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Corrosion resistance of AZ31 alloy after plastic working in NaCl solutions

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Materials

ABSTRACT

Purpose: The purpose of the study was to assess corrosion resistance of magnesium alloy AZ31 (Mg-Al-Zn alloy) after plastic working in NaCl solutions. It presents currently applied methods of magnesium alloys plastic working. Basic groups of magnesium alloys that are used for plastic working have been discussed.
Design/methodology/approach: Corrosion tests of AZ31 alloy were carried out in solution with concentration of 0.01-2 M NaCl with application of the system for electrochemical tests VoltaLab®PGP201. Resistance to electrochemical corrosion was evaluated on the ground of registered anodic polarisation curves by means of potentiodynamic method. Immersion tests were carried out in NaCl solutions in the time of 1-5 days. Scanning microscopy enabled to present microstructure of AZ31 after immersion tests.
Findings: Results of all carried out tests explicitly prove deterioration of corrosion properties of magnesium alloy AZ31 with the increase in molar concentration of NaCl solution.
Practical implications: It was determined that irrespective of molar concentration of NaCl solution pitting corrosion was found in the tested alloy. It proves that application of protective coating on elements made of the tested alloy is necessary.
Originality/value: Literature gives the results of corrosion tests with reference to cast alloy AZ31. Tests of corrosion resistance of hot rolled AZ31 in chloride solutions have been made for the first time.
Keywords: Metallic alloys; Magnesium alloy AZ31; Plastic forming; Corrosion

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1. Introduction

Magnesium and magnesium alloys thanks to their physical properties, most of all thanks to high relative strength, are used in construction of vehicles, in aeronautic and space technology as well as in electronic industry. Due to good casting properties of magnesium, it is used mainly in cast construction elements. Alloys for plastic working are occasionally used. Tests of material and production process of semi-finished products made of magnesium alloy after plastic working are currently undergoing intensive development [1,2].

Basic alloys for plastic working contain up to 8% Al with addition of Mn (up to 2%), Zn (up to 1.5%, although alloy containing 6% Zn was made), Si (ca. 0.1%) and minute quantities of Cu, Ni, Fe. The following groups of alloys can be distinguished [3]:

- Mg-Mn (M1, M2),
- Mg-Al-Zn (AZ21, AZ31, AZ 61 and AZ80),
- Mg-Zn - (Mn, Cu) (ZM21, ZC71).

Similarly to casting alloys, alloys containing elements (mainly Zn, RE, Y, Zr, Th) can be included in the second group. In this group the following groups of alloys can be distinguished:

- Mg-Zn-Zr (ZK30, ZK40 and ZK 60),
- Mg-Zn- RE (ZE10),
- Mg-Y-RE-Zr (WE43, WE54),
- Mg-Th (HK31, HM21, HZ11).

The third group, which is undergoing intensive tests, may include new ultra-light alloys containing Li of the following types:

- Mg-Li-Al (LA141).

Development of deformable magnesium alloys and methods of their plastic working has been really restricted so far. Magnesium alloys that undergo plastic working are used sporadically, which results from technological problems during plastic working and high costs of production [3].

The essential disadvantage of magnesium and its alloys is very low formability in ambient temperature; it results from the type of crystallographic lattice. In ambient temperature only one slip system in a plane is active (0001). Due to this fact, magnesium alloys can undergo deformation, depending on chemical composition, in the temperature over 200-225°C, when additional slip planes are activated (oblique and tetragonal). Apart from slip in low temperatures in magnesium alloys, a substantial contribution of twinning can be observed. The study [3] proves that in Al-Mg-Zn alloys deformed in the temperature up to 200°C, the structure shows slip bands and deformation twins. The change of deformation mechanisms results in a substantial increase of stress on plastic flow curves obtained in model tests of axially symmetric compression in the temperature below 300°C.

Magnesium features average level of stacking fault energy (78 mJ/m^2). In magnesium alloys stacking fault energy decreases; for example for an alloy with content of 3% Al it equals 27.8 mJ/m^2, whereas when the content increases to 6% Al, energy is decreased to 16.8 mJ/m^2. Together with the increase of temperature, a substantial increase of limiting formability takes place [4]. Decrease of stacking fault energy fosters the process of dynamic recrystallisation and grain size reduction during plastic working. In the temperature up to 200°C in microstructure one can observe the process of twinning and limited recrystallisation. Increasing the temperature to 300°C leads to limited dynamic recovery and to creation of so called recrystallized grain chain, so called „necklace”. Over 300°C, constant dynamic recrystallisation takes place, hence almost double increase in formability. Also deformation rate has the influence on deformation mechanism. Together with increase of deformation rate, the number of twins in the structure increases, whereas decrease in the rate fosters the process of continuous dynamic recrystallisation [3].

