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Effect of Al additions and heat treatment on corrosion properties of Mg-Al based alloys

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Methodology of research

ABSTRACT

Purpose: In this paper there is presented the corrosion behavior of cast magnesium alloys in as cast state and after heat treatment.

Design/methodology/approach: The following results concern scanning electron microscopy investigations in the SE as well BSE observation mode, for better phase contrast results, also qualitative microanalysis was applied for chemical composition investigations of the surface. Pitting corrosion resistance was carried out using the potentiodynamic electrochemical method (direct current), based on anodic polarisation curve. Based on the achieved anodic polarisation curves, using the Tefel extrapolation method near to the corrosion potential, the quantitative data were determined, which describe the electrochemical corrosion process of the investigated alloys: value of the corrosion potential E_{cor} (mV), polarisation resistance Rp (kΩ/cm^2), corrosion current density i_{cor} (μA/cm^2), corrosion rate Vp (mm/year) as well the mass loss Vc (g/m^2).

Findings: Surface morphology of the samples after corrosion test performed after and before heat treatment show irregular shaped pinholes and numerous cracks on the material surface layer.

Research limitations/implications: The applied cooling rate and alloy additions seems to be a good compromise for properties and microstructures, nevertheless further tests should be carried out in order to examine different cooling rates and parameters of solution treatment process and aging process.

Practical implications: Investigation results concerning the surface layer presents some interesting findings connected to the layer morphology, which can be of high interest for practical application for the reason of better layer quality as well as surface layer properties. Limitation of surface damage including irregular shaped pinholes and numerous cracks is of very high importance for decreasing the influence of pitting corrosion onto the surface layer corrosion resistance in very width range o applications.

Originality/value: The value of this paper is to define the influence of heat treatment parameters and aluminium addition on corrosion resistance properties of magnesium-aluminium cast alloys.

Keywords: Metallography; Metallic alloys; Corrosion resistance

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1. Introduction

During the last few decades the world has seen a rapid growth of application of magnesium and its alloys almost in every field of today's industry. This is due to numerous characteristics of the metal regarded to herein, which permit its use both as a structural element, and as a chemical addition to other metal alloys. It is 35% lighter than aluminium (2.7 g/cm^3) and over four times lighter than steel (7.86 g/cm^3) [1-7]. Magnesium alloys, beside a low density of (1.7 g/cm^3), have also some other advantages like good ductility, better noise and vibration dampening characteristics than aluminium and excellent castability, high stability of the size and shape, low shrinkage, low density connected to high strength compared to low mass, as well recyclability, which makes it possible to achieve recycled alloys with quality and properties very close to primary cast alloys, which makes it possible to apply these material instead of new manufactured Mg alloy for constructions of less importance [4-12].

Low mass with a very high strength makes it possible to manufacture elements made of this material by casting, by plastic deformation, mechanical treatment or welding. Alloying magnesium with aluminium, manganese, rare earths, thorium, zinc or zirconium increases the strength to weight ratio making them important materials for applications where weight reduction is important, and where it is imperative to reduce inertial forces [2]. The need for magnesium alloys is connected mainly to the development of automobile industry The requirement to reduce the weight of car components as a result of legislation limiting emission has created renewed interest in magnesium. Recent research and development studies of magnesium and magnesium alloys have focused on weight reduction, energy saving and limiting environmental impact. Together with mass decreasing the driving parameters improve, this is connected mainly to the dynamic behavior of the vehicles. A need for reduction of the transportation vehicle mass is very important, because more and more transportation vehicles is equipped with additive accessories (like airbags, safety belts, raising and lowering system for car windows, etc.) which increases mass and have influence not only on safety, but also on usable attractiveness of these vehicles [4-7, 13-18]. Volkswagen was the first to apply magnesium in the automotive industry on its Beetle model, which used 22 kg magnesium in each car of this model [2]. *General Motors* in their big cars (Savana & Express) use 26.3 kg of magnesium cast alloys, and in smaller cars (Safari, Astro) – 16.5 kg, Ford F - 150- 14.5 kg, VW Passat and Audi A4 and A6 from 13.6 to 14.5 kg, Alfa Romeo – 9.3 kg. The engine weight of Audi V8 Quattro model was reduced 5 kg compared to other Audi eight cylinder by using magnesium components. The instrument panel for the GMH-van vehicle is made of magnesium alloy which weights 12 kg as opposed to the 18 kg in steel [1-3]. Magnesium substitution in the automotive industry has steadily increased over the last decade, in parts such as valve covers, instrument panels, steering wheels, steering wheel armatures, cylinder block heads, transmission cases, clutch housings, lower crank cases, intake manifolds, brake and gas pedals and seat bodies [1-10] (Fig. 1). Steering wheel is the component that has received the greatest worldwide acceptance which made of Mg alloy. Assuming 20 kg of magnesium alloy components will be used instead of alternatives on 25% of the 40 million cars produced, one calculates that the car manufacturing industry requires 200.000 tons of magnesium alloys [2].

