

## Evaluation of the Corrosion Resistance of the Al-Si-Cu-Mg Alloy with the Addition of Zirconium

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Aluminum alloys are one of the most used materials today, and therefore great emphasis is placed on their continuous development. Improving the ratio of strength and stiffness to weight, improving plasticity, casting properties or resistance to corrosion are examples of properties of aluminum alloys that are constantly being improved. This work focuses on the evaluation of the corrosion resistance of the Al-Si5Cu2Mg alloy with graded addition of zirconium (0.05; 0.10; 0.15; 0.20 wt.%). Corrosion resistance was evaluated based on immersion, exposure and potentiodynamic polarization tests. The addition of Zr to the AlSi5Cu2Mg alloy improved the thermodynamic stability in all evaluations. The application of heat treatment led to even more significant increases in corrosion resistance in almost all evaluations. Microscopic observation of the samples revealed mainly pitting corrosion along with intercrystalline corrosion.

**Keywords:** Al-Si-Cu-Mg alloys, Corrosion resistance, Zirconium

### 1 Introduction

Aluminum alloys of the Al-Si-Cu-Mg type belong to the most important alloys used in various industries. Al-Si-Cu-Mg alloys make up more than 85% of the total castings used in the automotive industry (engine blocks, cylinder heads, etc.). They are also used, for example, in the aviation industry as structural elements of airplanes and helicopters. The tightening requirements for castings in the automotive and aviation industries are pushing for the development of new, more advanced aluminum alloys that will meet demanding criteria. Sufficient corrosion resistance is a very important condition for a casting used, for example, in an electric car as a tub for batteries, which must withstand a corrosive environment during the entire life of the car [1].

Corrosion can be defined as an event in which a physicochemical interaction occurs between the metal and the environment. The result are permanent changes in the physical, chemical and mechanical properties of the corroding material. Corrosion of metals is characterized by the fact that almost all phenomena occur as a result of an electrochemical reaction between the metal or alloy and the liquid phase. At the interface between the metal and the solution, the transfer of electric charge occurs, which leads to the formation of electrochemical reactions [2-4]. Electrochemical reactions can occur by two methods. The first method is the oxidation of the metal atom and

the formation of  $M^{n+}$  ions, which are released into the solution. This creates a flow of electrons in the metal in the direction solution - metal. Anodic current  $i_a$ , which arises as a result of oxidation processes, flows in the direction metal - solution. The second method is based on the reduction of ions or solution molecules. During this process, electrons are taken from the metal and a new chemical compound is formed. The resulting flow of electrons in the metal during this reaction has the direction metal - solution. The resulting cathodic current  $i_k$  also flows in the direction solution - metal [5,6].

The corrosion resistance of aluminum alloys is ensured by a compact  $Al_2O_3$  oxide layer on the surface of the material. However, aluminum oxide dissolves under the influence of acidic and basic environments (solutions) and is stable only in neutral solutions. After the oxide layer dissolves, a breakdown occurs and the corrosion process continues directly on the metal, so the surface of the alloy is broken evenly [7]. The corrosion resistance of aluminum alloys is determined by metallurgical and environmental aspects. From the point of view of metallurgical aspects, the corrosion resistance of alloys is influenced by the chemical composition, heat treatment and the presence of inclusions in the material. From a material and technical point of view, corrosion is evaluated as a process in which mechanical properties such as strength, toughness, dimensional stability, tightness or deterioration of heat transfer [8].

The effect of chemical composition on corrosion resistance can be described through the potential of individual additive elements and their electrode character towards Al (Al solid solution). Mentioned potential describe, that if the electrochemical potential of an element in the form of an intermetallic phase is higher than that of pure aluminum, this phase becomes more cathodic (does not corrode), while aluminum remains anodic. This causes anodic dissolution of the surrounding aluminum matrix and local corrosion. Elements with a more cathodic character are e.g. Cu, Mn and Si [9,10].

Copper is among the most electrochemically noble additive elements in aluminum alloys. Its presence is problematic from the point of view of corrosion resistance, as intermetallic phases with Cu content form unwanted cathodic regions that do not dissolve in the electrolyte and cause anodic dissolution of the surrounding aluminum matrix. This increases the susceptibility to local corrosion [11,12].

Silicon in the presence of magnesium forms the Mg<sub>2</sub>Si intermetallic phase with an anodic character, which is less electrochemically noble than the aluminum matrix. Alloys with a Si content exceeding its maximum solubility in the aluminum matrix contain Si excluded along the grain boundaries, which adversely affects the formation of intercrystalline corrosion and stress corrosion [13-15].

The aim of the presented article is to describe and evaluate the influence of the graded amount of Zr in the AlSi5Cu1Mg alloy on corrosion resistance in NaCl environment and atmospheric corrosion. The effect of corrosion on the samples in the cast state and after heat treatment was also evaluated.