Good characteristics of magnesium alloys is obtained thanks to heat and plastic working. As it has been proved, in magnesium alloys intensive dynamic recrystallisation process takes place during plastic working, which enables size reduction and improvement of mechanical characteristics [5]. Plastic working of magnesium and its alloys may be carried out, depending on the content of alloy components, only in a limited range of temperatures. Through heat and plastic working it is possible to obtain grains with average diameter below 10 μm in magnesium alloys. Magnesium alloys, depending on chemical composition and formability, can undergo hot forming by means of the following methods [2,6-12]:

- rolling,
- open die forging and stamping,
- extrusion forging,
- sheet press forming in heated matrices after rolling.

Rolling of ingots made of magnesium alloy is extremely costly and time-consuming due to the necessity of intermediate annealing, thus high price of obtained goods.

At present not only conventional methods, but also those technologies of magnesium sheet production that are still being developed. Magnesium sheet production from ingots is rarely used. Basis stock material is stock in the form of strand cast slabs with thickness of min. 120 mm. The process is carried out in the temperature range of 450-200°C.

Technologies of casting between rollers are in the phase of pre-production tests. Those technologies raise high expectations regarding cost decreasing and quality improvement [2].

At present, magnesium alloy rolling is in principle limited to a few basic grades of the group of Mg-Al-Zn alloys and Mg-Zn-Mn alloys. Also new alloys Mg- Th- (Mn or Zr) and Mg-Li-Al are rollable. Other alloys most often undergo extrusion forging and hot forging. Extrusion forging of magnesium alloys is most frequently carried out within temperature range of 320-450°C at the rate from 1 to 25 m/min. As far as alloy extrusion forging of magnesium alloys is concerned, the method of hydrostatic extrusion has been undergoing intensive development recently. Due to good thermal and mechanical conditions, the process of hydrostatic extrusion can be carried out in lower temperatures and it enables to obtain greater size reduction of magnesium alloys.

Alloy component in magnesium alloys for plastic working can generally be divided into 3 categories [3]:

- additives that increase both the strength and ductility of magnesium are in the order of decreasing strength - Al, Zn, Ca, Ag, Ce, Ni, Cu, Th, and those increasing plasticity - Th, Zn, Ag, Ce, Ca, Al, Ni, Cu,
- elements that increase ductility, without clear impact on magnesium strength are Cd, Tl i Li,
- elements that increase strength but decrease magnesium ductability include Sn, Pb, Bi i Sb.

Below there are descriptions of basic groups of magnesium alloys that can be used in plastic working.

Mg-Mn alloys

Mg-Mn alloys contain max. up to 2.5% Mn. The most popular alloys are M1 and M2 that contain respectively 1% and 2% manganese. These alloys are worked by means of extrusion; M1 alloy also undergoes plastic working by means of rolling. It is suitable for welding. These alloys feature the lowest mechanical properties in the group of magnesium alloys that are subject to plastic working. They feature good corrosion resistance. They are applied on various kinds of cladding and shields as well as on anodes.

Mg-Al-Zn alloys

This group of alloys is currently the most popular one. We can distinguish four basic alloys AZ21, AZ31, AZ61 and AZ80. AZ21 and AZ31 have average mechanical properties, they are weldable, perfect for rolling and extrusion. Those grades are used for production of metal sheets that are used for die-stamping. The process of extrusion is carried out in heated matrices. They are applied as elements of vehicle body construction. AZ61 and AZ80 alloys contain more alloy components and feature better mechanical properties. AZ61 alloy is weldable, it undergoes plastic working by means of extrusion and forging. AZ80 alloy features the highest mechanical properties among plastically worked alloys, whereas its plastic workability is relatively low. It is possible to make straight forgings out of this alloy.

Mg-Zn alloys

In this group of alloys you can distinguish two alloys ZM21 and ZC71. ZM21 contains zinc up to 2% and manganese ca. 1%, ZM21 is prone to rolling and extrusion, it features good weldability. ZC71 is a new alloy of magnesium and zinc, copper and manganese that features high strength up to 360 MPa. It can be formed by means of extrusion and forging, it is weldable.

Mg-Zn-Zr

This group includes alloys containing from 3 to 6% Zn and within the range of 0.4-0.6% Zr. It includes the alloys: ZK30, ZK40 and ZK60. Zirconium addition leads to intensive grain size reduction. These alloys feature high strength. They are formed in the process of forging and extrusion.

Mg-Zn-RE

In this group you can distinguish the basic alloy ZE10 (Mg-RE-Zn) that includes 1.25% Zn and 0.2% od rare earth elements. This alloy has average strength properties, but it features high plasticity.