The strategy regarding the use of Mg in vehicles to achieve a "second Mg age" is based on the applications for cast components; here further development of Mg properties (e.g. improved creep resistance) will be helpful. Mg components in body, sheet and extrusion applications will follow in niche and premium vehicles supported by new Mg alloys with improved ductility and energy absorption (Fig. 2).

Significant research is still needed on materials processing, alloy development, joining, surface treatment, corrosion resistance, and mechanical properties improvement [1-5, 19, 20]. Different coating methods are used to increase the properties of surface layer of magnesium alloys (corrosion resistance, lubricity, high frictional-resistance, non-wetting properties, hardness, decorative and etc.): e.g. galvanic coating, anodisation, coating using the PVD method, surface laser alloying/cladding (Fig. 3). The goal of this paper is to present of the investigation results corrosion resistance of the casting magnesium alloys in its as-cast state and after heat treatment.

2. Experimental procedure

The investigations have been carried out on test pieces of MCMgAl9Zn1, MCMgAl6Zn1 the one of the most popularly magnesium alloys in as-cast and after heat treatment states made in cooperation with the Faculty of Metallurgy and Materials Engineering of the Technical University of Ostrava and the CKD Motory plant, Hradec Kralove in the Czech Republic for the reason of influence of heat treatment and chemical composition on corrosion resistance of the investigated materials. The chemical compositions of the investigated materials are given in Table 1.

A casting cycle of alloys has been carried out in an induction crucible furnace using a protective salt bath *Flux 12* equipped with two ceramic filters at the melting temperature of 750±10ºC, suitable for the manufactured material.

To improve the quality of a metal surface a protective layer *Alkon M62* has been applied. The material has been cast in dies with betonite binder because of its excellent sorption properties and shaped into plates of 250×150×25. The cast alloys have been heated in an electrical vacuum furnace *Classic 0816 Vak* in a protective argon atmosphere.

The heat treatment involved the solution heat treatment (heating material in temperature 375°C the 3 hours, it later warming in the temperature to 430°C, holding for 10 hours) and cooling in air and then ageing at temperature of 190°C, holding for 15 hours and cooling in air (Table 2).

Pitting corrosion resistance of the analysed alloys was carried out using the potentiodynamic electrochemical method (direct current), based on a anodic polarisation curve. The corrosive agent was a 3% NaCl solution. The measurements were carried out in a three-electrode two-chamber glass electrolyser wit a volume of 150 cm^3, equipped with a water jacket connected to the thermostat of the UH-4 type ensuring a regulation with accuracy of ±0.1°C. The samples as well the investigated electrodes were made from the cast magnesium alloys, the reference electrode was appropriately a platinum and calomel electrode. The measurements ware performed at the room temperature after 20 minutes from the first contact of the investigated material with the electrolyte, by a potential change rate of 120 mV/min. The surface area of the tested samples of the cast magnesium alloys was equal 0.5 cm^2.

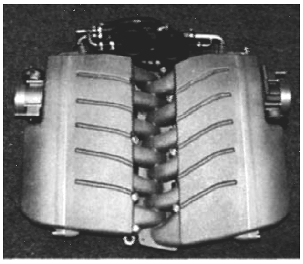
The corrosion current density i_{cor} was calculated on the basis of the potentiodynamic curves. For determination of the corrosion current density the areas with a corrosion potential E_{cor} were used according to the dependence given by Tafel. On the basis of the determined corrosion current values in the NaCl corrosion environment the polarization resistance Rp was determined using the embedded software function - „1st Stern Metod-Tafel extrapolation". The corrosion rate V_{cor} was determined by mind of the Stern & Tafel extrapolation for polarisation curves near the corrosion potential E_{cor} using the (1) equation:

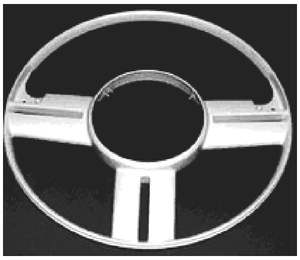
$$V_{cor} = \frac{3272 \cdot i_{cor} \cdot eq}{SA \cdot \rho}[mm/year] \quad (1)$$

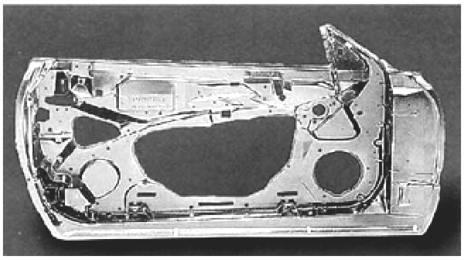
where:

i_{cor} -corrosion current density,
eq - electrochemical equivalent of the investigated material,
SA -tested surface,
ρ - density of the tested material.

The analysis of the investigated samples after the corrosion test was performed using the Zeiss SUPRA 25 scanning electron microscope with the EDAX Trident XM4 dispersive radiation spectrometer at the accelerating voltage of 20 kV.


