода, водород, сплавы, алюминий, активирующие добавки, сплавы Вуда, Розе, Дарсе, окисление, микроструктура, РФА.

Introduction. It is proposed to use aluminum (Kumar et. al., 2020; Trowell K.A. et. al., 2020; Martina Pini et. al., 2020; He et. al., 2016 a) and its activated alloys (AAA) as an alternative energy carrier that allows producing hydrogen and storing energy (Sarmurzina et. al., 2015; He et. al., 2016 b, 2020 a; Qiao et. al., 2018; Du et. al., 2018; Xiao et. al., 2018; Cundi Wei et.al., 2017, He et.al., 2020 b). Previously (Sarmurzina et.al., 2022), we studied the effect of an oxidizing medium (water, hydrogen peroxide, sodium hydroxide, sulfuric and hydrochloric acid) on the yield and rate of hydrogen evolution, as well as the composition of the resulting oxidation products of an activated aluminum alloy containing activator metals: gallium, indium, tin (by 5% mass, alloy Rau-85) depending on the temperature, as well as the fineness of the particles of the alloy. It was 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 purpose of this study is to develop a simple in composition and method for producing aluminum activated by the eutectics of low-melting metal alloys Wood, Rose and Darcet to produce hydrogen, with a high, controlled completeness of gas evolution and reducing the cost of the alloy.

Activating additives differ in the content of metals bismuth, tin, lead and cadmium. In the case of activated aluminum, diffusion of activator metals into the deep layers of aluminum occurs, opening the interface, causing cracking and destruction, which leads to a sharp increase in the reaction surface and, accordingly, to an increase in the rate of hydrogen evolution upon interaction with water. Alloys of Wood, Rosé and Darcet are available commercial products. The melting temperature for Darcet and Rose alloys is 94℃, Wood's is 60-68.5oC, respectively. Activated aluminum alloys are made by melting and casting.

The replacement of In and Ga in the activated aluminum alloy by Wood's, Rose's, and Darcet's eutectic alloys made it possible to minimize production costs.

Methods and materials. Aluminum in granules GOST 295-98 was purchased from JSC "Kazakhstan Electrolysis Plant", the only aluminum producer in Kazakhstan, part of the ERG group of companies ("Eurasian Group").

Wood's alloy is a fusible alloy, (composition: tin-12.5%; cadmium-12.5%; lead-25%; Bismuth-50%), m_p= 60-68.5°C, ρ=9.720 g/cm³.

Rose's alloy is a fusible alloy, composition: (tin-34%; lead-20%; Bismuth-46.0%). t_{melt} = 94°C, ρ=7.6 g/cm³.

Darcet's alloy (composition: Sn-25%; Pb-25%; Bi-50%) Melting point 94 °C [Chemist's Handbook 21. https//Chem. 21.info, ru; wikipedia.org/wiki].

Hydrochloric acid of analytical grade, boiling point of azeotropic mixture (20.22% by mass) = 108.6 °C, ρ 1.16 (35%), was used without further purification.

Aluminum-based alloys for hydrogen production contain an additive that destroys the aluminum oxide film and increases the reactivity of the alloy in water (Patent of RK №34988; Patent of RK №34806; Patent of RK № 34807). According to (Patent of RK №34806; Patent of RK № 34807) activating additive (% wt.) - 5-15; aluminum - the rest.

The completeness of gas evolution is 98-100 wt.% when the alloy interacts with water or 1-5% aqueous hydrochloric acid solution in the temperature range of 50° - 90°C. Aluminum alloys with an activating additive are prepared in an alundum crucible, on the bottom of which an alloy (Darcet, Rose or Wood) is placed in an amount of 5-15% by weight, and aluminum is placed on top in a SNOL 1300 muffle furnace at a temperature of 850°C. As soon as the aluminum melts (temperature above 660°C), an intensive mixing process begins due to induction currents. The duration of the heating-holding-cooling cycle was 1 hour. The total duration of metal heating during heat treatment is the sum of the heating time to a given temperature and the holding time at this temperature. The material subjected to hardening acquires greater hardness and brittleness. The contents of the crucible were poured into a mold cooled by running water to prepare an ingot. A powder with specified particle sizes of 80-1250 μm was made from the ingot by changing the gap between the jaws of the crusher (mm): 1, 2, 5 and 10.

Aluminum alloys were characterized using an energy dispersive X-ray scanning electron microscope (SEM/EDX). The X-ray spectral analysis of the alloy was carried out on an X-Ray Innov-X systems X-ray fluorescence spectrometer, which makes it possible to determine the elemental composition and metal content in the alloy with an accuracy of 0.01% (Table 1).

Analysis of the elemental composition of the samples and photography in various types of radiation were performed using an OXFORD INSTRUMENTS INCA ENERGY energy) dispersive spectrometer mounted on a JEOL Superprobe 733 electron probe microanalyzer at an accelerating voltage of 25 kV and a probe current of 25 nA.