Mg-Y-Re-Zr (WE43, WE54)

Alloys of WE43 and WE54 type are also formed by means of plastic working, mainly in the process of extrusion. After extrusion and heat treatment, WE43 alloy acquires tensile strength R_m=270 MPa, yield point R_{pe}=195 MPa and elongation A=15%. Moreover, this alloy features good resistance to creep in raised temperatures. WE43 is used in numerous applications, among other things in aeronautics.

Mg - Th (HK31, HM21, HZ11)

Basic alloy component is thorium at the amount from 0.8 to 3.2% and zirconium or manganese, also additions of zinc are used. The effect of hardening is obtained due to dissolution of thorium in solid solution. These alloys feature higher resistance to creep in comparison to WE43 and WE54 alloys (they are resistant up to the temperature of 350°C). HM21 alloy can be rolled whereas HK31 and HZ11 are submitted to extrusion.

Mg-Li alloys

One of the perspective groups of magnesium alloys for plastic forming are magnesium and lithium alloys. Lithium has a positive effect on plastic characteristics of magnesium, but it reduces substantially resistance of the alloy. Increase of plasticity by addition of lithium is obtained through reduction of C/A ratio of hexagonal unit cell of magnesium, due to which additional slip systems are activated more easily. Currently one commercial alloy of Mg-Li with Al and Mn marked as LA141 is known. It is produced in the form of metal sheets.

Application of magnesium alloys is limited, though, due to insufficient resistance to corrosion. Magnesium as a highly electronegative element features extreme susceptibility to passing into electrolyte solutions. It is extremely prone to electrochemical and chemical corrosion, in particular in the environment that contains chloride ions, which substantially limits the area of its alloys application. The reason for low corrosion resistance of magnesium is insufficient protective properties of the layer of oxides that is formed on the surface of in oxidising atmosphere or the layer of hydroxides in water solutions.

Literature gives the results of property (among others corrosion tests) related to cast AZ31 alloy and other magnesium alloys obtained by means of casting method [13-27]. The results of corrosion resistance tests of hot rolled AZ31 in chloride solutions are not known.

The purpose of this study was to evaluate resistance to corrosion of magnesium alloy AZ31 after rolling. Corrosion tests were carried out in NaCl solutions featuring various concentration of chloride ions (0.01-2 M NaCl). Potentiodynamic tests enabled to register anodic polarisation curves. Stern method was applied to determine parameters of corrosion resistance of the alloy.

Immersion tests were carried out in the time from 1-5 days. By means of scanning electron microscope with field emission FE SEM S-4200 Hitachi, in cooperation with spectrometer EDS Voyager 3500 Noran Instruments, qualitative and quantitative analysis of chemical composition in micro-areas was made.

2. Materials and methods

Samples made of magnesium alloy AZ31 - ASTM designation (MCMgAl3Zn1) after hot rolling were used as initial testing material. Chemical composition of the alloy is presented in Table 1. After casting, AZ31 alloy went through annealing in the temperature of 350°C for 1 h, and then cooled with air.

Table 1.
Chemical composition of magnesium alloy AZ31, % of mass

Al	Zn	Mn	Cu	Mg
2.83	0.80	0.37	0.02	rest

The tests were carried out in NaCl with various concentration of chloride ions. Measurements were made in 0.01; 0.2; 0.6; 1 and 2 M NaCl. Solution temperature during the test was 21±1°C.

Resistance to electrochemical corrosion was determined on the ground of registered anodic polarisation curves. For potentiodynamic tests system VoltaLab®PGP201 by Radiometer was used. Saturated calomel electrode (NEK) of KP-113 type served as reference electrode, whereas platinum electrode of PtP-201 type was used as auxiliary electrode. The tests started with determination of opening potential E_{OCP}. Later, anodic polarisation curves were registered, beginning with the measurement of potential with the value of $E_{pocz}= E_{OCP}-100$ mV.

