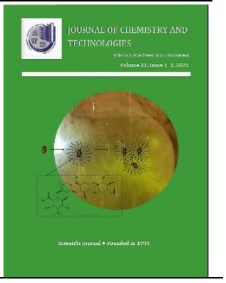
Journal of Chemistry and Technologies, 2025, *33*(2), 384-390

Journal of Chemistry and Technologies

pISSN 2663-2934 (Print), ISSN 2663-2942 (Online).

journal homepage: http://chemistry.dnu.dp.ua
editorial e-mail: chem.dnu@gmail.com



UDC 620.193:669.018.25

THE CORROSION BEHAVIOR OF Al_5SiFe INTERMETALLIC PHASE

Oleksandr A. Mityayev, Oleksii Yu. Voskoboinik*, Valentyna M. Povzlo, Vira O. Savchenko, Oleksii Ye. Kapustian
National university «Zaporizhzhia polytechnic», Universytetska st. 64, Zaporizhzhia, 69011, Ukraine

Received 9 February 2025; accepted 28 April 2025; available online 15 July 2025

Abstract

The present paper is devoted to the study of corrosion behavior of Al_5SiFe intermetallic phase that frequently present in secondary aluminium alloys. It has been shown that in acidic chloride-containing solutions with pH 0.5–1.5 and 1.5–2.5, the mean weight losses, average corrosion rate, and corrosion depth index were at their maximum and temperature of the corrosive medium has a negligible effect on the corrosion rate. Increasing of corrosion medium`s pH up to neutral values resulted the decreasing of corrosion indicators. The lowest corrosion losses were detected in neutral sodium chloride solutions due to oxygen depolarization in cathodic processes and the presence of a protective film formed by corrosion products. This was confirmed by the presence of a passive region on the anodic potentiodynamic curve obtained as a result of the electrochemical study of the Al_5SiFe intermetallic phase in a 3 % sodium chloride solution. Further increasing of the pH of solutions significantly accelerated the corrosion rate. This acceleration is apparently associated with the local destruction of the protective film by hydroxyl anions.

Keywords: corrosion; intermetallic phase; aluminium; electrochemical study.

КОРОЗІЙНА ПОВЕДІНКА ІНТЕРМЕТАЛІЧНОЇ ФАЗИ Al_5SiFe

Олександр А. Мітяєв, Олексій Ю. Воскобойнік, Валентина М. Повзло, Віра О. Савченко, Олексій Є. Капустян
Національний університет «Запорізька політехніка», вул. Унівеситетська, 64, м. Запоріжжя, 69011, Україна

Анотація

Представлена робота присвячена дослідженню корозійної поведінки інтерметалічної фази Al_5SiFe, яка часто є компонентом вторинних алюмінієвих сплавів. Показано, що в кислих хлорид-вмісних середовищах з pH 0.5–1.5 та 1.5–2.5 середні втрати маси, середня швидкість корозії та індекс глибини корозії мають максимальні значення, а температура корозійного середовища має незначний вплив на швидкість корозії. Збільшення pH корозійного середовища до нейтральних значень призводило до зниження корозійних показників. Найменші корозійні втрати спостерігалися в нейтральних розчинах хлориду натрію, що пояснюється деполяризацією киснем у катодних процесах і наявністю захисної плівки, утвореної продуктами корозії. Це підтверджується наявністю пасивної області на анодній потенціодинамічній кривій, отриманій в результаті електрохімічного дослідження інтерметалічної фази Al_5SiFe у 3 % розчині хлориду натрію. Подальше підвищення pH розчинів значно прискорювало корозію, що, ймовірно, пов'язано з локальним руйнуванням захисної плівки гідроксид-аніонами.

Ключові слова: корозія; інтерметалідна фаза; алюміній; електрохімічні дослідження.

**Corresponding author: e-mail: a.yu.voskoboynik@gmail.com*

doi: 10.15421/jchemtech.v33i2.322589

Introduction

Currently, the production of aluminium is focused on the recycling of secondary raw materials due to its economic and ecological feasibility. According to published forecasts, the implementation of an effective aluminium recycling strategy will generate 6 billion euros per year for the European economy [1]. Additionally, the recycling of post-consumer aluminium significantly impacts the ecology of countries that produce aluminium alloys by reducing harmful waste emissions [2]. Many countries, including Ukraine, do not have facilities for the production of primary aluminium alloys, therefore, their needs can be met through the development and implementation of recycling technologies.