## 2 AlSi5Cu2Mg alloy and sample preparation

The AlSi5Cu2Mg alloy belongs to the hypoeutectic alloys characterized by low density, good castability, plasticity and mechanical properties. Currently, the alloy is used in the automotive industry. The AlSi5Cu2Mg alloy was in a pre-modified state as part of the experimental work, and 5 material variants of the alloy were cast. AlSi5Cu2Mg alloy without addition of zirconium (reference alloy) and four alloys with addition of zirconium in a graded amount of 0.05; 0.10; 0.15 and 0.20 wt. %. The designation of the alloys was as follows: reference alloy and based on wt. % Zr as Zr05; Zr10, Zr15 and Zr20. After melting the batch of AlSi5Cu2Mg alloy, in four cases AlZr20 master alloy with a pre-calculated weight was inserted into the melt at a melt temperature of  $770 \pm 5$  °C. The casting temperature was kept at a constant value of  $770 \pm 5$  °C for each alloy. Casting took place in a metal mold with a temperature of  $200 \pm 5$  °C. The designation and chemical composition of the five experimental alloys is shown in Tab. 1.

**Tab. 1** Chemical composition of the experimental AlSi5Cu2Mg alloys with Zr addition

Alloy	Si [wt. %]	Fe [wt. %]	Cu [wt. %]	Mn[wt. %]	Mg[wt. %]	Ti [wt. %]	Zr [wt. %]
Reference	5.468	0.182	1.912	0.021	0.290	0.013	0.001
Zr05	5.672	0.191	1.909	0.022	0.291	0.013	0.051
Zr10	5.648	0.188	1.918	0.021	0.289	0.014	0.102
Zr15	5.551	0.191	1.914	0.021	0.290	0.014	0.124
Zr20	5.427	0.183	1.903	0.022	0.291	0.014	0.189

For the purposes of corrosion tests, samples were taken from the castings and prepared in the form of plates with a length of 50 mm, width of 25 mm and thickness of 10 mm. The surface of each sample was sanded with P1200 grit sandpaper, rinsed with water and then degreased with ethanol. 20 samples were made from each alloy, where 10 samples were heat treated. The heat treatment regime was as follows:

- solutionizing at  $500 \pm 5$  °C for 6.5 hours;
- rapid cooling in water with a temperature of  $85 \pm 5$  °C;
- artificial aging at  $250 \pm 5$  °C for 4 hours.

## 3 Methodology of experiments

### 3.1 Immersion and exposure tests

A simple form of investigating the corrosion resistance of materials is an immersion test, or full immersion test in which material samples are immersed in a liquid environment to determine its effect. The samples used in this test were thoroughly cleaned, washed in distilled water and ethanol and later dried. Subsequently, they were weighed on an analytical weight and placed in a container so that they did not touch the walls of the container and thus did

not affect the corrosion processes (Fig. 1a). They were immersed in a 3.5% NaCl solution. During the test, the level of the electrolyte and the concentration of the NaCl solution were constantly monitored. The temperature in the room where the samples were stored was maintained at a constant temperature of  $20 \pm 2$  °C.

The tests lasted 80 days and after their completion, it was possible to evaluate the appearance of the samples, the distribution of corrosion attack and the change in weight. After being removed from the solution, the samples were thoroughly cleaned, dried and weighed on an analytical weight. The rate of corrosion attack was assessed using a quantitative method - the so-called gravimetry. Weight losses were calculated based on the obtained values. In this way, the rate of corrosion attack was determined according to the relationship:

$$v = \Delta m \cdot 8760 / \rho \cdot S \cdot t \quad (1)$$

Where:

$v$ ...Corrosion rate ( $\text{mm} \cdot \text{year}^{-1}$ )

$\Delta m$ ... Difference between the initial and final weight of the sample (g),

$\rho$ ...Material density ( $\text{g} \cdot \text{mm}^{-3}$ ),  $2\,700 \text{ g} \cdot \text{mm}^{-3}$ ,