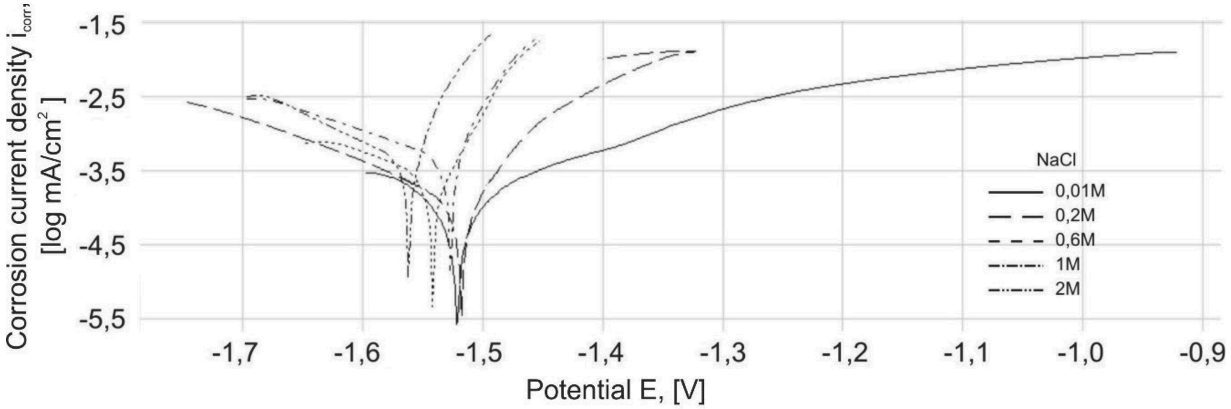


Fig. 1. Anodic polarisation curves registered for rolling samples

Table 2.
Results of electrochemical corrosion resistance tests of magnesium alloy AZ31 (mean measurement values)

Molar concentration NaCl, M	E_{corr}, mV	$I_{corr.}$, A/cm^2	R_p, Ωcm^2	Corr., mm/year
0.01	-1490	0.007	2600	0.260
0.2	-1488	0.023	1100	0.527
0.6	-1538	0.107	281	2.460
1.0	-1537	0.128	210	2.940
2.0	-1566	0.127	208	3.440

Table 3.
Results of the immersion test

NaCl concentration, M	Corrosion rate, mg/(cm^{2}day)		
	1 day	3 day	5 day
0.01	0.007133	0.033287	0.064197
0.2	0.009516	0.052309	0.106995
0.6	0.021399	0.052631	0.149793
1	0.038043	0.095107	0.256788
2	0.066575	0.156926	0.506443

Potential changed in the anodic direction at the rate of 1 mV/s. When anodic current reached density of 10 mA/cm^2, polarisation direction was changed. Thus, return curve was registered. Opening potential E_{OCP} of tested samples steadied after 30 minutes.

On the ground of registered anodic polarisation curves, typical elements describing resistance to electrochemical corrosion were determined, i.e.: corrosion potential, corrosion current density and corrosion rate. Stern method was applied to determine polarisation resistance.

Immersion tests were carried out in ambient temperature in 0.01-2 M NaCl solution in the time of 1-5 days. After specific preparation of the surface of samples they were weighted and mass m_0 was determined. After immersion of the alloy in NaCl solutions during 1-5 days, the samples were taken out and corrosion products were removed of the by means of reagent containing 200 g/l CrO_3 and 10 g/l $AgNO_3$. Next, they were flushed with distilled water, degreased by means of acetone, dried and weighted again, determining mass m_1. These tests enabled to determine corrosion rate.

In the study also tests made by means of scanning microscopy were carried out, which enabled to picture microstructure of AZ31 alloy after immersion tests.

3. Results

Potentiodynamic tests carried out in NaCl solutions with various molar concentration enabled to determine corrosion properties of magnesium alloy AZ31 obtained by means of hot rolling. Corrosion resistance test results (mean values of measurements) are compared in Table 2. Anodic polarisation curves of the selected samples are shown in Fig. 1.

It was determined that with the increase in chloride ions concentration, there is decrease in corrosion potential, polarisation resistance as well as the increase in corrosion current density and corrosion rate. Corrosion potential decreased from -1490 mV to -1566 mV. Together with the increase in molar concentration of the solution, polarisation resistance decreased from 2600 Ωcm^2 to

208 Ωcm^2. Corrosion current density for cast alloys increased from 0.007 A/cm^2 to 0.127 A/cm^2. Corrosion rate increased from 0.26 mm/year to 3.44 mm/year. The quoted corrosion parameters refer to mean measurement values. Table 3 shows the results of the immersion test.

Corrosion rate in the immersion test was determined according to the formula:

$$V = \frac{m_0 - m_1}{St} \qquad (1)$$

where:

V - corrosion rate, mg/(cm^{2}day),
m_0 - sample initial mass,
m_1 - sample mass after immersion test and removal of corrosion products,
S - sample surface area,
t - exposure time (day).

During observations carried out by means of scanning microscopy it was determined that alloy structure consists of solid solution α_{Mg} and individual emissions of Mg-Mn-Al, Mg-Si and Mg_2Si type.