X-ray diffraction analysis of the reaction products of alloys formed in various oxidizing media was carried out on a DRON-3 automated diffractometer with Cu_{K} radiation, β-filter. Conditions for shooting diffraction patterns: U=35 kV; I=20 mA; shooting θ-2θ; detector 2 deg/min. X-ray phase analysis on a semi-quantitative basis was performed on the basis of powder samples diffraction patterns using the method of equal weights and artificial mixtures. Quantitative ratios of crystalline phases were determined. The interpretation of the diffraction patterns was carried out using the data from the ICDD file cabinet: PDF-2 powder diffraction data base and diffraction patterns of minerals free from impurities.

Powder X-ray 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.

Thermogravimetric analysis (TGA) was carried out using thermogravimetry (Q-1000/D derivatograph of the F.Paulik, J.Paulik and L.Erdey system of "MOM" corp., with a heating rate of 10°C/min in a nitrogen gas atmosphere of 30 ml/min).

The volume of hydrogen released during the interaction of aluminum alloys containing activating additives of Wood, Rose and Darcet alloys in various oxidizing media was measured on a drum gas meter. All experiments were repeated at least three times and were carried out 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. To bring the volume of gas that passed through the meter to normal conditions, the following formula was used:

$$V=Vt\ ((P+B)x(273+20))/(101325x(273+t)) \quad (1)$$

where, V is the volume of gas measured by the meter and reduced to normal conditions (temperature 20°C and pressure 101325 Pa), dm^3; t is the temperature of the measured gas, °C; V_t - volume of gas measured by the meter at temperature t and pressure P, dm^3; P is the pressure of the gas passing through the meter, Pa; B - atmospheric pressure, Pa.

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

$$W_H = \frac{V}{m\Delta t} \quad (2)$$

where V is the volume of released hydrogen, m is the weight of the alloy sample;
t is the time between two gas clock readings.

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

Results and discussion. X-ray spectral analysis of aluminum alloys obtained on the X-Ray Innov-X systems spectrometer shown in Table 1 showed that the main component of activated alloys is aluminum, the content of which ranges from 90% to 93.04%, the content of Sn, Cd, Pb and Bi correspond to the initial proportion of these metals in alloys.

Table 1 - X-ray spectral analysis of aluminum alloys activated by metal activators

Alloys	Ratio	Content of metals, %				
		Al	Sn	Cd	Pb	Bi
Al:Rose's alloy	90:10	93,04	1,80	-	1,90	3,26
Al:Wood's alloy	90:10	91,12	1,85	1,11	2,92	3,00
Al:Darcet's alloy	90:10	90,0	1.25	0.02	1.20	2.98
Analysis on X-Ray spectrometer Innov-X systems						

Figure 1 shows the surface of an aluminum alloy in secondary electrons activated by the Darcet alloy at a ratio of AI:Darcet alloy = 90:10.

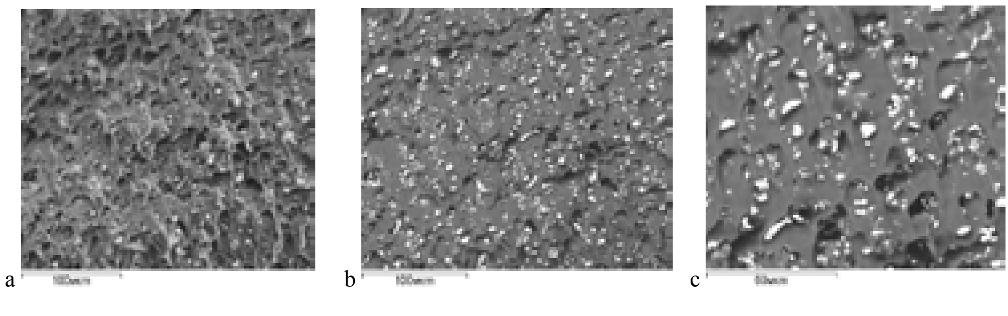


Figure 1. Image of the surface of an aluminum alloy in secondary electrons activated by the Darcet alloy at a ratio AI: Darcet alloy = 90:10 a) in secondary electrons; b) in back-scattered electrons; c) detail of the previous photo

The contrast in back-scattered electron images (composition or Compo) depends on the average atomic number of the mineral. The greater the concentration of heavy elements in a grain area, the lighter it looks.

The results of elemental analysis of the surface of samples of aluminum alloys activated by alloy eutectics by the EDXs method are shown in Figure 2-4 and Table 2.

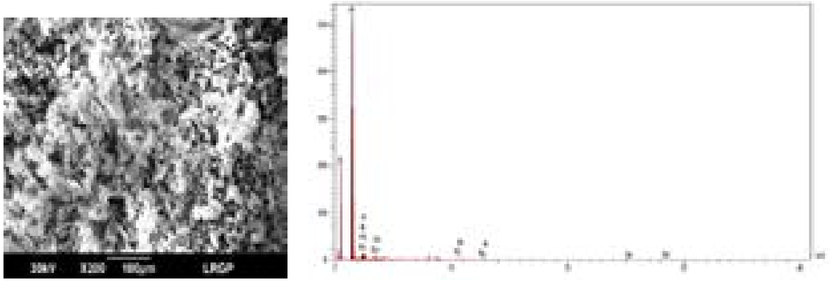


Figure 2. Microphotographs of the surface of an aluminum alloy activated with a Rose alloy at a ratio of AI: Rose alloy = 90:10 with a magnification: -x2000 and EDX spectra

