

**ВЫСОКОТЕМПЕРАТУРНАЯ И ЭЛЕКТРОХИМИЧЕСКАЯ КОРРОЗИЯ СПЛАВА Zn0.5Al,
ЛЕГИРОВАННОГО КАЛЬЦИЕМ, В РАЗЛИЧНЫХ СРЕДАХ****З.Р. Обидов, Р. Амини, О.Н. Назаров, Дж.Х. Джайлоев, И.Н. Ганиев, Р. Усманов**

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В статье приведены результаты исследования высокотемпературной и электрохимической коррозии сплава Zn0.5Al, легированного кальцием, в различных средах. Термогравиметрическим методом исследовано взаимодействие сплава Zn0.5Al, легированного кальцием, с кислородом воздуха в интервале температур 523–623 К, в твердом состоянии. Определены кинетические и энергетические параметры процесса высокотемпературного окисления сплавов. Процесс высокотемпературного окисления сплавов системы Zn-Al-Ca характеризуется монотонным снижением истинной скорости окисления и повышением эффективной энергии активации при содержании легирующего компонента в исходном сплаве Zn0.5Al до 1,0 мас.%. При легировании цинк-алюминиевого сплава 0,5 и 1,0 мас.% кальцием показано незначительное увеличение скорости окисления сплавов. Выявлено, что процесс окисления исследованных сплавов кислородом газовой фазы подчиняется гиперболическому закону. Установлено, что добавки кальция в пределах 0,01-0,1 мас.% значительно уменьшают окисляемость исходного сплава Zn0.5Al, а продуктами окисления сплавов являются оксиды ZnO, Al₂O₃, Al₂O₃ · ZnO, CaO, Al₂O₃ · CaO. Потенциостатическим методом в потенциодинамическом режиме со скоростью развертки потенциала – 2 мВ/с показано, что для всех образцов сплавов системы Zn0.5Al-Ca в кислых, нейтральных и щелочных средах наблюдается смещение электрохимических потенциалов коррозии, питтингообразования и репассивации в область отрицательных значений. Выявлено, что цинк-алюминиевые сплавы, легированные кальцием, наиболее устойчивы к питтинговой коррозии во всех исследованных средах, соответственно в кислой (0,01н), нейтральной (0,03-, 0,3-, 3,0 мас.%) и щелочной (0,01н) среде электролитов HCl, NaCl и NaOH. Установлено, что добавки кальция в пределах 0,01-0,1 мас.% уменьшают скорость коррозии цинк-алюминиевого сплава Zn0.5Al в 2–3 раза. Сплавы с кальцием рекомендуются в качестве анодных покрытий и протекторов для защиты от коррозии стальных изделий и конструкций, работающих при высоких температурах.

Ключевые слова: сплав Zn0.5Al с кальцием, термогравиметрическое и потенциодинамическое исследование, скорость окисления, энергия активации, скорость коррозии, анодное поведение сплавов

**HIGH TEMPERATURE AND ELECTROCHEMICAL CORROSION OF Zn0.5Al
ALLOY DOPED WITH CALCIUM IN VARIOUS MEDIA****Z.R. Obidov, R. Amini, O.N. Nazarov, J.Kh. Dzhayloev, I.N. Ganiev, R. Usmanov**