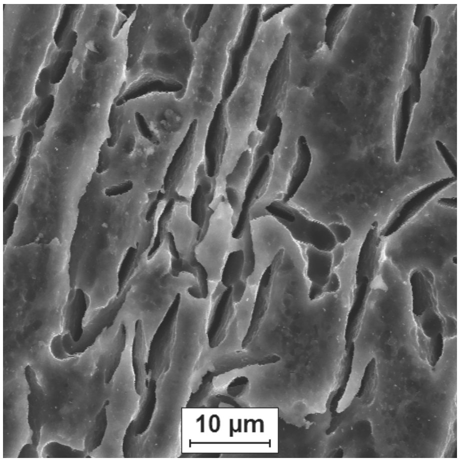


Fig. 2. Pits on the surface of AZ31 (0.01 M NaCl; 1 day)

After 1 day of testing in 0.01 M NaCl solution, corrosion has selective and non-uniform character. One can see pits on the surface of the alloy (Fig. 2). There are also visible undissolved inter-metallic phases of Mg_2Si type (Fig. 3). Fig. 3a shows the results of qualitative and quantitative analysis Mg_2Si phase made of electron scanning microscope FE SEM S-4200 Hitachi in cooperation with spectrometer EDS Voyager 3500 Noran Instruments.

After 3 and 5 days of exposure, successively, increasing number of pits can be observed on the surface of the alloy (Fig. 4). There are still non-corroded phases of Mg-Si and Mg-Mn-Al type (Fig. 5) on the surface. Fig. 5 shows the results of qualitative and quantitative analysis phases.

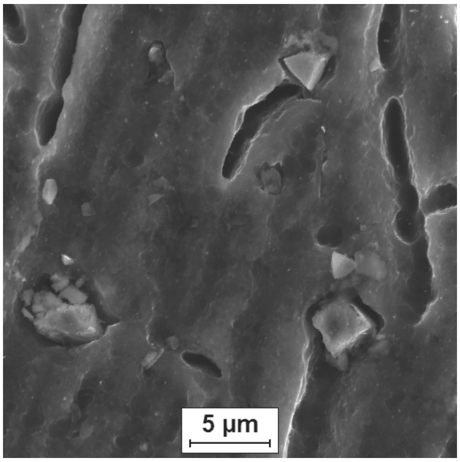
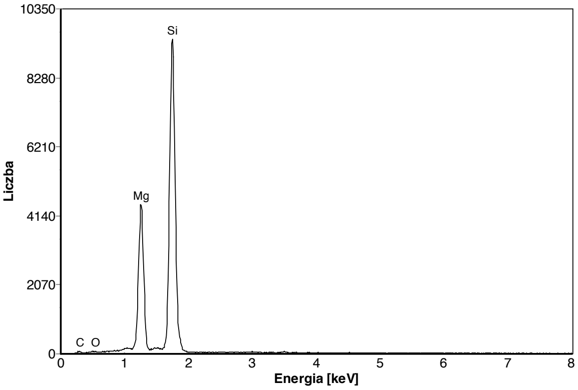


Fig. 3. Phases of Mg_2Si type (0.01 M NaCl; 1 day)



Element	Wt [%]
Si	71.09
Mg	28.91

Fig. 3a. X-rays spectrum and the results of quantitative analysis of the phase Mg_2Si

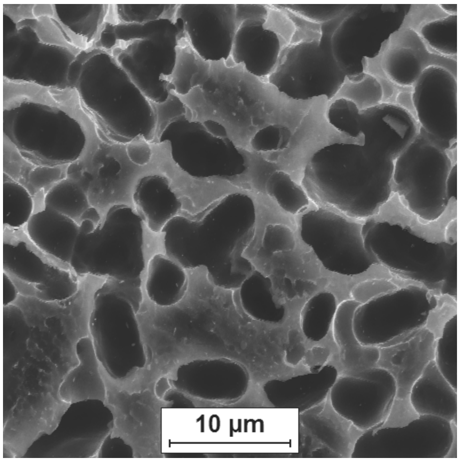


Fig. 4. Pits on the surface of AZ31 (0.01 M NaCl; 3 days)

