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RESISTANCE TO ELECTROCHEMICAL CORROSION OF THE EXTRUDED MAGNESIUM ALLOY AZ80 IN NaCl SOLUTIONS

ODPORNOST EKSTRUDIRANE MAGNEZIJEVE ZLITINE AZ80 PROTI ELEKTROKEMIJSKI KOROZIJI V RAZTOPINI NaCl

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The purpose of this study was to evaluate the electrochemical corrosion resistance of the extruded magnesium alloy AZ80 in NaCl solutions. The resistance to electrochemical corrosion was evaluated on the grounds of registered anodic polarisation curves. Potentiodynamic tests were performed in solution with a concentration of 0.01–2.00 M NaCl. In addition, immersion tests were performed, and they allowed us to determine the corrosion rate. Scanning electron microscopy was applied to observe the microstructure after the immersion tests (after removing the corrosion products). Phenomena that happen on the surface of the alloy were evaluated with the application of electrochemical impedance spectroscopy. The tests enabled us to determine the impedance spectra of the system and the data obtained during the measurement was matched to the equivalent system. An optical profilometer was used for the measurement of the geometrical features of the surface of the alloy. The results of the performed tests prove explicitly the deterioration of the corrosion characteristics of the alloy with an increase in the molar concentration of the NaCl solution. A decrease of the corrosion potential and the polarisation resistance was observed, as well as an increase of the corrosion current density. It was proved that irrespective of the concentration, pitting corrosion can be found on the surface of the alloy. The potential to use the extruded magnesium alloy AZ80 in the aircraft and automotive industries is connected with the necessity to apply protective layers on elements made from the tested alloy.

Keywords: extruded magnesium alloy AZ80, electrochemical corrosion, potentiodynamic and immersion tests, SEM, EIS

Namen te študije je bil oceniti odpornost ekstrudirane magnezijeve zlitine AZ80 proti elektrokemijski koroziji v raztopini NaCl. Odpornost proti elektrokemijski koroziji je bila ugotovljena na podlagi anodnih polarizacijskih krivulj. Izvršeni so bili potenciodinamski preizkusi v raztopini s koncentracijo od 0,01–2 M NaCl. Dodatno so bili izvršeni tudi preizkusi s potapljanjem, ki so omogočili določitev hitrosti korozije. Vrstična elektronska mikroskopija je bila uporabljena za slikanje mikrostrukture po potapljanju (po odstranitvi korozijskih produktov). Pojavi na površini zlitine so bili ocenjeni z elektrokemijsko impedančno spektroskopijo. Preizkusi so omogočili določitev impedančnega spektra sistema in z meritvami dobljeni podatki so se ujemali z ekvivalentnim sistemom. Optični profilometer je bil uporabljen za merjenje geometrijskih pojavov na površini zlitine. Rezultati izvršenih preizkusov so potrdili poslabšanje korozijskih lastnosti zlitine pri povečanju molske koncentracije raztopine NaCl. Opaženo je bilo zmanjšanje korozijskega potenciala in polarizacijske odpornosti, kot tudi povečanje gostote korozijskega toka. Dokazano je, da se ne glede na koncentracijo jamičasta korozija pojavi na površini zlitine. Možnosti uporabe ekstrudirane magnezijeve zlitine AZ80 v letalstvu in avtomobilski industriji je povezana z nujnostjo uporabe zaščitnih plasti na komponentah iz preizkušane zlitine.

Ključne besede: ekstrudirana magnezijeva zlitina AZ80, elektrokemijska korozija, potenciodinamski preizkus in preizkus s potapljanjem, SEM, EIS

1 INTRODUCTION

The application of magnesium alloys that can be subject to plastic strain is far less popular than for alloys obtained through casting. This results from a number of technological difficulties during plastic working (which are connected with their low formability at ambient temperature), as well as from high production costs. One of the methods of magnesium-alloy forming is extrusion, which is usually realised within the temperature range 320–450 °C at a rate of 1–25 m/min. Hot isostatic pressing (HIP) has been intensively developing for the last couple of years. Thanks to favourable thermal and mechanical conditions, hot isostatic pressing can be executed at lower temperatures and a larger grain size reduction for the magnesium alloys can be obtained. Equal channel angular pressing (ECAP) is also increasing in popularity[1–5].