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The article presents the results of a study of high-temperature and electrochemical corrosion of Zn0.5Al alloy doped with calcium in the various media. The thermogravimetric method was used to study the interaction of the Zn0.5Al alloy doped with calcium with atmospheric oxygen in the temperature range 523–623 K in the solid state. The kinetic and energy parameters of the process of high-temperature oxidation of alloys are determined. The process of high-temperature oxidation of Zn-Al-Ca alloys system is characterized by a monotonic decrease in the true oxidation rate and an increase in the effective activation energy when the alloying component in the initial Zn0.5Al alloy is up to 1.0 wt% doping with zinc-aluminum alloy 0.5 and 1.0 wt%. Calcium shows a slight increase in the oxidation rate of alloys. It was revealed that the oxidation process of the studied alloys with oxygen of the gas phase obeys the hyperbolic law. It was found that calcium supplements in the range of 0.01 - 0.1 wt%. The oxidizability of the initial Zn0.5Al alloy is reduced significantly, and the oxidation products of the alloys were ZnO, Al₂O₃, Al₂O₃ · ZnO, CaO, Al₂O₃ · CaO. By potentiostatic methods in the potentiodynamic mode with a potential sweep speed of 2 mV/s, it has been shown that for all samples of the Zn0.5Al-Ca alloys system in the acidic, neutral, and alkaline media, electrochemical potentials of corrosion, pitting formation, and re-passivation are shifted to the region of negative values. It was revealed that zinc-aluminum alloys doped with calcium are most resistant to pitting corrosion in all studied media, respectively, in acidic (0.01n), neutral (0.03-, 0.3-, 3.0 wt%) and alkaline (0.01n) electrolytes of HCl, NaCl and NaOH. It has been established that calcium additions in the range of 0.01 - 0.1 wt.% reduce the corrosion rate of zinc-aluminum alloy Zn0.5Al by a factor of 2–3. Calcium alloys are recommended as anodic coatings and protectors for corrosion protection of steel products and structures operating at high temperatures.

Key words: Zn0.5Al alloy, calcium, thermogravimetric and potentiodynamic studies, oxidation rate, activation energy, corrosion rate, anodic behavior

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INTRODUCTION

The issues of the interaction of metal alloys with gaseous and various aggressive media at high temperatures are the key in modern materials science. Zinc and aluminum based alloys are widely used in various fields of technology [1-7]. Recently, these alloys began to be used as protective coatings for steel products and structures. The most famous of them are Zn5Al and Zn55Al alloys known under the trademarks Galfan-I, II and Galvalum. Coatings are applied for the anodic protection of steel; the decisive factor is the compromise between the low polarization of the coating in the area of damage (which determines the protection of steel) and its corrosion resistance far from

this zone [8, 9]. It is also known that the introduction of a small amount of molybdenum into the composition of the zinc coating during electrolysis makes it possible to obtain Zn-Mo coatings with alloys that have a higher protective ability than zinc coatings. The effectiveness of their use in atmospheric conditions of increased rigidity (marine environments, coastal zones, tropics and other environmental factors) is investigated [10]. There are various modifications of zinc-aluminum alloys alloyed with the third component. In particular, a positive effect of a number of metals of the periodic system on the corrosion resistance of zinc – aluminum alloys was studied in literatures [11-18]. The purpose of this work is to study the high-temperature electrochemical corrosion of the Zn0.5Al alloy,

doped with calcium, designed as anodic protective coatings, and protectors to increase the corrosion resistance of steel structures and products.

MATERIALS AND METHODS

Investigation of the effect of temperature and chemical composition on the kinetics of high-temperature oxidation of Zn0.5Al alloy doped with calcium, in the solid state, was carried out by thermogravimetry method with continuous weighing of samples in the air according to the procedure described in [19-22]. Samples of the alloy for the study were obtained from zinc grade KH.TC (granular), aluminum A7 grade and its alloys with calcium metal (10% Ca) in alumina crucibles in a shaft furnace of electric resistance of the C.SHOL type in the temperature range 650 ... 750 °C. The elemental composition of the studied alloys was monitored by X-ray microanalysis on a SEM instrument of the AIS 2100 series. A potentiodynamic study of the corrosion-electrochemical behavior of Zn0.5Al alloy doped with calcium was carried out in acidic (0.01n), neutral (0.03, 0.3, 3.0 wt%) and alkaline environment (0.01n), HCl, NaCl and NaOH electrolytes. The sweep speed of the electrode potential on the PI-50.1.1 potentiostat was 2 mV/s. The silver chloride (HSE) served as the reference electrode, and platinum served as the auxiliary electrode. The technique of electrochemical study of alloys is described in the reference [23].

RESULTS AND DISCUSSION

Thermogravimetric studies of the oxidation kinetics of Zn0.5Al-Ca alloys were carried out at temperatures of 523, 573 and 623 K. The interaction of the Zn0.5Al alloy with various concentrations of calcium with oxygen in the gas phase at the temperatures is significantly different from the oxidation of the initial Zn0.5Al alloy. The linear dependence is maintained for 12-15 min, further, as the oxide film is formed, the nature of the oxidation process becomes hyperbolic and the formation of the protective oxide surface ends by 30 minutes. Doping of the Zn0.5Al alloy with calcium (in the range of 0.01-0.1 wt%) contributes to a certain decrease in the true oxidation rate (Fig. 1, 2). A significant effect on the oxidizability of alloys is their chemical composition. Among the alloys, the Zn0.5Al alloy with 0.01 wt% calcium has a minimum oxidation rate and maximum activation energy of 186.1 kJ/mol (Table 1).