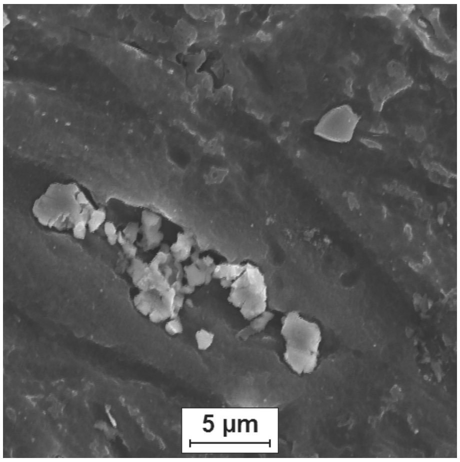
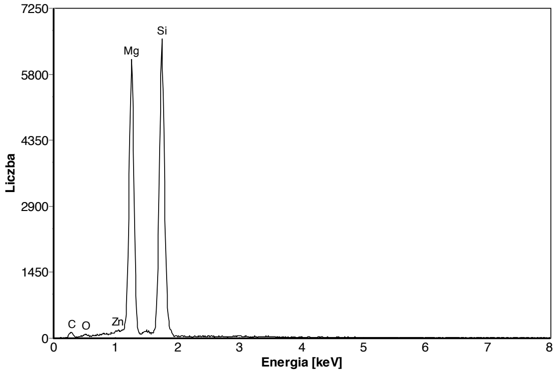
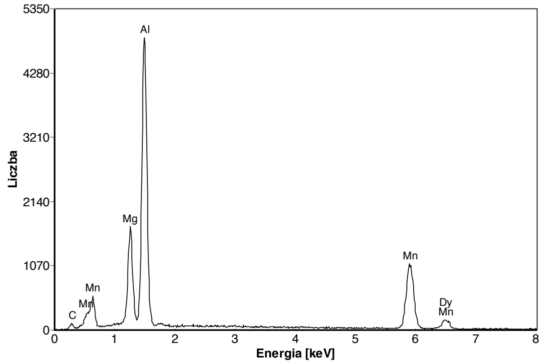


Fig. 5. Phases of Mg-Si and Mg-Mn-Al atype (0.01 M NaCl; 5 days)



Element	Wt [%]
Si	58.03
Mg	41.97



Element	Wt [%]
Mn	29.95
Mg	18.95
Al	51.10

Fig. 5a. X-rays spectrum and the results of quantitative analysis of the phases Mg-Si and Mg-Mn-Al type

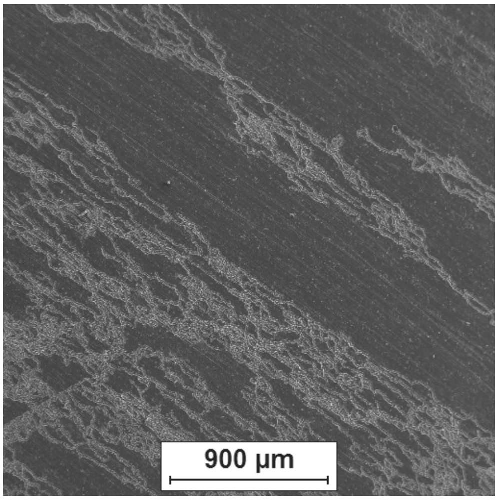


Fig. 6. Surface of the alloy after exposure in 0.6 M NaCl for 3 days

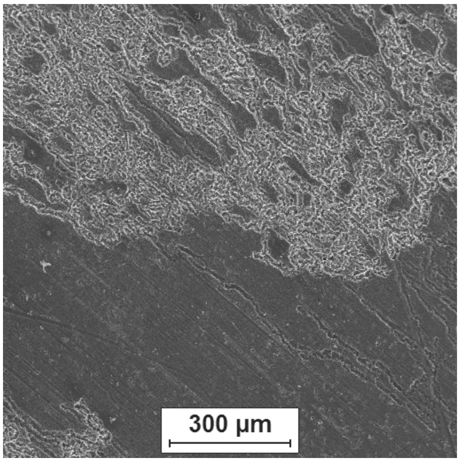


Fig. 7. Surface of the alloy after exposure 0.6 M NaCl (5 days)

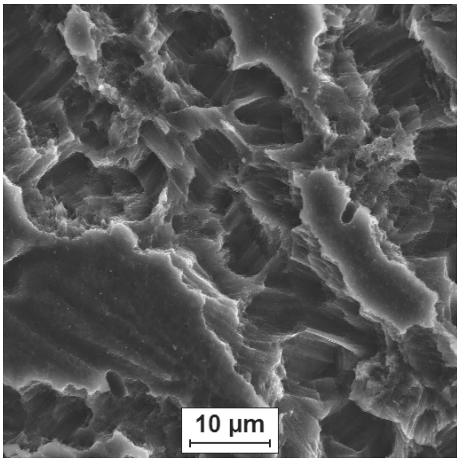


Fig. 8. Pits on the surface of the alloy after tests in 0.6 M NaCl for 3 days

After immersion tests in 0.6 M NaCl, non-uniform course of corrosion can be seen. Tested samples of AZ31 alloy were corroded to a different extent (Figs. 6 and 7). There is a part of the surface that was not corroded.