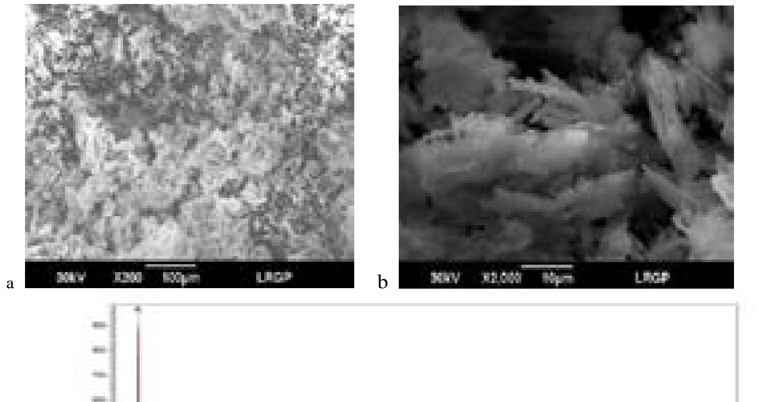
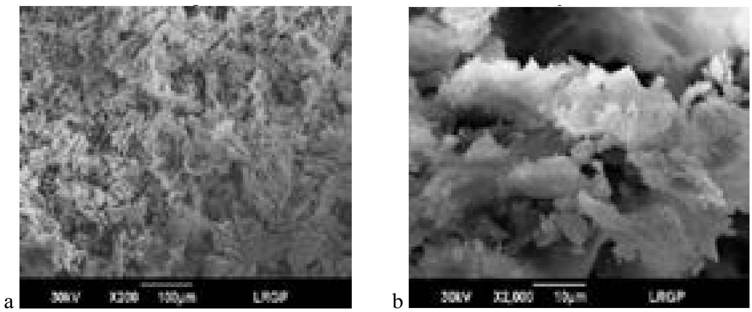


Figure 3. Micrographs of the surface of an aluminum alloy activated with Darcet alloy at a ratio AI: Darcet alloy = 90:10 with magnification: a - x200; b-x2000 and EDX surface spectra



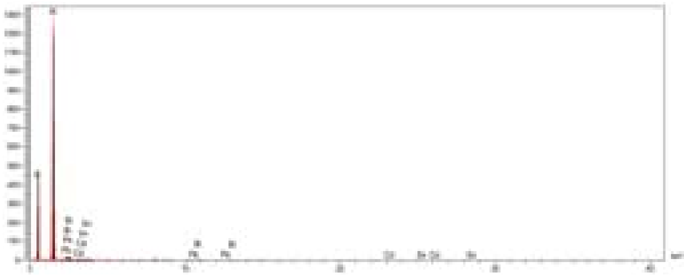


Figure 4. Micrographs of the surface of an aluminum alloy activated by Wood's alloy at a ratio of AI:Wood's alloy = 90:10 with magnification: a - x200; b-x2000 and EDX surface spectra

Table 2 - Results of elemental analysis of aluminum alloys by energy dispersive X-ray spectroscopy

Elements	W, %		
	Alloy Al : Rose	Alloy Al : Darcet	Alloy Al : Wood
Al	56,58	57,31	54,63
O	39,14	38,85	42,28
Bi	2,19	2,36	1,56
Pb	1,32	0,93	0,81
Sn	0,77	0,54	0,39
Cd	-	-	0,32

SEM - image of the surface of the alloy samples are shown in Figure 5-10. The studies 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 (Sarmurzina et. al., 1988; He et. al., 2016b).

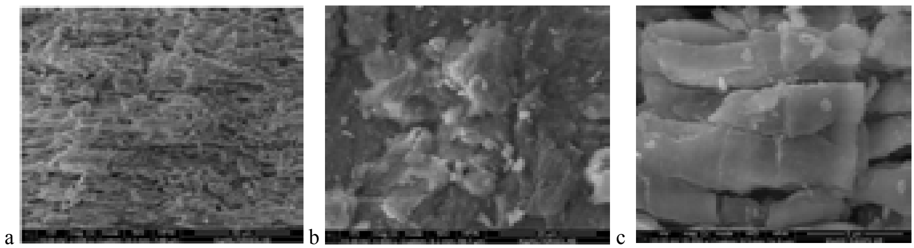


Figure 5. SEM image of the surface of an aluminum alloy activated with Rose alloy at the ratio AI: Rose alloy = 90:10 at magnification: a-x2000; b-x5000; c - x20 000

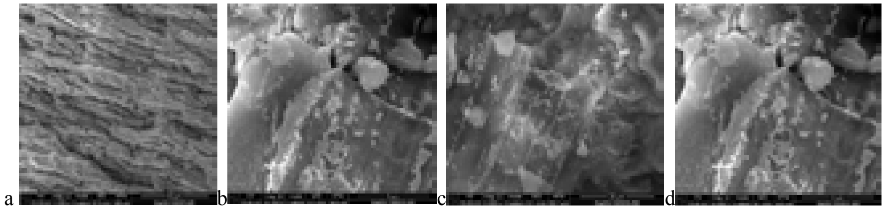


Figure 6. SEM image of the surface of an aluminum alloy activated by Wood's alloy at a ratio AI:Wood's alloy=90:10 at magnification: a-x2000; b-x5000; c - x5000; g- x20 000