The main problem when using magnesium alloys in the aircraft and automotive industries is their susceptibility to electrochemical corrosion. Magnesium as a highly electronegative element that features extreme susceptibility to passing into electrolyte solutions. The standard electrochemical potential of magnesium E_o is –2.37 V, whereas in a 3 % solution of sodium chloride it is –1.63 V (SCE). Magnesium alloys feature good corrosion resistance in weather conditions and when they are put to the reaction of alkaline, chromate and water-fluoric solutions of acids as well as to the majority of organic chemical compounds, e.g., hydrocarbons, aldehydes, alcohols (with the exception of methanol), phenols, amines, esters and most oils. Magnesium is not resistant to the influence of water containing trace elements of heavy-metals ions, sea water, inorganic and organic acids and acid salts (e.g., ammonium), anhydrous methanol, gasoline containing lead (and its compounds), and freon containing water[6–12].

It is extremely prone to electrochemical and chemical corrosion, in particular in an environment that contains chloride ions, which substantially limits the area of this alloy's application. The reason for the low corrosion resistance of magnesium is the insufficient protective properties of the layer of oxides that is formed on the surface in an oxidising atmosphere or the layer of hydroxides in water solutions. Electrochemical corrosion is most often displayed by metal defects on the surface (spots and pits) or by the deterioration of the material's strength[13–19].

The purpose of this study was to evaluate the resistance to electrochemical corrosion of the magnesium alloy AZ80 after extrusion. Corrosion tests were made in NaCl solutions featuring a concentration of chloride ions within the range of 0.01–2.00 M NaCl. Potentiodynamic tests allowed us to register anodic polarisation curves. Immersion tests in NaCl solutions were performed in the time period 1–5 d. A scanning electron microscope served to make images of the AZ80 alloy surface after the corrosion tests. Phenomena that happen on the surface of the alloy were evaluated with the application of electrochemical impedance spectroscopy. The surface morphology after the corrosion tests was evaluated by means of a surface analyser.

2 EXPERIMENTAL

Samples of AZ80 alloy after extrusion served as stock material for the tests. The chemical composition and the mechanical properties of the alloy are presented in **Tables 1** and **2**.

Table 1: Chemical composition of magnesium alloy AZ80 in mass fractions, *w*/%

Tabela 1: Kemijska sestava magnezijeve zlitine AZ80 v masnih deležih, *w*/%

Al	Zn	Si	Mn	Cu	Fe	Mg
8.2	0.34	0.02	0.13	<0.03	0.005	bal.

Table 2: Mechanical characteristics of magnesium alloy AZ80 after extrusion

Tabela 2: Mehanske lastnosti magnezijeve zlitine AZ80 po ekstruziji

R_m/MPa	$R_{p0.2}$/MPa	A/%
343	258	13.5

The resistance to electrochemical corrosion was evaluated on the grounds of registered anodic polarisation curves with the application of the VoltaLabPGP201 testing system by Radiometer. A saturated calomel electrode (NEK) of the KP-113 type served as a reference electrode. A platinum electrode of the PtP-201 type served as an auxiliary electrode. The tests were performed in solutions featuring various molar concentrations of NaCl solution (0.01–2.00 M NaCl). The temperature of the solutions during the test was (21 ± 1) °C.

The immersion tests were performed at ambient temperature with the application of the immersion method in 0.01–2.00 M NaCl solution for 1–5 d. After grinding of the surface of the samples, they were weighed and the mass m_0 was determined. After immersion of the alloy in the NaCl solution for 1–5 d, samples were taken out and the corrosion products were removed in the reagent containing 200 g/L CrO_3 and 10 g/L $AgNO_3$. Next, they were washed with distilled water, degreased with acetone, dried and weighed again with a determination of the mass m_1. The performed tests enabled us to determine the corrosion rate. Potentiodynamic tests and immersion tests were performed for three samples of AZ80 alloy for each concentration of NaCl solution.

A scanning electron microscope with field emission FE SEM S-4200 Hitachi in cooperation with a spectrometer EDS Voyager 3500 Noran Instruments was used to make qualitative and quantitative analyses of the chemical composition in micro-areas. The analysis of the morphology of the AZ80 surface was presented in diagrams and profilographs made with the application of an optical surface analyser MicroProf by FRT.