On the corroded surface the pits feature bigger depth and diameter (Fig. 8).

After the test 2 M NaCl there are only few non-corroded places. Corrosion has a more uniform character (Fig. 9). Large number of pits can be observed on the surface of the alloy (Fig. 10).

At the bottom of pits there are still non-etched inter-metallic phases, which is illustrated in Fig. 11.

With higher magnification, areas with oriented microstructure, connected with plastic strain, can be observed. They are visible after the test both in 0.6 M NaCl (Fig. 12) and in 2 M NaCl (Fig. 13). Due to anisotropy of properties in rolled alloy it is expectable that there is crystallographic direction in which corrosion will proceed with greater intensity.

4. Conclusions

Currently, magnesium alloys that are subject to plastic working are used occasionally. As mentioned before, the main problem in the development of those techniques of magnesium alloy processing is their low plasticity.

By means of thermal and plastic working, grains with average diameter of up to 10 µm can be achieved in magnesium alloys. Grain size reduction below 10 µm can be obtained only by introduction of huge strain. Application of unconventional strain methods enables to obtain size reduction of magnesium alloys up to submicrometric or nanometric size. Therefore such methods of strain support the methods of conventional strain.In magnesium alloys strain processes are realised in elevated temperature. Therefore it is virtually impossible to get nanometric grain size obtained by development of shear band development. Applied methods of big strain, that lead to grain size reduction in magnesium alloys, include most frequently ECAP method or hydrostatic extrusion (HIP). Application of magnesium alloys is substantially limited due to low corrosion resistance, especially in chloride solutions. Hence it was advisable to carry out corrosion tests in solutions that feature a wide range of NaCl solutions concentration. Results of all performed tests explicitly indicate deterioration of corrosion characteristics of magnesium alloy AZ31 with he increase of molar concentration of NaCl solution. Potentiostatic tests performed in solutions with concentration of 0.01-2 M NaCl showed that with the increase of chloride ions concentration you can observe decrease of corrosion potential and polarisation resistance, as well as increase of corrosion current density and corrosion rate of AZ31 aloy. The results of immersion test also show deterioration of corrosion properties of magnesium alloy AZ31 with the increase of molar concentration of the solution. Corrosion rate in the solution with concentration of 0.01 M NaCl increased from 0.007133 mg/(cm^{2}day) after 1 day to 0.064197 mg/(cm^{2}day) after 5 days. Tests carried out in the time of 1 to 5 days in 2 M NaCl solution proved that corrosion rate increased substantially, from 0.066575 mg/(cm^{2}day) up to 0.506443 mg/(cm^{2}day).