12 cyl. intake manifold Audi (AZ91;8.43 kg)


VW 3L Lupo steering wheel core (AM50)


Die cast inner door without window frame, Daimler/Chrysler-SL (AM50; 4.5 kg)


VW steering lock (AZ91;295 g)


Corvette-wheel (AZ91; 8.6 kg)

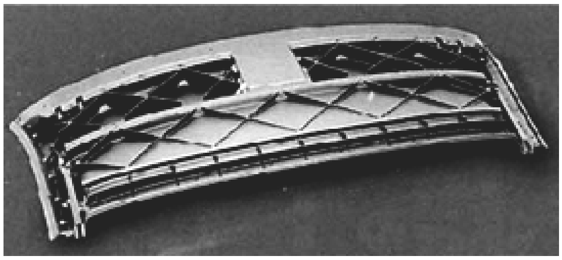

Part of the top system of Porsche 911 (AM50; 2.8 kg)

Fig. 1. Examples of automotive components made of Mg alloys [3]

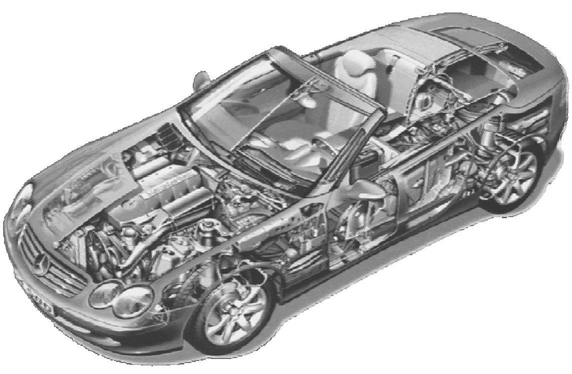
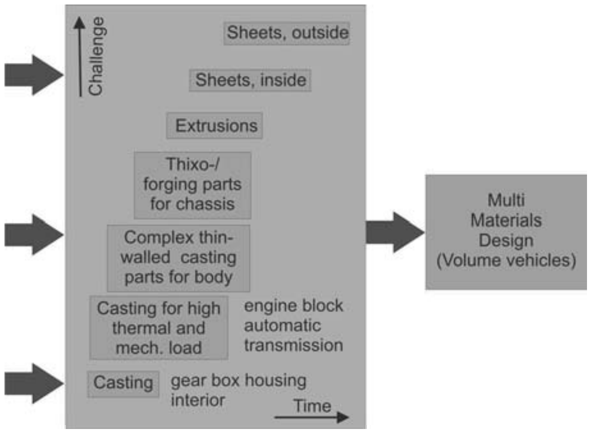



Fig. 2. Strategy for magnesium applications in vehicles [3]

Methods of pretreatment of the metal surface	
	Mechanical: smoothing by polishing, honing, grinding or blasting
	Cleaning: use of organic solvents and/or alkaline cleaning agents

Methods of applying inorganic coatings on the Mg surface	Electroless treatment	Electrochemical treatment	Physical methods
	Chromating	Anodisation (DOW 17, HAE, ANOMAG)	PVD
	Chromiumfree system	Anodical plasmachemical treatment (ASD) (MAGOXID, TAGNITE)	Flame or plasma spraying
	Electroless nikel	Electroplating (Zn, Cu, Ni, Cr, etc.)	Laser or electrobeam treatment

Methods of applying organic coatings on the Mg surface or on the inorganic undercoatings	Painting
	Water paints
	Powder paints/EPS
	Structural paints
	Immersion paints
	Anti friction paints

Fig. 3. Surface treatment of magnesium-based materials [3]

Table 1.
Chemical composition of investigation alloy

	The mass concentration of main elements, %						
Material type	Al	Zn	Mn	Si	Fe	Mg	Rest
MCMgAl9Zn1	9.09	0.77	0.21	0.037	0.011	89.79	0.0915
MCMgAl6Zn1	5.92	0.49	0.15	0.037	0.007	93.33	0.0613

Table 2.
Parameters of heat treatment of investigation alloy

Sing the state of heat treatment	Conditions of solution heat treatment		
	Temperature, °C	Time of heating, h	Way coolings
0	As-cast		
	Solution treatment		
1	430	10	Water
2	430	10	Air
3	430	10	Furnace
	Aging treatment after solution heat treatment with cooling in the water		
4	190	15	Air

3. Discussion of experimental results

As a result of the performed potentiodynamic investigations the polarisation curves and anodic loops for magnesium cast alloys in as-cast state as well after heat treatment (Fig. 4) were achieved. The polarisation curves of the investigated materials consist of branches of the anodic curves, which corresponds to the corrosion reaction process, as well of branches of cathodic curves, which corresponds to the hydrogenic depolarisation.

The curves show, that the investigated materials undergo pitting corrosion, which they are susceptible for, particularly in case of the cast magnesium alloys of the Mg-Al-Zn type. On the basis of the achieved anodic polarisation curves, using the Tefel extrapolation method near to the corrosion potential, the quantitative data were determined, which describe the electrochemical corrosion process of the investigated alloys: value of the corrosion potential E_{cor} (mV), polarisation resistance Rp (kΩ/cm^2), corrosion current density i_{cor} (μA/cm^2), corrosion rate Vp (mm/year) as well the mass loss Vc (g/m^2) (Table 3). The anodic polarisation curve process, and the corrosion current density determine the pulping rate of the tested surfaces (Fig. 4).