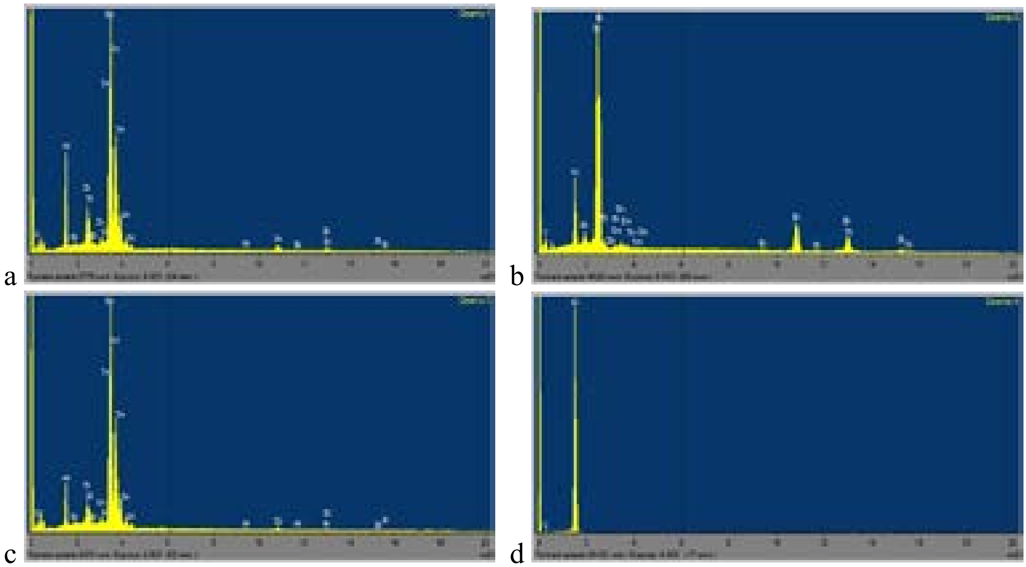


Figure 7. Spectra of a bright white grain of an aluminum alloy activated by the Darcet alloy (ratio 90:10): a) spectrum 1; b) spectrum 2; c) spectrum 3; d) spectrum 4.

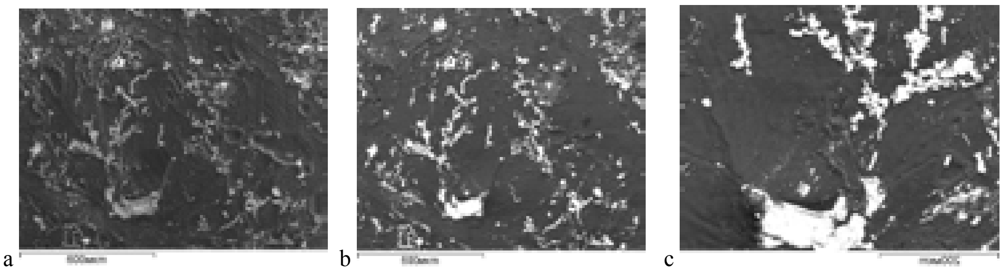


Figure 8. Image of the surface of an aluminum alloy activated with a Rose alloy at a ratio of AI: Rose alloy = 90:10 a) in secondary electrons; b) in back-scattered electrons; c) detail of the previous photo

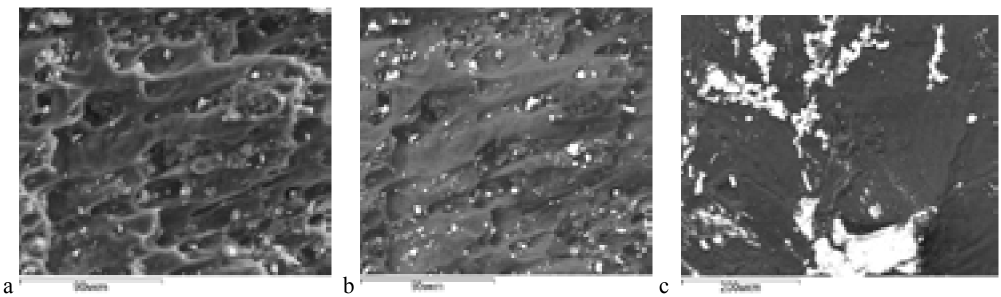


Figure 9. Image of the surface of an aluminum alloy activated by Wood’s alloy at a ratio of AI:Wood’s alloy=90:10 a) in secondary electrons; b) in back-scattered electrons; c) detail of the previous photo