$t$ ...Time of exposure of samples in a corrosive environment (h),

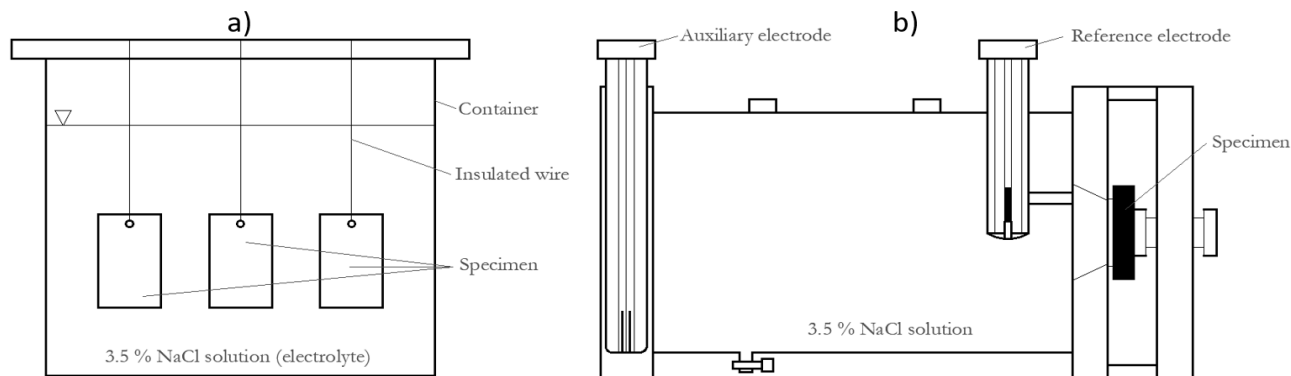
$S$ ...Area of the sample exposed to the corrosive environment ( $\text{mm}^2$ ).

Exposure tests were performed to verify the corrosion resistance of the experimental material due to atmospheric corrosion. The samples were stored out-

doors for 6 months, where they were exposed to atmospheric corrosion. Before and after the test, the samples were weighed in order to determine possible weight losses or increments. Finally, the corrosion rate was evaluated gravimetrically according to the equation (1).

### 3.2 Potentiodynamic polarization tests

Potentiodynamic tests (PD tests) explain the corrosion behavior of the experimental material and characterize the corrosion mechanisms, corrosion rate and susceptibility to material corrosion in a certain environment. These tests are performed using a laboratory potentiostat VSP Biologic SAS in a closed corrosion chamber in a three-electrode connection. The samples were stored in a solution with 3.5% NaCl at laboratory temperature. Corrosion measurements were performed in a three-electrode cell system in a closed chamber on a Biologic SP300 laboratory potentiostat. The first electrode serves as the working electrode, which was connected to the experimental sample. The second electrode consisted of a platinum mesh (auxiliary electrode) and the third electrode was the reference (Fig. 1b). The samples were tested at potentials ranging from -200 mV to +500 mV relative to the open circuit potential (OCP) with a plotting rate of 1 mV/s after 10 minutes of potential stabilization. The measured curves were analyzed using the Tafel extrapolation method using EC Lab V10.42 software, which determined the values of corrosion potential  $E_{\text{corr}}$ , corrosion current density  $i_{\text{corr}}$  and corrosion rate  $r_{\text{cor}}$ .



**Fig. 1** Schematic representation of sample storage during: a) immersion; b) potentiodynamic test

## 4 Results

### 4.1 Immersion and exposure tests

The change in the average value of the samples weight from experimental AlSi5Cu2Mg alloys with graded addition of Zr in the cast state (AC) and after heat treatment (HT) due to the environment of 3.5% NaCl solution and due to atmospheric corrosion is shown in Tab. 2. Based on the weight values before and after the immersion test, weight losses were determined. The experimental AlSi5Cu2Mg alloy showed

lower mass loss values after the addition of Zr than the reference alloy. A decrease of 17 to 23% was observed in the cast state and 35 to 40 % after heat treatment, depending on the addition of Zr.

Weight loss due to atmospheric corrosion was minimal. The largest decrease was shown by the alloy with 0.05 Zr AC with a value of 0.0031 g, and for the heat-treated condition, the alloy 0.20 Zr HT with 0.0016 g. On the contrary, no loss of material due to atmospheric corrosion was recorded with the reference alloy after heat treatment.

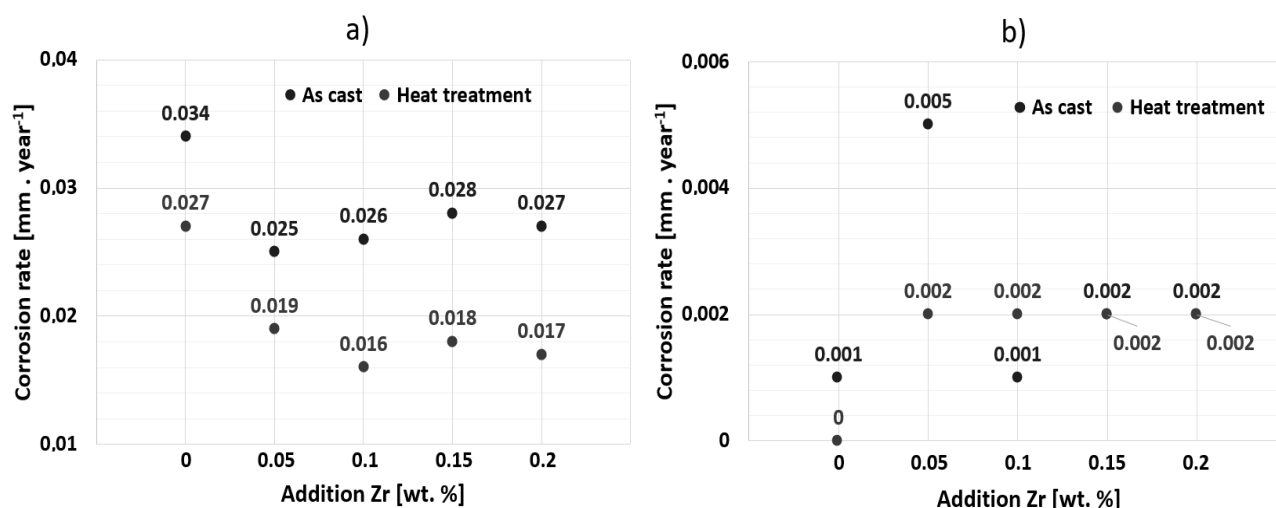
**Tab. 2** Average weights of experimental alloys *AlSi5Cu2Mg* for and after immersion and exposure test [g]