Doping calcium from 0.5 to 1.0 wt% to the alloy is impractical, since it leads to an increase in the oxidation rate and, accordingly, decreases the activation energy of oxidation of alloys. The effective activation energy of the oxidation process of these alloys varies from 168.4 to 174.5 and 174.9 kJ/mol (Table 1).

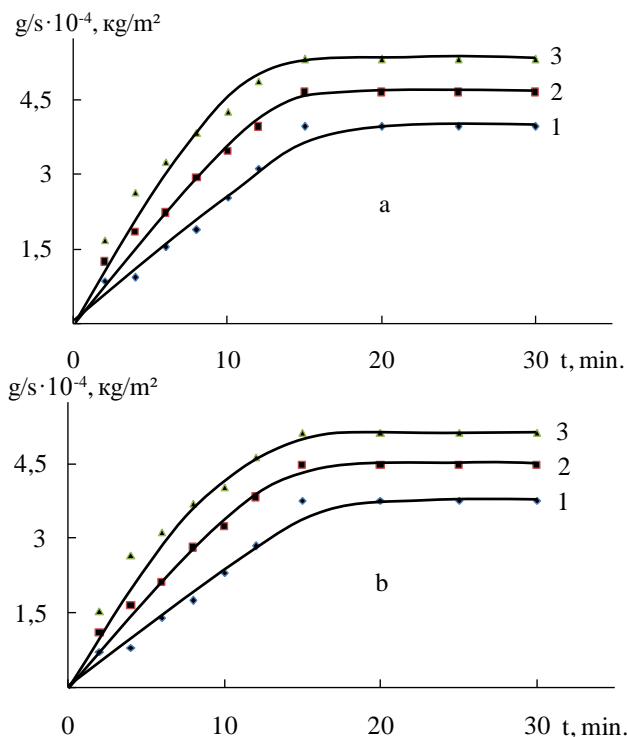


Fig. 1. Kinetic curves of the oxidation process of the Zn0.5Al (a) alloy doped with 0.01 wt% (b) calcium at T = 523 (1), 573 (2) and 623 (3) K

Рис. 1. Кинетические кривые процесса окисления сплава Zn0.5Al (а), легированного 0,01 мас.% (б) кальцием при T = 523 (1), 573 (2) и 623 (3) K

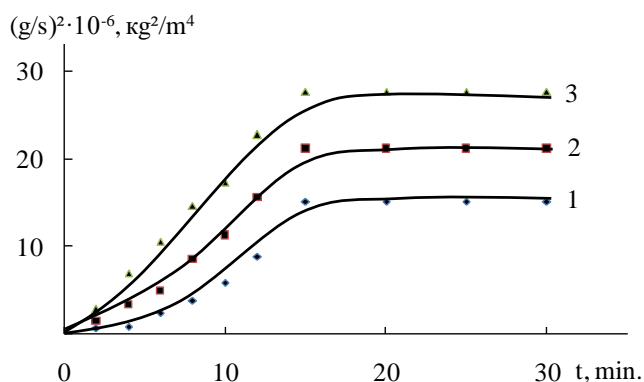


Fig. 2. Square-law curves of process of high-temperature oxidation of the Zn0.5Al alloy containing 1.0 wt% calcium at T = 523 (1), 573 (2) and 623 (3) K

Рис. 2. Квадратичные кривые процесса высокотемпературного окисления сплава Zn0.5Al, содержащего 1,0 мас.% кальция при T = 523 (1), 573 (2) и 623 (3) K

The high-temperature oxidation of the studied alloys by the oxygen gas phase obeys the hyperbolic law, as it can be observed from the dependence curves $(g/s)^2 - t$ (Fig. 2), it does not fit by straight lines, as well as by the analytical dependences $y = Kt^n$, where $n = 2-4$ (Table 2). Studying the products of oxidation of alloys, in particular, an oxide film that forms when heated on the surface of samples, important information can be obtained about their oxidation mechanism. Oxidation

products resulting from the oxidation of the Zn0.5Al alloy doped with calcium, investigated by X-ray phase analysis [24, 25]. It is observed that the oxidation products of the Zn0.5Al alloy (Fig. 3a) and the alloy doped with 0.1 wt% calcium (Fig. 3b) consist of ZnO, Al₂O₃, Al₂O₃·ZnO, CaO, Al₂O₃·CaO oxides (Fig. 3).