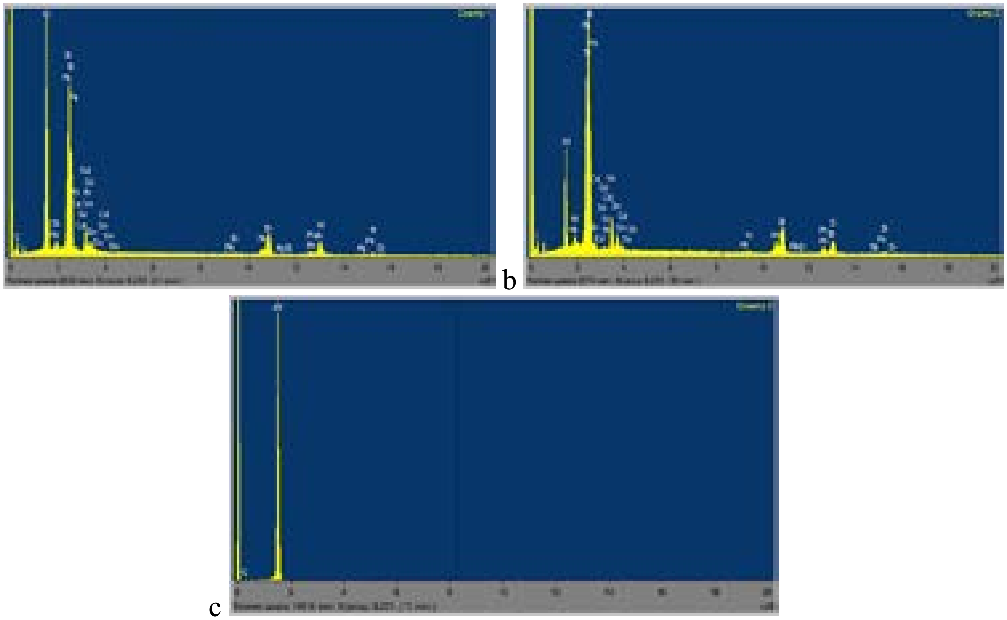

a b c

Figure 10. Spectra of a bright white grain of an aluminum alloy activated by Wood's alloy (ratio 90:10): a) spectrum 1; b) spectrum 2; c)- spectrum 3.

Table 3 - Results of X-ray elemental microanalysis of aluminum alloy grains activated with eutectics of Darcet, Rose and Wood alloys, the ratio of aluminum to the activating additive is 90:10.

Spectrum	Grain	Element content, mass. %					Total
		Al	Sn	Bi	Pb		
Aluminum Alloy Activated by Darcet Alloy							
Spectrum 1	bright white grain	12,06	75,47	12,47			100,00
Spectrum 2	bright white grain	9,10	4,06	86,85			100,00
Spectrum 3	bright white grain	7,41	84,32	8,27			100,00
Spectrum	Matrix gray in the photo	100					
Aluminum Alloy Activated by Rose Alloy							
Spectrum	Grain	Al	Sn	Bi	Pb		Total
Spectrum 1	bright white grain	0,69	10,69	43,31	45,30		100,00
Spectrum 2	bright white grain	1,25	10,897	46,79	41,08		100,00
Spectrum	Matrix gray in the photo	100					100,00
Aluminum Alloy Activated by Wood Alloy							
Spectrum	Grain	Al	Sn	Bi	Pb	Cd	Total
Spectrum 1	bright white grain	25,83	0,66	50,99	11,62	10,90	100,00
Spectrum 2	bright white grain	10,83	14,46	53,10	20,99	0,62	100,00
Spectrum	Matrix gray in the photo	100					100,00

Based on the data in Table 3, it can be concluded that bright white grains are heterogeneous in composition, the activating components Sn, Bi, Pb, Cd in the composition of the eutectic alloy participate in the formation of complex eutectics, have different distributions in the volume and on the aluminum surface.

X-ray phase analysis confirmed the eutectic nature of the alloys under study. However, an interesting feature inherent in eutectic alloys was discovered here: the chemical composition of the grain surface differs sharply from the chemical compositions in the bulk of the grain, which indicates the appearance of interphase nonequilibrium (Sarmurzina et. al., 1988).

XRD diffraction pattern of an aluminum alloy sample activated with Darcet alloy (ratio 90:10) is shown in Figure 11.

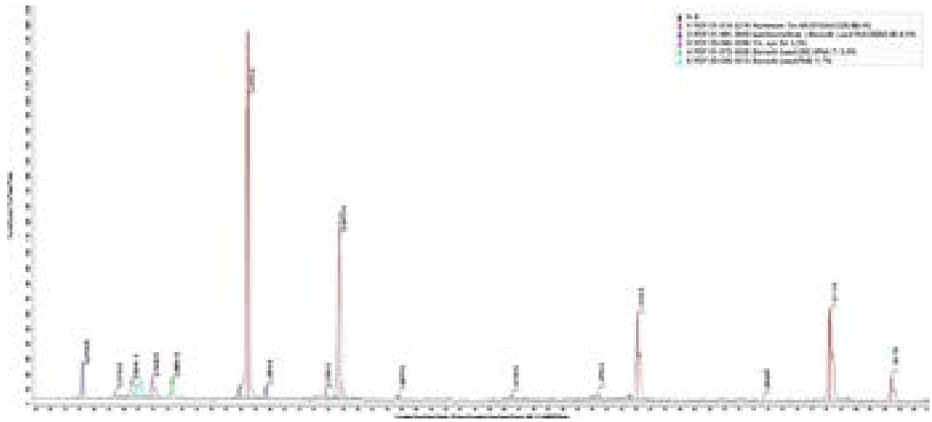


Figure 11. XRD diffraction pattern of an aluminum alloy sample activated with Darcet alloy (ratio 90:10)

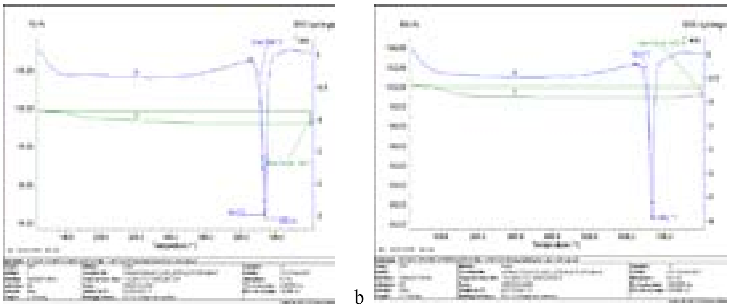
The phase composition according to the X-ray diffraction pattern of the AI alloy sample: Darcet alloy is shown in Table 4.