It should be noted that the low quality of secondary aluminium alloys substantially limits their application, particularly in the production of special-purpose items. The structures of secondary aluminium alloys are often characterized by significant heterogeneity caused by the formation of secondary phases in solid solutions, including intermetallic compounds, oxides, sulfides, etc. [3]. These inclusions, by analogy with corrosion-resistant steels [4; 5], could serve as cores for the initiation and development of pitting and crevice corrosion in equipment and components made of aluminium alloys under operating conditions [6; 7].

Secondary aluminium alloys often contain iron, which forms complex intermetallic phases such as β Al_5SiFe, Al_7Cu_2Fe, and π-$Al_8Si_6Mg_3Fe$, among others [8]. In particular, secondary silumins contain the β Al_5SiFe intermetallic phase, which is the core of corrosion processes in corrosive environments of varying activity [9]. Despite the critical role of Al_5SiFe in the corrosion development of secondary silumins, its own corrosive properties have not been studied. Therefore, the present manuscript is devoted to studying the corrosion behavior of the Al_5SiFe intermetallic phase in solutions with a wide range of pH values that serve as models of working environments.

Experimental part

The synthetic intermetallic phase Al_5SiFe was obtained *via* a metallurgical method in a vacuum induction furnace (OKB – 862) [10; 11]. The initial simple substances were fused at 800 °C for 1 hour under a vacuum of 7×10^{-4} Pa to prevent the formation of oxidation products. Intensive stirring of the reaction mixture was required to obtain a homogeneous phase and to avoid liquation. The intermetallic phase was cast into samples at a temperature of 720 °C. An automatic control system ensured the stability of the set temperature (±5 °C) throughout the melting, holding, and casting stages. The composition of the intermetallic phase was evaluated using a REM-1061 electron microscope equipped with an energy-dispersive X-ray spectroscopy (EDS) system. The relative error of the measurement was 0.1 wt%. The analysis was performed at six points in different areas of the sample. The obtained data confirmed both the proposed composition and the homogeneity of the studied sample. The samples used for the study had a cylindrical form, with a length of 25 mm and a diameter of 5 mm. The shape of the samples was chosen due to the features of technological processing and the convenience of contact surface calculation.

The corrosion behavior of the Al_5SiFe intermetallic phase was studied across a pH range from 0.5 to 13.5. The corresponding solutions were prepared to imitate three types of corrosive media. Aqueous solutions of hydrochloric acid were used as an acidic corrosive medium (pH 0.5–5), aqueous solutions of sodium chloride were used as a neutral medium (pH 6–8), and solutions of sodium chloride and sodium hydroxide were used as an alkaline medium (pH 8-13). The pH values of the solutions were measured at +20±1 °C using a pH-meter-millivoltmeter pH-301. Three measurements were conducted for each pH value. The explosure time was 0.1667 h. Sample weights were measured using laboratory scales PS510.R2, which meet the requirements of DSTU EN 45501:2017. The measurement accuracy was ± 0.0001 g. The explosure of studied samples to a corrosive medium was conducted in jacketed vessel jointed with thermostatic water bath. The temperature of corrosive medium was additionally controlled with mercury thermometer. Corrosion weight losses (Δm), mean corrosion rate (CR), and corrosion depth index (CDI) were evaluated according to DSTU 9.912-89.

Corrosion indicators for the Al_5SiFe intermetallic phase were estimated based on the samples' weight loss, following DSTU 3830-98. In particular, the mean corrosion rate (CR) was calculated using the following formula:

$$SR = (\Delta m/(S\cdot t))\cdot 10^{-4},$$

where in: Δm – total corrosion weigh loss, g.; S – total area of parallel samples`surface, cm^2; t – duration of the study, hours

Table 1

The study results of the corrosion resistance of the Al_5SiFe intermetallic phase