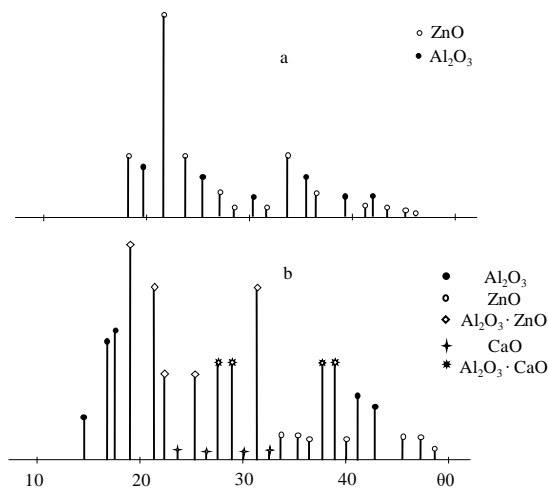


Fig. 3. Bar graphs of oxidation products of the Zn0.5Al alloy (a) containing 0.1 wt% (b) calcium

Рис. 3. Штрихдифрактограммы продуктов окисления сплава Zn0.5Al (a), содержащего 0,1 мас.% (б) кальций

Table 1
Kinetic and energy parameters of the oxidation process of the Zn0.5Al alloy doped with calcium at solid state
Таблица 1. Кинетические и энергетические параметры процесса окисления сплава Zn0.5Al, легированного кальцием, в твердом состоянии

Content Ca in the alloy, wt%	Temperature of oxidation, K	The true oxidation rate $K \cdot 10^{-4}$, $\text{kg/m}^2 \cdot \text{s}$	Effective activation energy, kJ/mol
0.0	523	3.68	168.4
	573	3.91	
	623	4.11	
0.01	523	2.21	186.1
	573	2.50	
	623	2.65	
0.05	523	2.30	184.0
	573	2.61	
	623	2.76	
0.1	523	2.51	180.4
	573	2.80	
	623	2.96	
0.5	523	3.18	174.9
	573	3.32	
	623	3.63	
1.0	523	3.33	171.5
	573	3.47	
	623	3.78	

Table 2

The results of mathematical processing of the curves of the oxidation process of the Zn0.5Al alloy doped with calcium in the solid state

Таблица 2. Результаты математической обработки кривых процесса окисления сплава Zn0.5Al, легированного кальцием, в твёрдом состоянии

Calcium content in alloy, wt%	Oxidation temperature, K	Polynomials alloy oxidation curve	Approximation confidence level R^2 , %
0.0	523	$y = -0.000t^4 - 0.000t^3 + 0.010t^2 - 0.176t$	0.987
	573	$y = -0.000t^4 - 0.001t^3 + 0.020t^2 - 0.471t$	0.985
	623	$y = -0.000t^4 - 0.001t^3 + 0.044t^2 - 0.786t$	0.981
1.0	523	$y = -0.001t^4 - 0.012t^3 + 0.241t^2 - 0.249t$	0.994
	573	$y = -0.001t^4 - 0.016t^3 + 0.281t^2 - 0.697t$	0.991
	623	$y = -0.001t^4 - 0.019t^3 + 0.310t^2 - 0.905t$	0.988

The assessment of the pitting corrosion resistance of the Zn0.5Al alloy with calcium can be carried out by comparing the values of the stationary potentials of free corrosion and pitting formation under the same research conditions. Additives of the alloying component (0.01-1.0 wt%) contribute to the displacement of the electrochemical potentials of the Zn0.5Al alloy in the region of negative values (Table 3).