Alloy	Average weight before test		Average weight after test		Weight change	
	Immersion	Exposure	Immersion	Exposure	Immersion	Exposure
Ref. AC	4.1736	3.9679	4.1514	3.9673	0.0222	0.0006
Ref. HT	4.2334	4.4897	4.2161	4.4897	0.0173	0
Zr05 AC	4.3464	3.5721	4.3294	3.5690	0.0170	0.0031
Zr05 HT	4.4108	4.5721	4.3980	4.5708	0.0128	0.0013
Zr10 AC	4.2310	4.5282	4.2132	4.5277	0.0178	0.0005
Zr10 HT	4.3017	4.2990	4.2913	4.2980	0.0104	0.0010
Zr15 AC	4.2426	4.2129	4.2242	4.2118	0.0184	0.0011
Zr15 HT	4.5103	4.3920	4.4958	4.3908	0.0145	0.0012
Zr20 AC	4.0313	3.8981	4.0132	3.8965	0.0181	0.0016
Zr20 HT	4.2293	4.3310	4.2181	4.3294	0.0112	0.0016

Lower values of weight losses were also related to lower corrosion rates of the experimental Zr-alloyed alloys (Fig. 2a). Based on these results, it can be seen that the most intense corrosion attack due to the environment of 3.5% NaCl solution took place in the experimental alloy without the addition of Zr in both conditions. The addition of Zr decreased the corrosion rate and slowed down the corrosion attack. The application of heat treatment led to a significant decrease in the corrosion rate and thus to the slowing

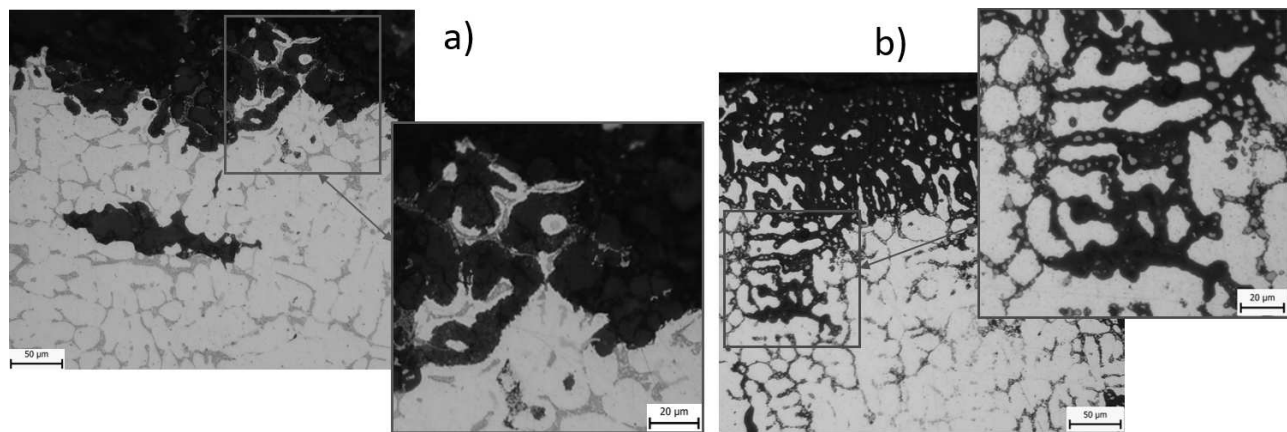
down of corrosion phenomenon.

Due to the influence of atmospheric corrosion, the corrosion attack was only minimal and the velocities reached low values (Fig. 2b). The highest rate of corrosion attack was achieved by the Zr05 alloy in the cast state. The other alloys reached velocities of 0.001 to 0.002 mm. year<sup>-1</sup>, while the reference alloy did not show a weight loss after heat treatment and therefore a velocity of 0 mm. year<sup>-1</sup> was recorded.