The parameters measured during the corrosion tests, acieved for the investigated magnesium alloys, as well for the environment - where the tests were carried out, cannot be considered separately. The values like corrosion potential E_{cor} or corrosion current density i_{cor}, can be used for comparison of the properties of the investigated materials both in as-cast state or after heat treatment, because all the performed measurements were made in the same environmental conditions.

The analysis of anodic polarisation curves, potential, corrosion resistance and density as well corrosion current confirm a slightly higher corrosion resistance of the MCMgAl6Zn1 material in as cast state compared to the MCMgAl9Zn1 samples, where the corrosion potential is equal -1621.2 mV, polarisation resistance 0.83 kΩ/cm^2, and the current density in the passive range 4.6 μA/cm^2 (Table 3).

During anodic scanning of the MCMgAl6Zn1 alloy after different solution heat treatment conditions there was achieved a lower corrosion current density i_{kor}, and that fore also a higher polarisation resistance compared to MCMgAl9Zn1 alloys with a higher Al content, what implicates better corrosion resistance of this material.

Similarly corrosion resistance investigations of the cast magnesium alloys were performed after heat treatment. The lowest value of corrosion current density i_{cor}, determining the smallest anodic pulping of the cast magnesium alloys with different aluminium content and connected with it best corrosion resistance after heat treatment show the MCMgAl6Zn1 alloys 3.4 μA/cm^2, and the current density in the passive range 1.3 μA/cm^2. Whereas the minimal resistance to the corrosion agent, which is connected to the failure propagation into the material as well to the material surface, show the MCMgAl9Zn1 alloys polarisation resistance 1.13 kΩ/cm^2, and the current density in the passive range 8.0 μA/cm^2 (Table 3, Fig. 4).