Table 4 - Phase composition of the aluminum alloy activated by the Darcet alloy at a ratio of 90:10

Pattern	Components	Chemical formula	Total Percentage, %
PDF 01-074-5276	Aluminum Tin	Al0.975Sn0.025	88,4
PDF 01-081-3929	lead bismuthide Bismuth	Pb0.05Bi0.95	4,3
PDF 03-065-0296	Tin, syn	Sn	3,2
PDF 01-072-5625	Bismuth Lead	Bi0.3Pb0.7	2,4
PDF 00-026-0215	Bismuth Lead	PbBi	1,7

According to XRD data (Table 4), the phase composition of the activated aluminum alloy includes phases of intermetallic compounds of various compositions: Aluminum Tin, Al0.975Sn0.025, lead bismuthide Bismuth Pb0.05Bi0.95, Bismuth Lead Bi0.3Pb0.7, Bismuth Lead PbBi and Tin, syn Sn.

To obtain information on the melting temperatures of activated aluminum alloys, the method of differential scanning calorimetry was used. The results of thermal analysis are shown in Figure 12. DSC and TG curves are relatively identical for aluminum alloys activated with Rose and Wood's alloys, respectively (Figures 25.26). An analysis of the DSC curves showed that at temperatures of 668 and 670°C, an endothermic thermal effect is observed, which corresponds to the melting temperature of the aluminum alloy.



a b

Figure 12. DSC / TG curves of aluminum activated by a) Rose alloy, b) Wood (ratio 90:10)

The reactivity of activated aluminum alloys with respect to distilled water in terms of hydrogen evolution was studied depending on the temperature, the amount of the activating additive in the alloy, and the pH of the water.

Tables 5 and 6 provide information on the volumes of hydrogen evolution under various conditions and the rates of hydrogen evolution.

Table 5 - The volume of hydrogen released during the interaction of water with aluminum activated by Wood's, Rose, Darcet alloys at various temperatures and the amount of activating additive

Aluminum alloy	Volume of released hydrogen, sm^3								
	50^0C			70^0C			90^0C		
	Amount of activa-ting additive,%			Amount of activating additive,%			Amount of activating additive,%		
	5	10	15	5	10	15	5	10	15
Aluminum Activated by Wood Alloy	98	82	85	153	128,3	165	394	280	359
Aluminum Activated by Darcet Alloy	47	118	80	79,5	600	121,7	133	690	393
Aluminum Activated by Rose Alloy	44	46,7	50	125,7	149,1	52,6	126,9	300	122,3

The amount of released hydrogen and the release rate increase with increasing temperature from 50 to 90°C and depend on the percentage of the alloy. It was found that the optimum temperature for the interaction of alloys with distilled water is 90°C, the content of the activating additive in the alloy is 10-15 wt%. For aluminum alloys activated by Darcet and Wood alloys, the optimal ratio is Al:alloy = 85:15.

When the ratio of components in the alloy Al: Wood's alloy=95:5, the volume of released hydrogen is 394 ml. The evolution of hydrogen upon interaction with water at the maximum rate occurs for the first time in 10 minutes of the reaction at all ratios of components in the alloy at 90°C and is 128-200 ml/g*min.

An increase in the reaction temperature makes it possible to reduce the amount of the activating additive.

For alloy Al: Rose alloy, the volume of released hydrogen at a ratio of 90:10 does not exceed 280-300 ml, aluminum conversion under these conditions reaches 25-28% depending on the composition of the activated alloy.

At a temperature of 90°C, the largest volume of released hydrogen is observed for an alloy of composition AI: Darcet alloy, at a ratio of 90:10, the volume of released hydrogen is 690 cm^3.

The maximum rate of hydrogen evolution of the alloy upon interaction with water at all ratios of components in the alloy is set at 90°C.

With an aluminum to alloy ratio of 85:15 for alloy Al: Wood's alloy is 200 ml/g*min, for alloy Al: Rose alloy -100 ml/g*min and 140 ml/g*min for alloy Al: Darcet alloy (table 6).

The average rate of H2 generation under these conditions is for alloy Al: Darcet alloy - 59 ml/g*min, Al: Woods alloy - 29 ml/g*min and Al: Rose alloy - 20 ml/g*min.