**Fig. 2** Corrosion rate  $r_{cor}$  depending on wt. % Zr and alloy state: a) immersion test; b) exposure test

Macroscopic evaluation on samples of all alloys showed the presence of pitting corrosion in combination with local intercrystalline corrosion. In Fig. 3a pitting corrosion can be seen on the as-cast sample from the reference alloy. Pitting corrosion occurs along grain boundaries in the eutectic region. Corrosion pits appear on the surface and spread to the depth of the alloy, while the  $\alpha$ -phase dissolves. The heat treatment

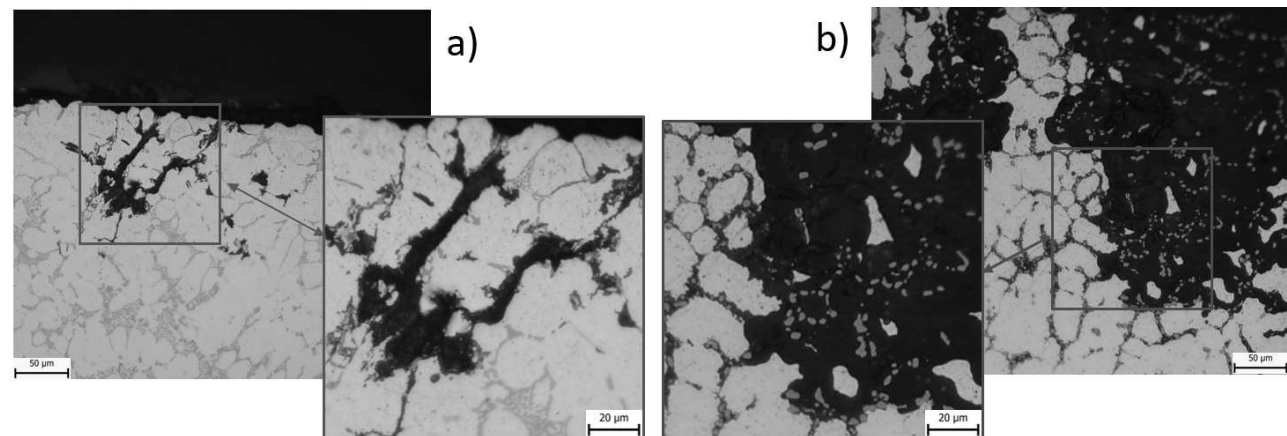
of the reference sample caused rather local attack by pitting corrosion (Fig. 3b), probably in the areas with the narrowest passive layer. Corrosion progressed deeper than in the as-cast alloy sample, and there was also considerable dissolution of the  $\alpha$ -phase in the surface layers. However, the eutectic retained its morphology, thus demonstrating resistance to corrosion attack.



**Fig. 3** Evaluation of corrosion attack of reference alloy samples after immersion test: a) as cast state, b) after HT

A cross-section of a Zr05 AC alloy sample (Fig. 4a) shows a more even distribution of pitting corrosion on the surface of the sample. On the contrary, after heat treatment, the corrosion is uneven, but in a smaller amount, it progresses deeper into the material

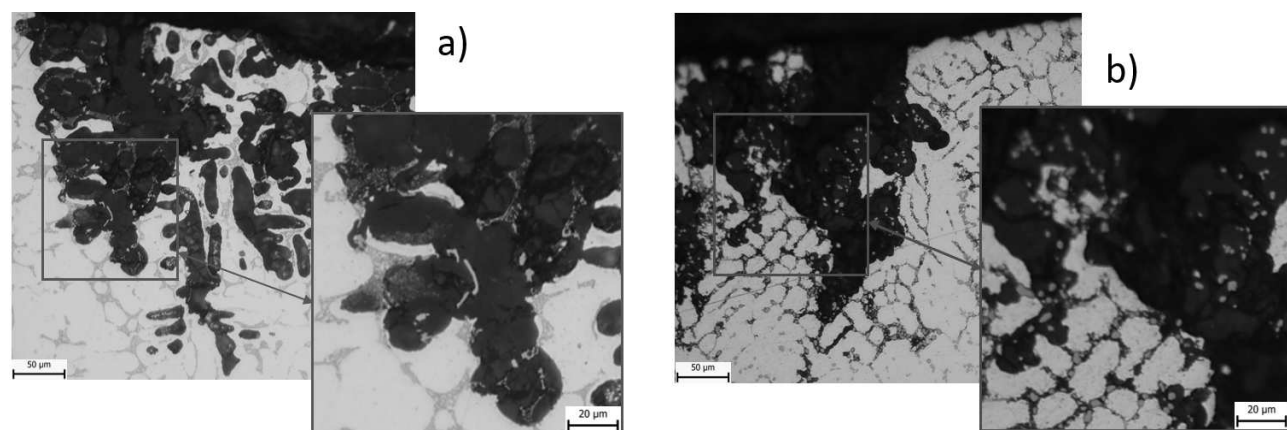
(Fig. 4b). In both states, samples from the alloy with an addition of 0.05 wt. % Zr, considerable dissolution of the  $\alpha$ -phase is observed, where there are undissolved eutectic particles in the form of plates (sample AC) or round particles (HT sample).