Marking of samples, №	Mean loss of the mass, (Δm), g/m^2	pH and temperature of solution	Mean corrosion rate, (Δm), $g/(m^2*h)$	Corrosion depth index (CDI), mm/year	Group of resistance (DSTU 13819-68)	Points	Characteristic areas
1, 2, 3	0.3520	0.5-1.5, 20°C	2.0708	5.26	not resistive	9	I – area of active dissolution in acidic medium
25, 26, 27	0.3672	0.5-1.5, 40°C	2.1601	5.49	not resistive	9	
4, 5, 6	0.3246	1.5-2.5, 20°C	1.9094	4.85	not resistive	9	
28, 29, 30	0.3452	1.5-2.5, 40°C	2.0305	5.16	not resistive	9	
7, 8, 9	0.0528	3.5-4.5, 20°C	0.3110	0.79	low resistive	7	II – area of passive state
31, 32, 33	0.1692	3.5-4.5, 40°C	0.9953	2.53	not resistive	8	
10, 11, 12	0.0055	5.5-6.5, 20°C	0.0322	0.08	low resistive	6	
34, 35, 36	0.0202	5.5-6.5, 40°C	0.1189	0.30	low resistive	6	
13, 14, 15	0.0016	6.5-7.5, 20°C	0.0094	0.02	resistive	4	
37, 38, 39	0.0067	6.5-7.5, 40°C	0.0392	0.10	resistive	5	
16, 17, 18	0.0043	8.5-9.5, 20°C	0.0252	0.06	resistive	5	III – area of dissolution in basic medium
40, 41, 42	0.0119	8.5-9.5, 40°C	0.0701	0.17	low resistive	6	
19, 20, 21	0.0281	10.5-11.5, 20°C	0.1657	0.42	low resistive	6	
43, 44, 45	0.0538	10.5-11.5, 40°C	0.3165	0.80	low resistive	7	
22, 23, 24	0.1593	12.5-13.5, 20°C	0.9370	2.38	not resistive	8	
46, 47, 48	0.3166	12.5-13.5, 40°C	1.8622	4.73	not resistive	9	

Electrochemical studies (potentiodynamic polarization test).

Disk-shaped samples of Al_5SiFe were connected to a copper, silicone-coated wire, placed into a silicone mold, and then the molds were filled with epoxy resin. After solidification, one side of the sample was polished to obtain the working surface of the intermetallic phase. After polishing, the samples were immediately studied using electrochemical methods. The test was carried out using an MTech COR-500 potentiostat. The study was conducted in a 3 % aqueous solution of sodium chloride (pH≈7), 3 % aqueous solution of sodium chloride that was acidified by hydrochloric acid to pH≈1 and 3 % aqueous solution of sodium chloride that was alkalinized by sodium hydroxide to pH≈13. The Al_5SiFe sample was used as the working electrode, a silver chloride electrode (SCE) served as the reference electrode, and a platinum grid electrode was used as the counter electrode. A potential change rate of 10 mV/s was applied. Obtained data were processed using BioLogic EC-Lab V11.50 software.

Results and disscussion

According to the results of the corrosion study of samples №1–3 of the intermetallic phase Al_5SiFe in acidic chloride-containing solutions with pH 0.5–1.5 at a temperature of +20 °C, it was shown that the mean loss of their mass (Δm), mean corrosion rate (CR), and corrosion depth index (CDI) were 0.352 g, 2.071 $g/m^2 \cdot h$, and 5.26 mm/year, respectively. According to DSTU 13819-68, the corrosion indexes of these samples can be assessed as 9 points (Table 1). It was established that in the same solution, but at a temperature of +40 °C, the corrosion indexes (Δm, CR, and CDI) increased by 1.04 times, which is consistent with previously published materials [12, 13] for corrosion-resistant steels and alloys. An increase in the pH of the medium up to 1.5–2.5 at +20 °C promotes a decrease in the corrosion losses of samples №4–5 to 0.325 g, the mean corrosion rate to 1.909 $g/m^2 \cdot h$, and corrosion depth index to 4.85 mm/year. At pH 1.5–2.5 and +40°C, the indexes decreased by 8.4 % and 6.4 % (samples №28-30) in comparison with pH = 0.5–1.5 and +40 °C. It should be mentioned that in such pH intervals, the studied samples underwent active corrosion dissolution with the maximum degree on the 9-point scale; hence, the intermetallic phase Al_5SiFe is considered to be low-resistive under these conditions (Table 1). It was estimated that mean weight loss, mean corrosion rate, and corrosion depth index of samples №7–9 decreased to 0.053 g, 0.311 $g/m^2 \cdot h$, and 0.75 mm/year, respectively, with an increase in pH of the solutions up to 3.5–4.5 at +20 °C in comparison with pH = 1.5–2.5 and +20 °C (Table 1).