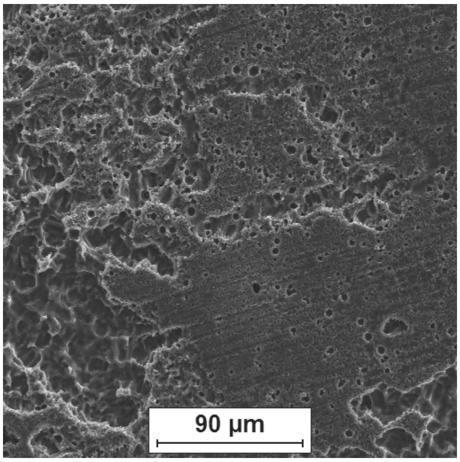


Fig. 9. Surface of the alloy after exposure in 2 M NaCl for 3 days

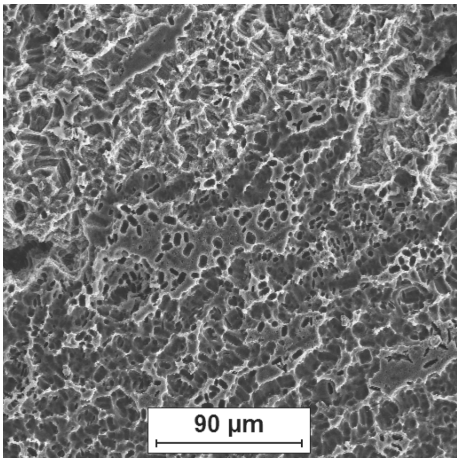


Fig. 10. Surface of the alloy after exposure in 2 M NaCl for 5 days

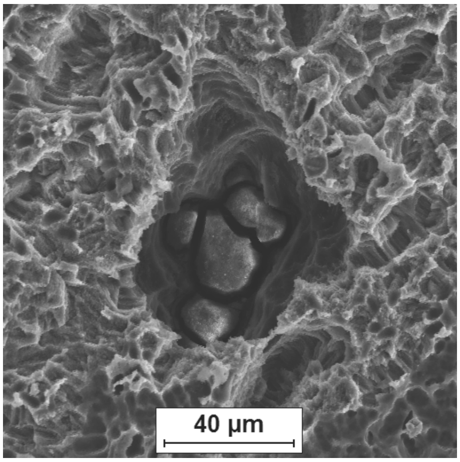
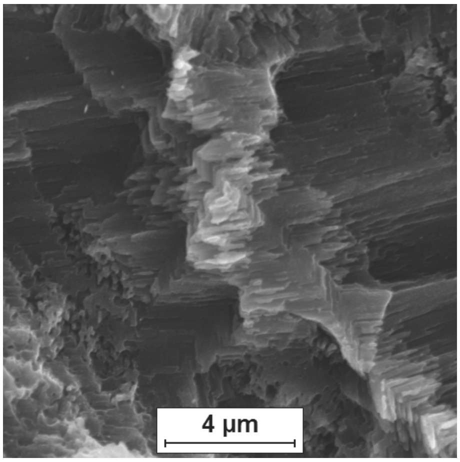


Fig. 11. Inter-metallic phases of Mg-Al type (2 M NaCl, 5 days)

In the structure of the alloy micro-cells are formed between solid solution α_{Mg} and intermetallic phase $Mg_{17}Al_{12}$ (and other phases of Mg-Al type). In low NaCl concentrations corrosion is selective. When the solution is more aggressive, corrosion process intensifies. Anodic solubilization of alloy surface takes place and a large number of pits can be observed. The surface of AZ31 alloy after corrosion tests in 2M NaCl solution is the most corroded one. With the increase of molar concentration of NaCl solution, diameter and depth of the pits is getting bigger. After tests in 0.6 M and 2 M NaCl solution, areas with directed microstructure, connected with plastic strain, can be observed.

a)



b)

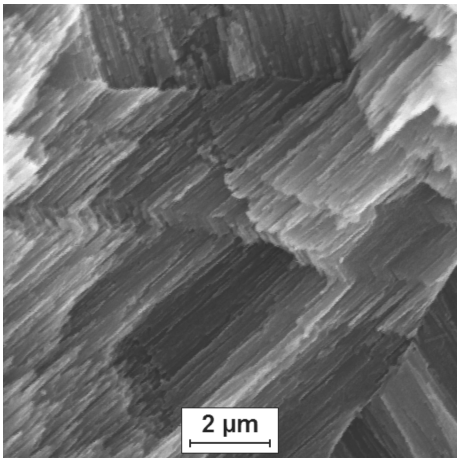
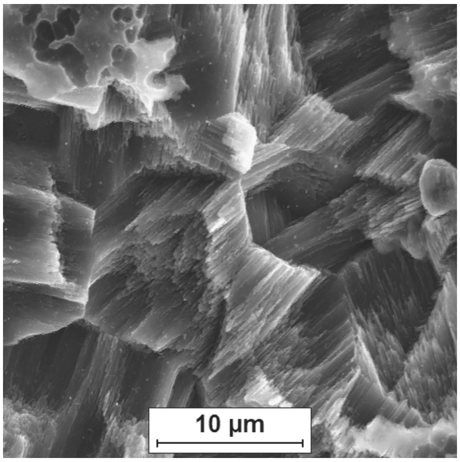


Fig. 12. Areas with directed microstructure: a) 0.6 M NaCl - 3 days, b) 0.6 M NaCl - 5 days

Due to anisotropy of characteristics in rolled alloy it is expected that there is a crystallographic direction in which corrosion will be more intensified. To sum up, it must be highlighted that irrespective of molar concentration of NaCl solution, pitting corrosion si present in the tested alloy. It proves the lack of resistance of magnesium alloy AZ31 after plastic forming to such corrosion type. Test results prove that protective coating on elements made of the tested alloy is necessary [28-32].

a)



b)

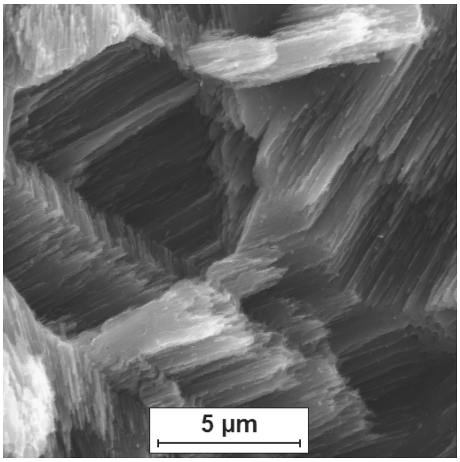


Fig. 13. Areas with directed microstructure: a) 2 M NaCl - 3 days, b) 2 M NaCl - 5 days

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