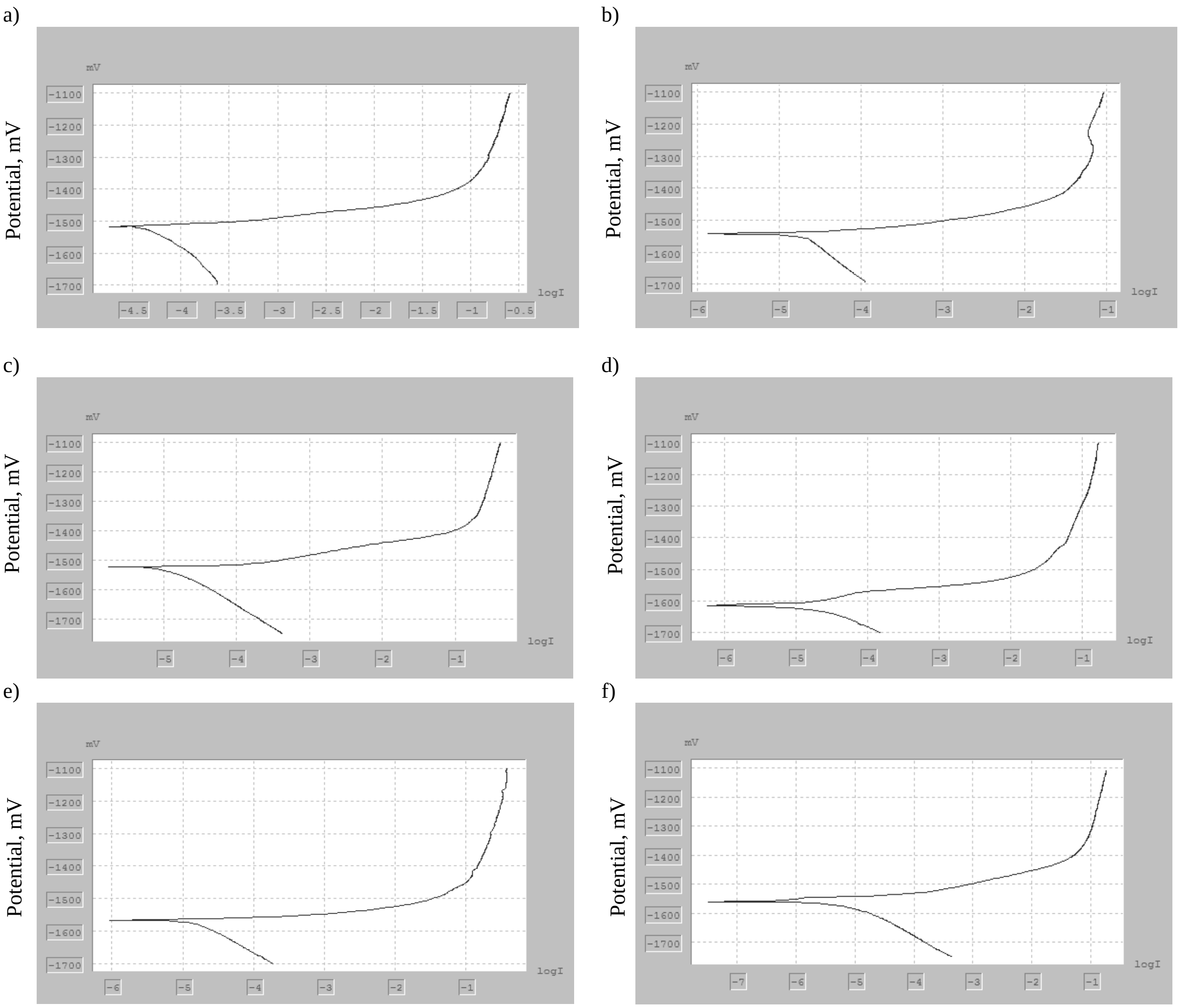


Fig. 4. Anodic curves of the magnesium cast alloys: MCMgAl9Zn1: a) in as-cast state, b) after solution heat treatment with cooling in the water, c) after aging treatment; MCMgAl9Zn1 d) in as-cast state, e) after solution heat treatment with cooling in the water, f) after aging treatment

Table 3.
The parameters measured during the corrosion tests

Investigation alloys	Sing the state of heat treatment	Corrosion potential E_{cor} (mV)	Polarisation resistance Rp (kΩ/cm^2)	Corrosion current density i_{cor} (μA/cm^2)	Corrosion rate Vp (mm/year)	Mass loss Vc (g/m^2)	Breakout potential E_n (mV)	Repassivation potential E_{cp} (mV)
MCMgAl9Zn1	0	-1551.6	0.4	13.7	0.31	1.48	-1470	-1692.3
	1	-1555.6	0.31	15.8	0.35	1.69	-1497.4	-1700
	2	-1537.4	0.27	18.9	0.43	2.03	-1468.6	-1689.8
	3	-1573.3	0.21	21.1	0.48	2.27	-1508.6	-1704.1
	4	-1522.6	1.13	8	0.18	0.85	-1443	-1691.1
MCMgAl6Zn1	0	-1621.2	0.83	4.59	0.10	0.50	-1547.6	-1672
	1	-1609.2	1.19	3.27	0.07	0.35	-1542.6	-1669
	2	-1586.8	1.56	3.09	0.07	0.33	-1500	-1666
	3	-1642.8	0.94	6.09	0.14	0.66	-1567.8	-1680
	4	-1555.2	3.4	1.3	0.03	0.14	-1460	-1659.2

This is dependent on surface corrosion acceleration in the corrosive agent, which is presented based on the calculated value of the corrosion rate Vp as well on the mass loss Vc of the investigated samples (Table 3). The analysis of the achieved results for the alloys with 9.6% aluminium content has confirmed the analogy of corrosion resistance increase of the materials after precipitation hardening in comparison to the as-cast alloys, and to the alloys after solution heat treatment. The lowest parameter values of the pitting corrosion process in all the investigated alloys have the samples after solution heat treatment cooled with furnace.

The values of the breakout potential E_n, which by growing pinholes on the tested samples surface occurs, and the value of the repassivation potential E_{cp} (Table 3), where no more active pinholes below the value exist, were both determined using the potentiodynamic curves progress. As a result of the comparison of the corrosion loops width of the investigate alloys (of E_n and E_{cp} parameters in the range, where new pinholes are not growing and in the existing ones corrosion processes can proceed), as well of the inclination angle and amplitude of the curve loops it can be conclude, that the lowest parameters (breakout potential and passivation potential) are achieved for materials after solution heat treatment cooled with furnace, whereas the best results are achieved for materials after precipitation hardened.

Surface morphology of the investigated samples after corrosion test performed before and after heat treatment (Figs. 5-9) show irregular shaped pinholes and numerous cracks in the surface layer of the material. Solution heat treatment of samples cooled with furnace is a special case of heat treatment, which is characterized with the lowest material properties connected to pitting corrosion. There occurs a non uniform precipitation process of compounds which are in very high amount present in the alloy in form of eutectic or intermetallic phase, what causes an increase of areas, where breaks of the passive layer occurs, and that for an more intensive corrosion process is present. Dissolution of surface by the corrosive agent begins along the β-phase edges, which works as a galvanic cathode accelerating the α-phase corrosion. This process concentrates on the alloy matrix especially in the area between the grain boundaries.

The smallest visible surface layer destruction is characteristic for magnesium cast alloys after ageing (Figs. 5d, 7d).

On the surface of the samples there are present also corrosion products, which builds compact conglomerates with characteristic needle shape formed in the majority of the cases inside of the pinholes (Figs. 8, 9). The EDS microanalysis confirm the occurrence of corrosion products on the sample surface (Fig. 10).

4. Summary

Pitting corrosion resistance test of the analysed alloys was carried out using the potentiodynamic electrochemical method (direct current), based on anodic polarisation curve. The analysis of the achieved results for the alloys with 9, 6% aluminium content has confirmed the analogy of corrosion resistance increase of these materials after precipitation hardening in comparison to the as-cast alloys, and to the alloys after solution heat treatment. A big influence on corrosion resistance has also aluminium content and connected with it amount of precipitations, because together with Al concentration increase also the amount of precipitations increases which are responsible for forming of new corrosion nuclei.