At the same time, at +40 °C, the indicators Δm, CR, and CDI of samples №31–33 also decreased to 0.169 g, 0.1 $g/m^2 \cdot h$, and 2.5 mm/year, respectively, with an increase in the pH of chloride-containing media to 3.5–4.5 in comparison with pH = 1.5–2.5 and +40 °C. It should be noted that at +20 °C, the aforementioned indicators for the studied samples №7-9 decreased by 6.1 times with an increase in the medium's pH from 1.5–2.5 to 3.5–

4.5. These samples were classified as low-resistant according to DSTU 13819-68. Nevertheless, at +40 °C, the corrosion indicators (Δm, CR, and CDI) of samples №31–33 decreased only by 2 times with the same increase in the medium's pH. Under these conditions, the Al_5FeSi intermetallic phase is considered as low-resistant to corrosion processes. The analysis of the corrosion behavior of samples №4–6, №7–9, and №28–30, №31–33 revealed that the effect of the temperature of chloride-containing media on their corrosion losses was slightly higher than the effect of pH value. It should be noted that these correlations are typical for the pitting corrosion of corrosion-resistant steels and alloys [14–16]. It was estimated that in chloride-containing solutions with pH 5.5–6.5, at temperatures of +20 °C and +40 °C, the corrosion indicators of samples №10-12 and №34-36 were significantly lower compared to samples №7–9 and №31–33 in solutions with pH 3.5–4.5 (Table 1). In particular, the aforementioned indicators decreased by 9.6 times at +20 °C and 8.4 times at +40 °C, which was caused by the formation of a protective film of corrosion products on the surface of the sample.

However, under the abovementioned conditions, samples may be exposed to local corrosion, which is controlled by oxygen depolarization on cathodic areas. The presence of pores in the protective coating and contact of the intermetallic phase Al_5FeSi with the medium contributes to the formation of micro-galvanic pairs, wherein the corrosion products covering the surface act as the cathode. The electrochemical characteristics of these micro-galvanic pairs depend on the pH of the corrosive medium, and they are likely densest and exhibit the best protective characteristics in neutral mediums. In solutions with pH 6.5–7.5, minimal corrosion indicators (Δm, CR, and CDI) were observed, which were on average 3.4 times lower at +20 °C and 3.0 times lower at +40 °C compared to solutions with pH 5.5-6.5. At the same time, the indicators were 2.7 times lower at +20 °C and 1.8 times lower at +40 °C compared to solutions with pH 8.5–9.5 (Table 1). Notably, in chloride-containing solutions with pH 6.5–7.5, temperature had the least effect on the corrosion loss indicators of the studied samples. This observation is likely associated with the electrochemical characteristics of the corrosion products and the density of the protective film they form. Under the conditions of the study, the film formed by corrosion products contained the least number of pores and other microdefects in the coating responsible for contact between the corrosive medium and the intermetallic phase Al_5SiFe, thus preventing the anode dissolution of the latter. Meanwhile, corrosion products accumulated around the intermetallic phase under the coating. These products underwent hydrolysis, further acidifying the medium and, as a result, increasing local corrosion losses. The obtained data are consistent with previously published results [4; 5], which describe the same tendency for corrosion product acidification due to limited access of oxygen to the anode area. Under these conditions, intensive dissolution of the austenite matrix of corrosion-resistant steels was observed in acidic chloride-containing media. At the same time, inclusions of oxides [17], nitrides [18], and titanium sulfides, around which local corrosion damages were found in chloride-containing solutions, did not undergo corrosive destruction. The higher corrosion resistance of these inclusions is due to their electrochemical properties, specifically the more positive value of their standard electrode potential compared to steel in the same environment. These inclusions played the role of the cathode area, where the reduction of dissolved oxygen occurred. According to published results [9], the intermetallic phase in AK8M3 aluminum alloy also acted as a cathodic inclusion, initiating local corrosion processes in its vicinity. This data suggests that the intermetallic phase in the studied aluminum alloy also plays the role of a cathodic inclusion, around which local corrosion impairments occur. This hypothesis is based on the much higher corrosion losses of the alloy compared to the intermetallic phase and the different mechanisms of their corrosion disruption.