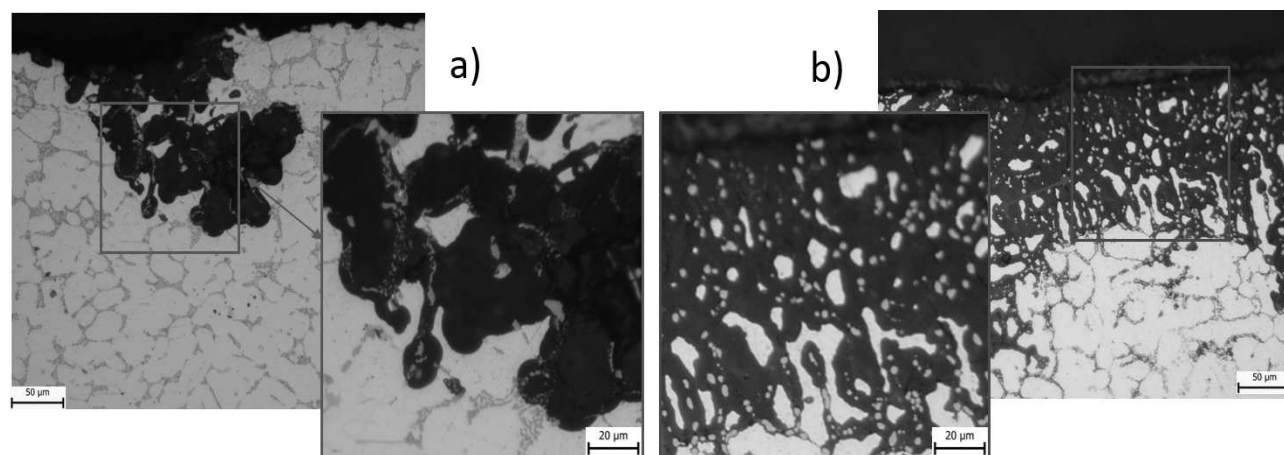
**Fig. 4** Evaluation of corrosion attack of Zr05 alloy samples after immersion test: a) as cast state, b) after HT

Addition of 0.15 wt. % Zr caused that a more localized corrosion attack was already observed in the Zr10 AC alloy sample than in the Zr05 AC alloy sample, but with deeper penetration into the material (Fig. 5a). Corrosion attack after heat treatment of the Zr05

HT alloy sample (Fig. 5b) is almost identical to that of the Zr10 HT alloy. Increasing the amount of Zr to 0.15 wt. % resulted in a decrease in depth or slowing down of the intensity of corrosion attack for both states of the alloy (Fig. 6).

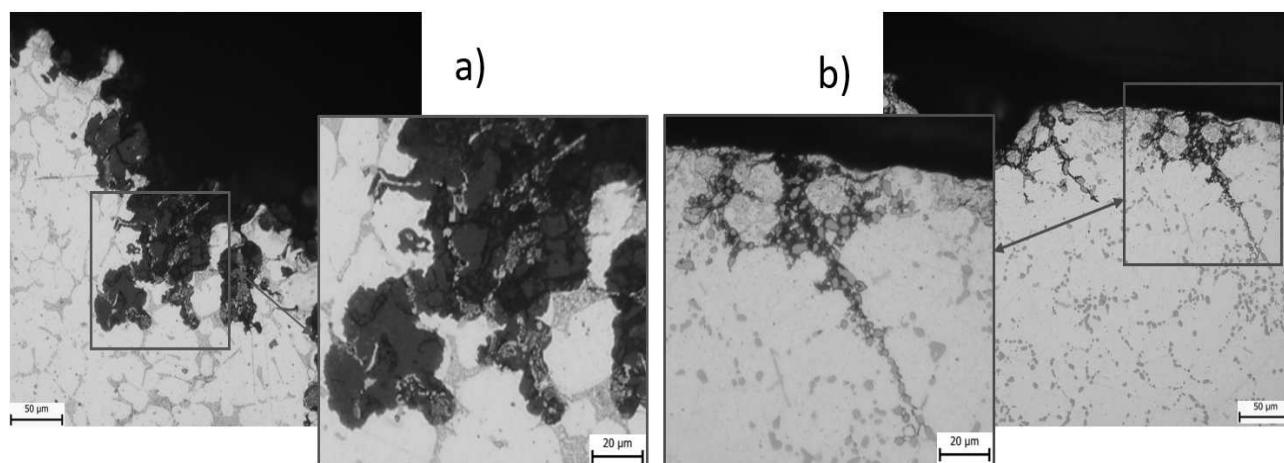


**Fig. 5** Evaluation of corrosion attack of Zr10 alloy samples after immersion test: a) as cast state, b) after HT



**Fig. 6** Evaluation of corrosion attack of Zr15 alloy samples after immersion test: a) as cast state, b) after HT

Samples from alloys with the highest amount of Zr addition 0.20 wt. % (Fig. 7) confirm the trend of decreasing the intensity of corrosion attack with an increase in the amount of Zr in the alloy. In both cases, it is the appearance of a smaller number of corrosion pits forming on grain boundaries in the area of eutectic Si.