Table 6 - The maximum rate of hydrogen evolution during the interaction of aluminum activated by Wood's, Rose, Darcet alloys with distilled water

Aluminum alloy	Maximum gas evolution rate, ml/g·min								
	$90^{0}C$			$70^{0}C$			$50^{0}C$		
	Amount of activating additive,%			Amount of activating additive,%			Amount of activating additive,%		
	5	10	15	5	10	15	5	10	15
Aluminum Activated by Wood Alloy	128	160	200	90	116	80	18	24	20
Aluminum Activated by Darcet Alloy	70	120	100	66	114	20	5	10	10
Aluminum Activated by Rose Alloy	120	60	140	20	20	40	4	8	20

It was found that lowering the pH by adding HCI to distilled water increases the volumetric yield of hydrogen 5 times higher from 200 ml to 1100 ml and the average rate of hydrogen evolution from 20.5 to 100 ml/g*min (Fig.30-32).

The hydrogen yield increases with increasing HCl concentration and corresponds to the theoretically calculated value. The rates of hydrogen release for aluminum alloys activated by Wood and Rose alloys are almost equal, the conversion of aluminum reaches 100%, the volume of H_2 released during the reaction of the alloy with acidified water reaches the theoretically calculated value at a lower temperature (70°C).

Comparative kinetic curves of hydrogen evolution during the interaction of aluminum activated with the Rose alloy with distilled water and acidified water (1% HCI solution) at a temperature of 90℃, shown in Figure 13, indicate that the reactivity of the alloy increases significantly when it interacts with acidified distilled water, the hydrogen yield increases to the theoretically possible and amounts to 1180 ml over a period of time.

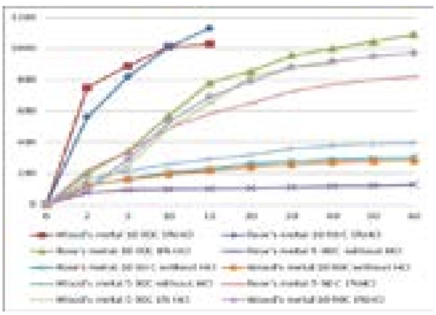


Figure 13. Comparative kinetic curves of hydrogen evolution during the interaction of aluminum alloys activated by Rose and Wood alloys with distilled water and aqueous HCl solutions at a temperature of $90^{o}C$

In a hydrochloric acid medium, electrolytic dissociation shifts towards the formation of hydrated ions, hydronium ion H_3O^+ and OH^- (irreversible stage), hydronium ion H_3O^+ is an active oxidizing agent.

The conversion of aluminum reaches 99 - 100% at a temperature of 90°C and the content of the activating additive in the alloy is 10%. The process ends within 10-15 minutes of the reaction, average speed reaches 100-102 ml/g*min, while without acid the average rate of hydrogen evolution is 20-59 ml/g*min.

When the pH of water is reduced by the addition of hydrochloric acid to values below zero, the volumetric yield of hydrogen increases and reaches 99% relative to the theoretical one, and the heat is 33712 kJ/kg.

The average rate of hydrogen evolution during the interaction of aluminum activated with Wood's alloy, Rose and Darcet, depending on temperature and HCl concentration for 10 minutes, is shown in Figures 14,15.

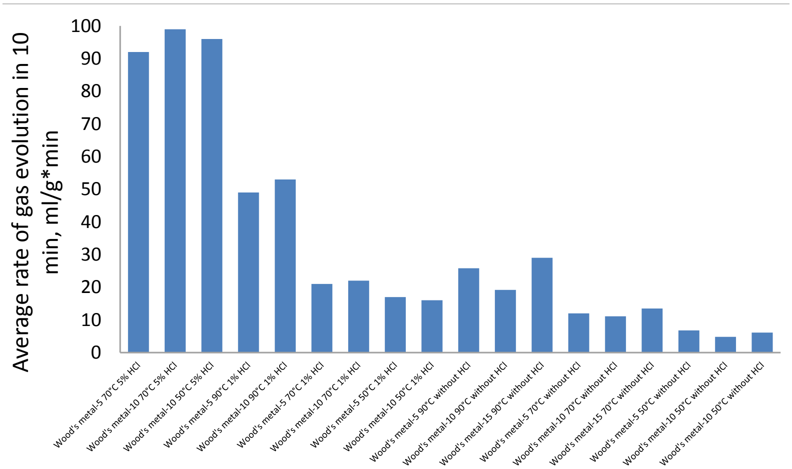


Figure 14. Average release rate of H2, during the interaction of the activated alloy Al: Wood's alloy depending on temperature and HCl concentration (within 10 minutes)

Thus, it has been shown that the higher the temperature and acidity of water, the greater the rate of hydrogen evolution over the same period of time. In a hydrochloric acid medium, the electrolytic dissociation of water shifts towards the formation of hydrated ions - hydroxonium ion H_3O^+ and OH^- (irreversible stage). Hydronium ion H_3O^+ is an active oxidant. The increase in speed is also due to an increase in electrical conductivity with an increase in the acidity of the medium. In terms of the rate of hydrogen evolution in acidified water, aluminum alloys containing Wood, Rose and Darcet alloys as an activating additive are not inferior to an alloy based on aluminum activated with indium, gallium and tin.

SEM with EDXs was used to study the microstructure and phase components of the resulting products during the interaction of alloys with water as a function of temperature. The complex of these methods provides sufficient and objective information about the composition of the studied objects.