As a result of the comparison of the corrosion loops width of the investigate alloys as well of the inclination angle and amplitude of the curve loops it can be conclude, that the lowest parameters (breakout potential and passivation potential) are achieved for materials after solution heat treatment cooled with furnace, whereas the best results are achieved for materials after precipitation hardened.

Surface morphology of the investigated samples after corrosion test performed before and after heat treatment shows irregular shaped pinholes and numerous cracks in the surface layer of the material. The majority of the defects are present in case of solution heat treatment with furnace cooling and are coming into existence in neighborhood to the occurred precipitations, what causes discontinuity of the surface and that fore a significant mass loss. The smallest visible surface layer destruction is characteristic for magnesium cast alloys after ageing.

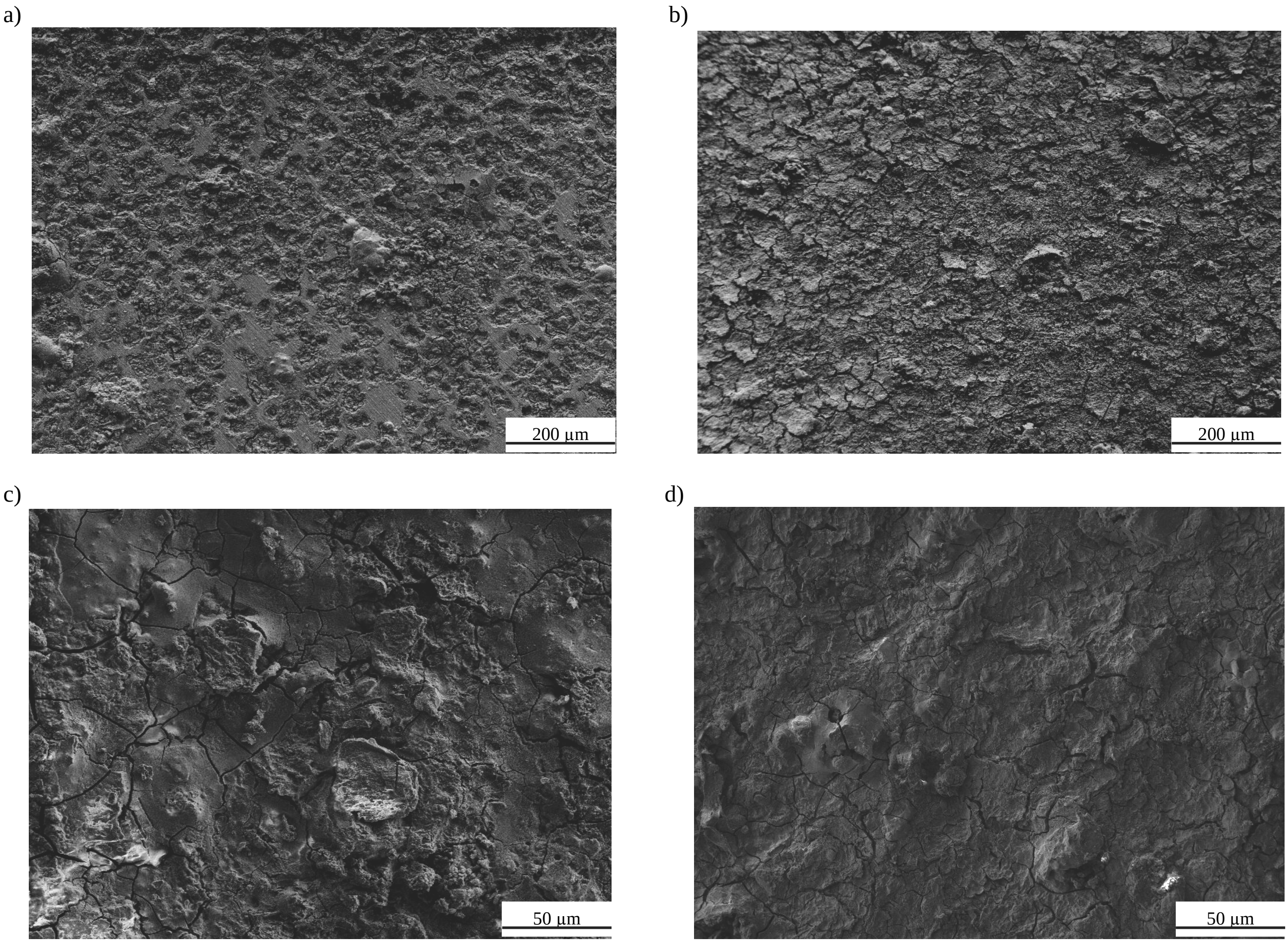


Fig. 5. Microstructure of the magnesium alloy MCMgAl9Zn1: a) in as-cast state, b) after solution heat treatment with cooling in the water, c) after solution heat treatment with cooling in the air, d) after aging treatment after corrosion tests