In general, studies indicate an improvement in the corrosion resistance of the Zn0.5Al alloy when doped with calcium. The results show the ability of alloys to self-heal pitting damage resulting from corrosion. Calcium supplements within a concentration of 0.01-0.1 wt% reduce the corrosion rate of the Zn0.5Al alloy by a factor of about 2-3 (Table 3). It should be

noted that the dynamics of changes in the corrosion-electrochemical potentials favorably affect the changes in the corrosion resistance of alloys generally.

CONCLUSION

Thus, according to experimental studies of the kinetics of high-temperature oxidation of the Zn0.5Al alloy with calcium additions it was found that alloys with 0.5 and 1.0 wt% calcium compared with low-alloy alloys (0.01-0.1 wt% Ca) has the highest value of the true oxidation rate and the smallest value of the effective activation energy. It was revealed that the alloying component significantly reduces the oxidizability of the Zn0.5Al alloy in the range of 0.01-0.1 wt% calcium. It was determined that the oxidation products

of the studied alloys consist of ZnO, Al₂O₃, Al₂O₃·ZnO, CaO, Al₂O₃·CaO oxides.

Studies of the corrosion-electrochemical behavior of the Zn0.5Al alloy with calcium in acidic (electrolyte-HCl), neutral (electrolyte-NaCl) and alkaline (electrolyte-NaOH) media showed the possibility

of increasing the corrosion resistance of the anodic protective coatings by optimizing the composition (corrosion rate is 2–3 times lower than that of the initial Zn0.5Al alloy). Alloys with calcium can be used as protective coatings and protectors for corrosion protection of steel structures and products operating at high temperatures.

Table 3

Electrochemical corrosion characteristics (HSE) of the Zn0.5Al alloy doped with calcium in acidic, neutral and alkaline media

Таблица 3. Коррозионно-электрохимические характеристики (х.с.э.) сплава Zn0.5Al, легированного кальцием, в кислых, нейтральных и щелочных средах

Environment	Content of Ca in the alloy, wt%	Electrochemical potentials, V (HSE)				Corrosion rate	
		-E _{ir.corr.}	-E _{corr.}	-E _{p.f.}	-E _{rep.}	$i_{corr.} \cdot 10^{-2}$ A/m ²	$K \cdot 10^{-3}$ g/m ² ·h
0.01n HCl	0.0	1.110	1.118	0.980	0.995	0.154	1.87
	0.01	1.183	1.165	1.036	1.046	0.050	0.61
	0.05	1.198	1.187	1.042	1.051	0.052	0.63
	0.1	1.221	1.225	1.053	1.058	0.055	0.67
	0.5	1.244	1.256	1.064	1.082	0.061	0.74
	1.0	1.267	1.277	1.080	1.090	0.062	0.75
0.03% NaCl	0.0	0.960	0.968	0.745	0.809	0.037	0.45
	0.01	0.995	1.001	0.782	0.804	0.015	0.18
	0.05	1.011	1.020	0.810	0.822	0.016	0.20
	0.1	1.029	1.034	1.020	1.028	0.017	0.21
	0.5	1.042	1.047	0.832	0.842	0.033	0.40
	1.0	1.048	1.056	0.848	0.853	0.035	0.43
3.00% NaCl	0.0	1.070	1.086	0.779	0.804	0.055	0.67
	0.01	1.153	1.166	0.863	0.869	0.022	0.26
	0.05	1.160	1.170	0.872	0.885	0.025	0.30
	0.1	1.178	1.185	0.885	0.896	0.029	0.35
	0.5	1.194	1.198	0.904	0.915	0.052	0.63
	1.0	1.192	1.202	0.918	0.923	0.056	0.67
0.01n NaOH	0.0	1.048	1.058	0.892	0.900	0.127	1.55
	0.01	1.094	1.096	0.928	0.933	0.043	0.52
	0.05	1.119	1.131	0.948	0.963	0.045	0.55
	0.1	1.133	1.159	0.989	0.998	0.049	0.60
	0.5	1.160	1.173	1.067	1.083	0.054	0.66
	1.0	1.191	1.195	1.093	1.099	0.058	0.71

Conflict of interests

The authors declare no conflict of interests regarding the publication of this article.

The final manuscript has been read and approved by all the co-authors.

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