The results of semi-quantitative XRF analysis of crystalline phases of the reaction products of aluminum activated by Wood, Rose and Darcet alloys with water in comparison with the Rau-85 reagent are shown in Table 7.

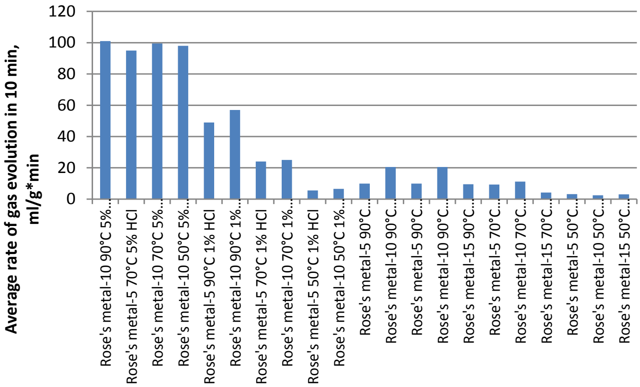


Figure 15. Average rate of hydrogen evolution during the interaction of activated Al alloy: Rose alloy depending on temperature and HCl concentration (within 10 minutes)

Table 7 - Results of X-ray phase analysis of the reaction products of aluminum alloys activated by Wood's, Rose and Darcet alloys with distilled water at 60°C (the alloy is crushed in a crusher)

Alloys	Pattern	Components	Chemical formula	Total percentage [%]
Rau 85	PDF 85-1327	Aluminum	Al	49,34
	PDF 01-077-2745	Indium Tin	(In3 Sn) 0.5	23,17
	PDF 01-073-9037	Indium Tin	In0.1818 Sn0.8182	18,34
	PDF 49-0133	Aluminum Oxide Hydroxide	Al O (O H)	9,15
Al:Rose's alloy 90:10	PDF 85-1327	Aluminum	Al	49,34
	PDF 01-072-5625	Bismuth Lead	Bi0.3 Pb0.7	11,30
	PDF 75-0993	Bismuth Oxide	Bi2 O2.7	2,94
	PDF 27-0054	Bismuth Oxide	Bi O	4,80
	PDF 44-1246	Bismuth	Bi	14,64
	PDF 53-0652	Lead Bismuth Oxide	Pb5 Bi8 O17	8,51
	PDF 49-0133	Aluminum Oxide Hydroxide	AlO(O H)	19,03
Al:Wood's alloy 90:10	PDF 85-1327	Aluminum	Al	36,78
	PDF 01-072-5625	Bismuth Lead	(Bi0.3 Pb0.7)	19,43
	PDF 44-1246	Bismuth	Bi	14,94
	PDF 49-0133	Aluminum Oxide Hydroxide	Al O (OH)	28,85
Al:Darcet's alloy 90:10	PDF 85-1327	Aluminum	Al	28,88
	PDF 01-0709	Bismuth Oxide	Bi2 O3	6,83
	PDF 75-0993	Bismuth Oxide	Bi2 O2.7	6,82
	PDF 82-1317	Bismuth Lead Oxide	Bi24 Pb2 O40	5,70
	PDF 00-059-0331	Bismuth Oxide	Bi2 O3	5,11
	PDF 01-085-1329	Bismuth, syn	Bi	13,45
	PDF 50-0870	Lead Bismuth Oxide	Pb7 Bi6 O16	9,34
	PDF 53-0652	Lead Bismuth Oxide	Pb5 Bi8 O17	8,95
	PDF 49-0133	Aluminum Oxide Hydroxide	AlO (O H)	14,92
Analysis on a powder diffractometer D8 Advance (Bruker)				

According to XRD data (Table 7), the phase composition of the reaction products of aluminum activated with Rose, Wood and Darcet alloys, their total percentage content differs significantly both in the number of phases and in their content.

The reaction products include intermetallic compounds of various compositions: Lead Bismuth Oxide Pb5 Bi8 O17 and Pb7Bi6O16, Bismuth Lead Oxide Bi24Pb2040 were found for aluminum alloys activated by Rose and Darcet alloys, respectively, for all three Bismuth Oxide alloys with the compositions Bi2 O2.7, Bi O, Bi2 O3 and Bismuth, syn Bi and Aluminum Oxide Hydroxide - AlO(O H).

The results indicate an incomplete reaction and are consistent with the kinetic data for hydrogen evolution given above.

The reaction products contain the original aluminum. The diffraction patterns of the samples contain a mixture of crystalline and amorphous phases.

Conclusion. New economically expedient alloys have been developed that exclude the use of non-ferrous dispersed metals and contain eutectic alloys of Wood, Rose and Darcet as activator metals.

The parameters of interaction of aluminum alloys activated with Darcet, Wood, Rose alloys with water are optimized depending on the temperature, the amount of the activating additive in the alloy, and the pH of the water. The completeness of gas evolution (gas production for the developed alloys at the optimal ratio of alloy: activating additive 90:10 was 98-99%. Hydrogen evolution during the interaction of the alloy with water occurs at a maximum rate at 90^{o}C. The amount of hydrogen released and the rate of evolution increase with increasing temperature from 50 to 90^{o}C and depend on the percentage of the activating additive in the alloy. Hydrogen evolution during the interaction of the alloy with water at a maximum rate occurs at 90°C.

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