It has been shown that in aqueous alkaline solutions of NaOH with pH 10.5–11.5 at +20 °C, the corrosion indicators (Δm, CR, and CDI) for samples №19–21 of the intermetallic phase are 0.0281 g, 0.1657 $g/m^2{\cdot}h$, and 0.421 mm/year, respectively (Table 1). These values are 6.5 times higher compared to solutions with pH 8.5–9.5 at +20 °C. However, in the same alkaline solutions at +40 °C, the corrosion indicators (Δm, CR, and CDI) for samples №43–45 are 0.0538 g, 0.3165 $g/m^2{\cdot}h$, and 0.804 mm/year, respectively. These indicators are by 1.9 times higher than at +20 °C and 10.5–11.5, and by 4.5 times higher than in solutions with pH 8.5–9.5 at the same temperature. Therefore, the increase in pH of the corrosive medium in this range has a more pronounced effect on the corrosion weight losses of Al_5SiFe compared to the effect of increasing the temperature to +40 °C. This phenomenon is associated with differences in the mechanisms of the above-mentioned factors. In particular, it is known [14; 19–21] that the susceptibility of corrosion-resistant steels to pitting corrosion increases with temperature. This can be explained by an increase in the porosity of passive films and changes in their chemical composition. In alkaline medium, oxygen reduction occurs on the sample surface, further alkalizing the medium and increasing corrosion losses (Δm, CR, and CDI). It is evident that increasing the pH of the NaOH solution from 8.5–9.5 to 10.5–11.5 intensively alkalizes the medium at the surface of the sample and consequently increases corrosion losses. This tendency was observed with further increases in the pH of the corrosion medium. It was found that in NaOH solutions with pH 12.5–13.5 and temperature 20 °C, the mean weight losses of samples №22–24 and their CR and CDI increase to 0.1593 g, 0.937 $g/m^2 \cdot h$, and 2.38 mm/year, respectively (Table 1). These values are 5.7 times higher compared to solutions with pH 10.5–11.5 at +20 °C, and 2 times lower compared to the same medium at +40 °C.

It should be noted that the corrosion indicators (Δm, CR, and CDI) for samples №46–48 in NaOH solutions with pH 12.5–13.5 at +40 °C are similar to the indicators for samples №28–30 in HCl solutions with pH 1.5–2.5 at the same temperature. At the same time, corrosion indicators for samples №4-6 (pH =1.5–2.5, +20 °C) by two times higher compared to samples №22–24 (pH =12.5–13.5, +20 °C) (Table 1). This can be explained by the negligible effect of temperature on the level of corrosion losses of samples №1–3, 25–27, 4–6, and 28–30 in acidic media. Under these conditions, metal ionization is accompanied by the cathodic reaction of proton reduction ($2H^+ + 2e^- \rightarrow 2H_{ads}$). It should also be noted that the inclusions of the intermetallic phase Al_5SiFe in АК8М3 alloy [9] undergo hydrogen embrittlement as a result of hydrogen diffusion into the material. Together with low resistance to additional embrittlement [10; 11], this can lead to stress corrosion fracture.

The described effects of corrosion products on the corrosion behavior of the material in solutions with different pH are further confirmed by the results of the electrochemical study of the Al_5SiFe intermetallic phase in a 3 % NaCl solution and are consistent with previously obtained data [4; 5].

The results of the electrochemical studies (Figure) revealed that a corrosion potential E_{corr} = –1.247 V was established on the sample after its immersion into the studied solution. Anodic polarization of the sample to -1.16 V promotes a rapid increase in the anodic current, which is equivalent to its corrosion losses. It was found that further anodic polarization of the sample up to -0.649 V does not affect the anodic current of the sample or its corrosion in the solution. The above-mentioned corrosion behavior corresponds to the slowest corrosion of the samples in neutral or nearly neutral solutions (Table 1).