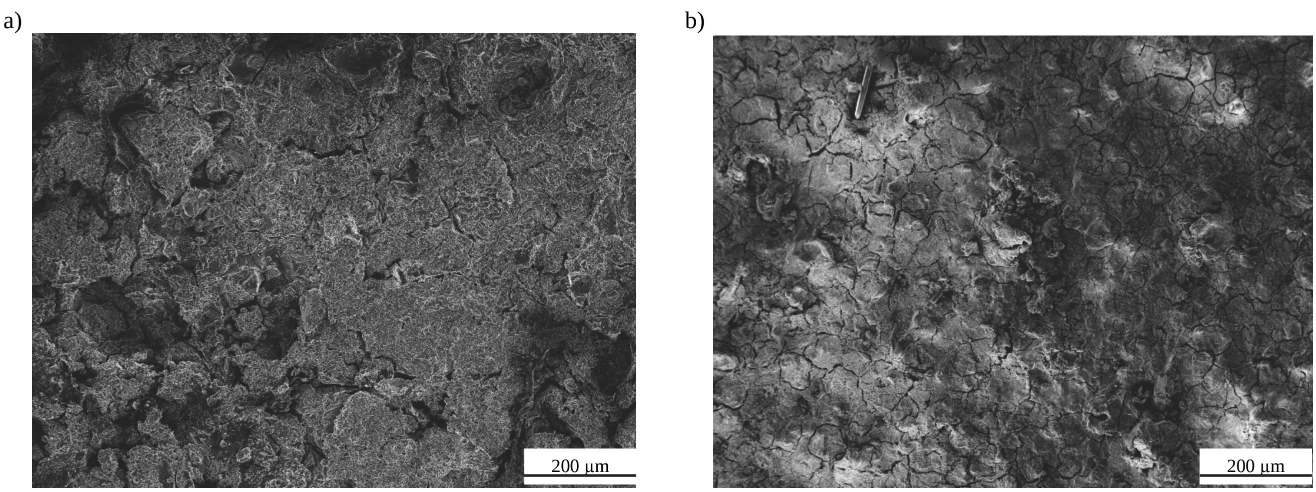


Fig. 6. Microstructure after solution heat treatment with cooling in the furnace after corrosion tests of the magnesium alloy: a) MCMgAl9Zn1, b) MCMgAl6Zn1