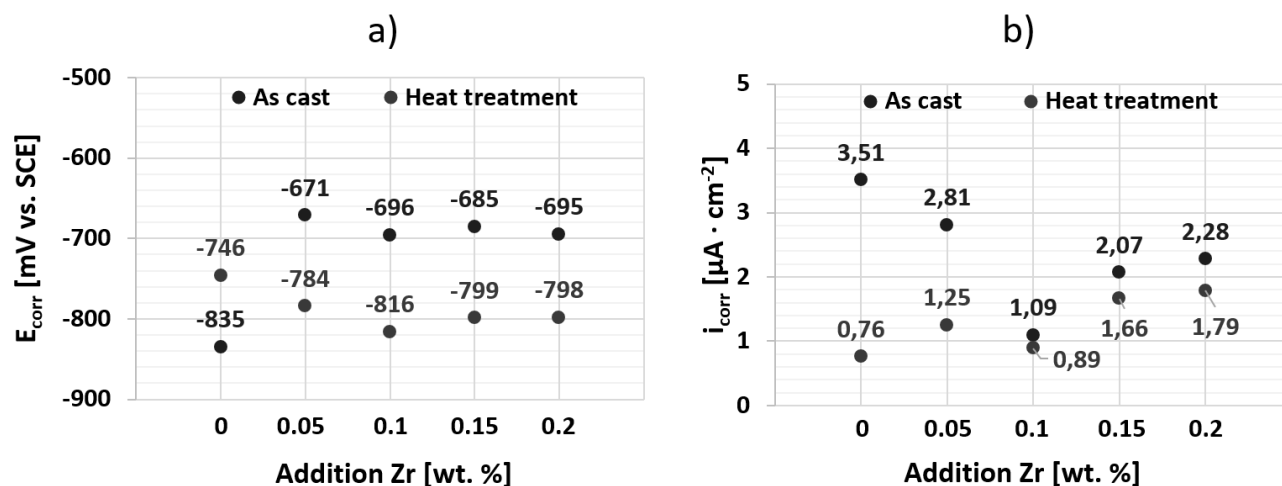
**Fig. 7** Evaluation of corrosion attack of Zr20 alloy samples after immersion test: a) as cast state, b) after HT

## 4.2 Potentiodynamic polarization tests

$E_{\text{corr}}$  and  $i_{\text{corr}}$  values were measured using potentiodynamic polarization tests. The values for each sample are graphically processed in Fig. 8 (the value represents the average of 5 measurements). The resulting values of the corrosion potential  $E_{\text{corr}}$  represent the thermodynamics of corrosion, while the values of the corrosion current density  $i_{\text{corr}}$  represent the characteristics in terms of corrosion kinetics. The corrosion potential of the as-cast reference alloy showed a value of -835 (mV vs. SCE) and heat treatment improved the  $E_{\text{corr}}$  values towards positive values (Fig. 8a). The addition of Zr to the as-cast alloy resulted in a shift of the  $E_{\text{corr}}$  values towards positive values by approximately 20%. By subsequently increasing the wt. % Zr in the alloy had only minimal changes. The presence of Zr in the cast alloy clearly increased the thermodynamic nobleness of AlSi5Cu2Mg alloys in the given corrosion environment. The opposite trend occurred in samples

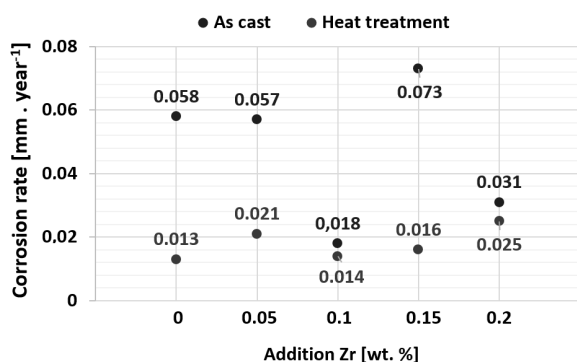
after heat treatment, when the addition of Zr, on the contrary, reduced the values of the corrosion potential  $E_{\text{corr}}$  or thermodynamic nobleness (for the Zr10 HT alloy sample there was a decrease of approximately 9 % compared to the reference alloy).