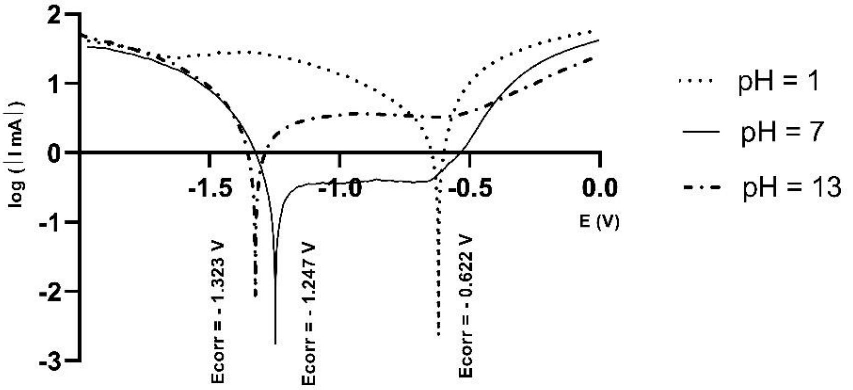


Figure. Tafel plots recorded for Al_5SiFe intermetallic phase in chloride containing solutions with various pH

Further anodic polarization results in a rapid increase in the anodic current, caused by the local breakdown of the porous layer on the surface. These processes are also characteristic of samples studied in alkaline medium (Table 1). It should be noted that the corrosion behavior of the samples in acidic chloride-containing solutions (Table 1) is similar to what was observed during polarization in the range E_{corr} = –1.247 V to –1.160 V, with the most intensive increase in anodic current. The corrosion potential of the studied inermetallic phase at pH≈1 is –0.622 V. It should be noted that at pH≈1 passivation region has not been observed what testify the absence of protective film in abovementioned conditions. In solution with pH≈13 the passivation region is preserved but weakly expressed what confirms the lability of protective film in such medium. The corrosion potential of the intermetallic phase in alkaline medium has been insignificantly shifted to negative values and registered at −1.323 V. Generally, the obtained results of electrochemical study are similar to the data obtained for $Al_8Mg_3FeSi_6$ intermetallic compound [22].

Hence, in acidic chloride-containing solutions with pH 0.5–1.5 and 1.5–2.5, the corrosion indicators (Δm, CR, and CDI) of the Al_5SiFe intermetallic phase samples depend only slightly on the temperature of the corrosive medium. In weakly acidic solutions with pH 3.5–4.5, the indicators are 10 times lower at +20 °C, but only 2 times lower at +40 °C. In neutral chloride-containing solutions with pH 5.5–6.5 and 8.5–9.5, the corrosion indicators of the intermetallic phase are the lowest and show little dependence on the temperature of the medium. This fact is associated with the formation of a protective layer of corrosion products, which inhibits the active corrosion of the samples. The increase in the solution's pH to 10.5–11.5 and 12.5–13.5 raises both the corrosion indicators of the Al_5SiFe intermetallic phase and the role of the temperature in the corrosion processes. These effects are caused by the local destruction of the protective layer on the surface of the sample under the aforementioned conditions.

Conclusion

Results of corrosion studies of the Al_5SiFe intermetallic phase samples revealed that the highest corrosion indicators (Δm, CR, and CDI) were registered in acidic chloride-containing media with pH 0.5–2.5. In strongly acidic media, the corrosion rate almost does not depend on the temperature of the corrosion environment. Increasing the pH of the corrosive medium to 3.5–4.5 resulted in a reduction of the corrosion indicators by 6.1 times at +20 °C and 2.0 times at +40 °C. In neutral or near-neutral media with pH 6.5–7.5, corrosion indicators were the lowest due to oxygen depolarization of cathodic processes and the presence of a protective layer formed by corrosion products. Under these conditions, the corrosion indicators at +40 °C were 4.2 times higher compared to +20 °C.

Increasing of the studied solutions` pH in the ranges of 8.5–9.5, 10.5–11.5, and 12.5–13.5 at +20 °C accelerated the corrosion processes by 2.7, 6.5, and 5.7 times, respectively. Raising the temperature to +40 °C further accelerated the corrosion rate by 1.8, 4.5, and 5.7 times, respectively. The corrosion behavior of the Al_5SiFe intermetallic phase was affected by the increased hydroxide content, which caused local degradation of the protective layers formed by corrosion products. Electrochemical studies revealed the electrochemical characteristics of the Al_5SiFe intermetallic phase, including its relatively high corrosion potential and its tendency towards passivation.

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