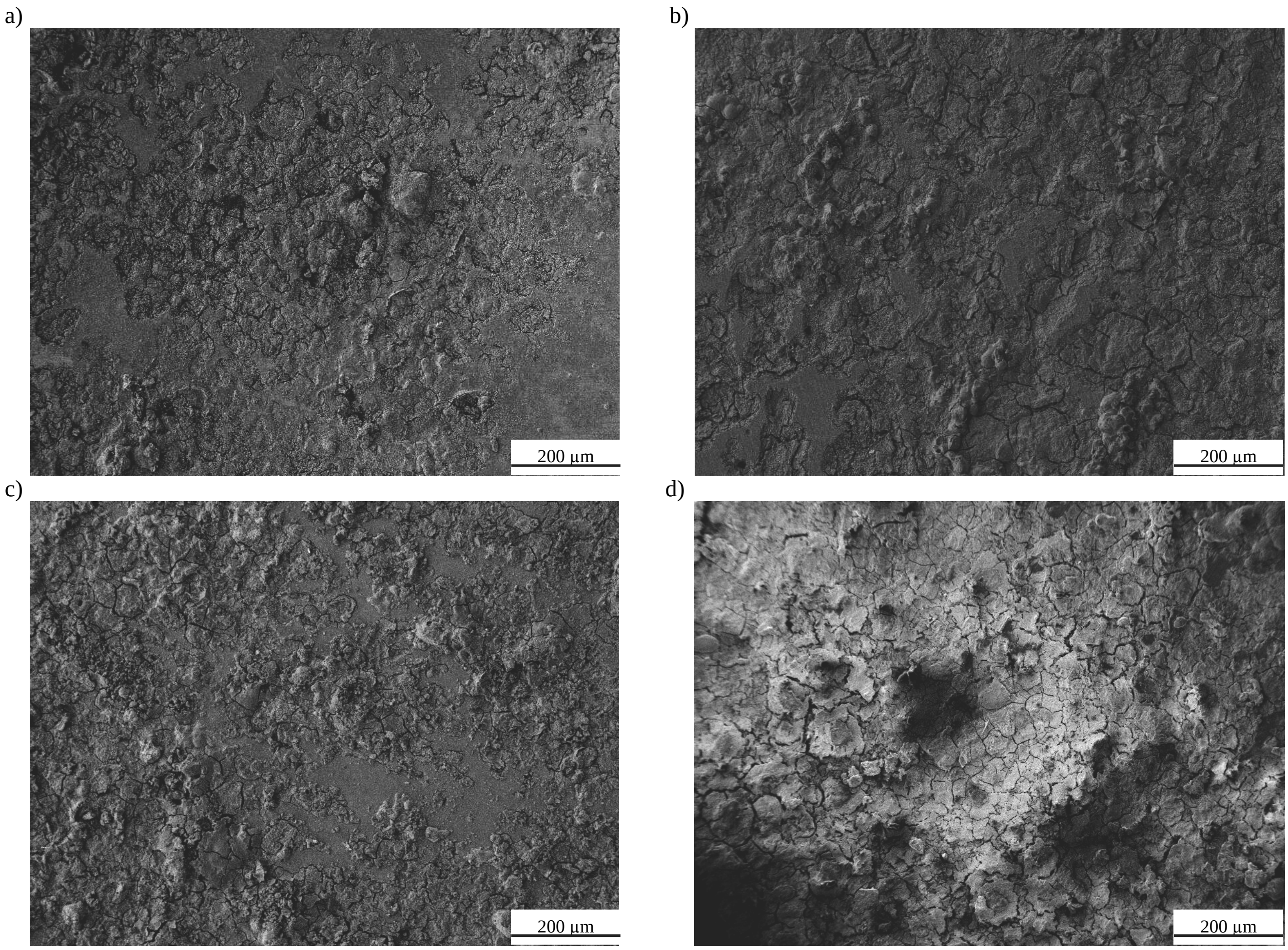


Fig. 7. Microstructure of the magnesium alloy MCMgAl6Zn1: a) in as-cast state, b) after solution heat treatment with cooling in the water, c) after solution heat treatment with cooling in the air, d) after aging treatment after corrosion tests

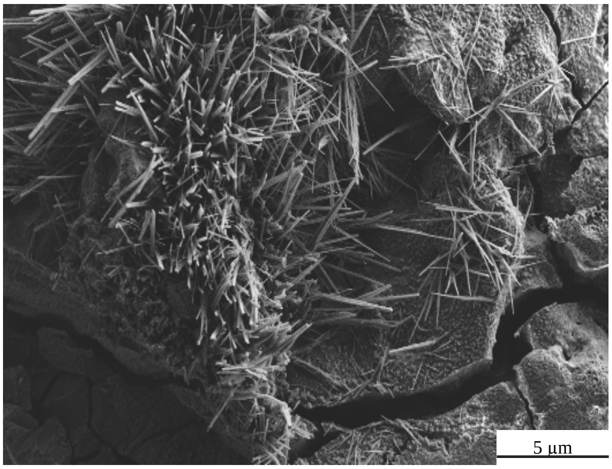


Fig. 8. Microstructure of the magnesium alloy MCMgAl9Zn1 after solution heat treatment with cooling in the air after corrosion tests

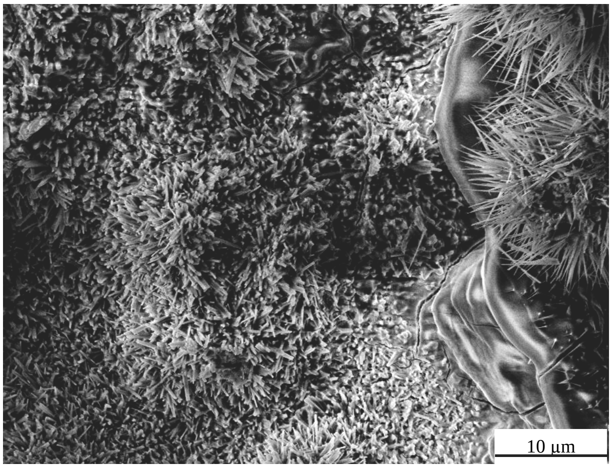


Fig. 9. Microstructure of the magnesium alloy MCMgAl6Zn1 after aging treatment after corrosion tests

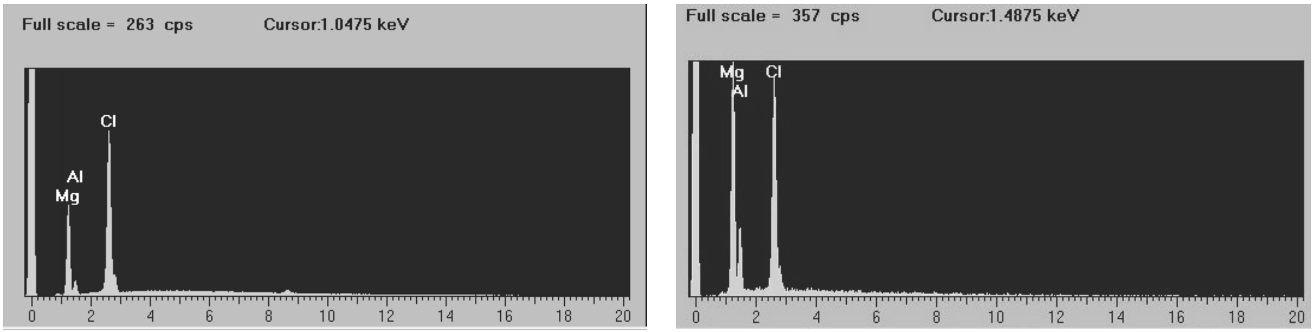


Fig. 10. Spectrum of the point-wise chemical composition analysis from corrosion products area

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