The corrosion current density  $i_{\text{corr}}$  of the reference alloy after heat treatment showed a clear improvement compared to the as-cast state (Fig. 8b). The decrease in corrosion current density  $i_{\text{corr}}$  by the application of heat treatment means that the corrosion reactions took place more slowly and the experimental alloys degraded more slowly in the corrosion environment than the as-cast alloys. By adding and gradually increasing Zr, the corrosion current density in the as-cast state decreased to a minimum in the Zr10 AC alloy sample (higher corrosion resistance). By increasing the addition of Zr to 0.15 and 0.20 wt. % there was a small increase in  $i_{\text{corr}}$  values. Heat treatment together with the addition of Zr led to a slight increase in the current density values of  $i_{\text{corr}}$  compared to the reference alloy.



**Fig. 8** The results of the potentiodynamic test depending on the wt. % Zr and alloy state: a)  $E_{corr}$ ; b)  $i_{corr}$

The resulting corrosion rate corresponds to the values of the current density  $i_{corr}$ , with the exception of the Al-Si5Cu2Mg alloy with 0.15 wt. % Zr (Fig. 9). The corrosion rate of the investigated alloy in the heat-treated state along with graded addition of Zr had fluctuating results, but the values ranged in a narrow range of 0.014 to 0.025 (mm. year<sup>-1</sup>).



**Fig. 9** Corrosion rate during potentiodynamic test depending on wt. % Zr and alloy state

## 5 Discussion

The results of immersion tests in a 3.5% NaCl solution clearly show that the application of heat treatment reduced the kinetics of corrosion phenomena. The addition of Zr to the alloy had an equally positive effect when compared to the reference alloy. However, the amount of Zr in the alloy no longer played such a significant role, and the corrosion rate changed only slightly. Several factors contributed to the increase in corrosion resistance. The first can be the combination of oxygen and zirconium and the formation of an oxide layer  $ZrO_2$  on the surface of the experimental samples. The presence of the oxide layer  $ZrO_2$  slowed down at the interface of the surface of the experimental alloys and the corrosion environment. The second factor affecting the reduction of the

kinetics of corrosion phenomena was also the refinement of the microstructure of the experimental alloys after the addition of Zr, which led to a more even distribution of the alloying elements. Refinement of the microstructure due to the addition of Zr is described in previous research [13]. A finer microstructure led to a reduction in the number of locally occurring corrosion microcells, i.e. galvanic cells that were created between individual structural components of the experimental alloys. An increase in corrosion resistance after the addition of Zr due to the formation of an oxide layer of  $ZrO_2$  and a refinement of the microstructure was also observed in studies [14,15].

Potentiodynamic polarization tests in the evaluation of the corrosion potential of  $E_{corr}$  showed, similarly to immersion tests, a positive effect after the addition of Zr to the alloy. However, the amount of Zr added had no significant effect on the results. On the other hand, when evaluating the current density  $i_{corr}$ , which is a more important factor from the point of view of kinetics, the best results were shown by the alloy with the addition of 0.10 wt.% Zr. Similar results were also obtained in studies [16], where Zr had an equally positive effect on improving the corrosion resistance of aluminum alloy in NaCl solution. In the same study, the positive effect of the addition of Zr is described in such a way that intermetallic phases of the  $Al_3Zr$  type have a negligible effect on the formation of electrochemical (galvanic) corrosion in Al alloys due to the small potential difference of this intermetallic phase and the Al matrix, which indicated a positive effect of the addition of Zr on their corrosion resistance.

The clear positive effect of heat treatment on corrosion resistance in almost all evaluations can be attributed to the change in microstructure. The intermetallic phases rich in Cu and Mg, present especially along the grain boundaries, were dissolved into a solid solution due to heat treatment, which led

to a decrease in the anodic and cathodic polarization of these phases against the aluminum matrix and a decrease in susceptibility to intercrystalline corrosion. However, the precipitation of dispersed particles in the matrix could increase the susceptibility of the alloy to pitting corrosion.

## 6 Conclusions

The aim of the paper was to analyze and evaluate the corrosion resistance of the AlSi5Cu2Mg alloy with graded addition of zirconium in the cast state and after heat treatment. From the measured results, it can be concluded that the addition of the transition element Zr to the alloy had a positive effect on the thermodynamic corrosion stability of the investigated alloy in both conditions and in the 3.5% NaCl environment. The beneficial effect of Zr was manifested through the formation of an oxide layer of ZrO<sub>2</sub> and the refinement of the microstructure. However, the application of heat treatment proved to be a more effective option for improving the corrosion properties of the AlSi5Cu2Mg alloy. The dissolution of phases rich in Cu and Mg into a solid solution led to a slowing down of corrosion phenomena and thus to a decrease in the corrosion rate of the experimental alloys. Atmospheric corrosion was only minimally evident on the AlSi5Cu2Mg alloy with graded addition of zirconium.

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