In order to obtain information about the physical and chemical properties of the surface of the samples made from the AZ61 alloy, tests with the application of electrochemical impedance spectroscopy (EIS) were performed. The measurements were made with the application of an AutoLab PGSTAT 302N measuring system equipped with a FRA2 (Frequency Response Analyser) module. The tests of electrochemical impedance spectroscopy are a linear measurement of the electrical response of the tested metallic material to stimulation with an electromagnetic signal over a wide range of frequencies. The performed tests enabled a direct comparison of the real behaviour of the object with its equivalent system, which is a model that relates to the physically realized impedance.

3 RESULTS AND DISCUSSION

The potentiodynamic tests results are presented in **Table 3**. The anodic polarisation curves are shown in **Figure 1**.

Table 3: Potentiodynamic tests results of AZ80 alloy – mean values

Tabela 3: Rezultati potenciodinamičnih preizkusov zlitine AZ80 – srednje vrednosti

Molar concentration /M*	E_{corr}/mV	I_{corr}/(A/cm^2)	R_p/(Ω cm^2)
0.01	–1434	0.010	2610
0.2	–1540	0.062	427
0.6	–1573	0.093	280
1.0	–1576	0.128	203
2.0	–1593	0.252	104

* M = mol/L (ISO 80000)

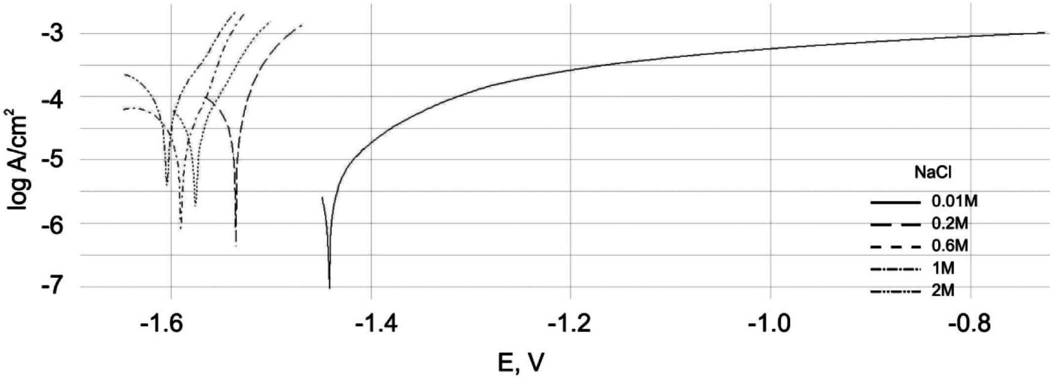


Figure 1: Anodic polarisation curves of AZ80 alloy
Slika 1: Anodna polarizacijska krivulja zlitine AZ80

The tests proved that the corrosion characteristics of the alloy decrease with the increase of the chloride ion concentration. The corrosion potential decreased from E_{corr} = –1434 mV (0.01 M NaCl) to E_{corr} = –1593 mV (2 M NaCl). It was observed that the polarisation resistance decreased from R_p = 610 Ω cm^2 (0.01 M NaCl) to R_p = 104 Ω cm^2 (2 M NaCl). The corrosion current density increased from i_{corr} = 0.01 μA/cm^2 (0.01 M NaCl) to i_{corr} = 0.252 μA/cm^2 (2 M NaCl).

Table 4 presents selected results of the immersion test. The corrosion rate *V* in the immersion test was determined from Equation (1):

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

where *V* is the corrosion rate (mg/(cm^2 d)), m_0 is the initial mass of the sample (mg), m_1 is the sample mass after removal of the corrosion products (mg), *S* is the area (cm^2), and *t* is the exposure time (d).

Table 4: Immersion test results
Tabela 4: Rezultati preizkusov s potapljanjem

Concentration NaCl M	Corrosion rate, V/(mg cm^{-2} d^{-1})	
	1 d	5 d
0.01	0.256	0.343
0.6	1.103	1.295
2.0	2.332	3.850

The results of the immersion tests for the tested alloy confirmed, just as the potentiodynamic tests did, that the alloy was more prone to electrochemical corrosion when the molar concentration of the solution increased. The corrosion rate in a solution with a concentration of 0.01 M NaCl increased from 0.256 mg/(cm^2 d) after the 1st day to 0.343 mg/(cm^2 d) after 5 d. The tests performed in the time period 1 d to 5 d in the 2 M NaCl solution showed that corrosion rate increased from 2.332 mg/(cm^2 d) to 3.85 mg/(cm^2 d).

The results of the tests performed with the scanning microscope FE SEM S-4200 Hitachi are presented in **Figures 2** and **3**.

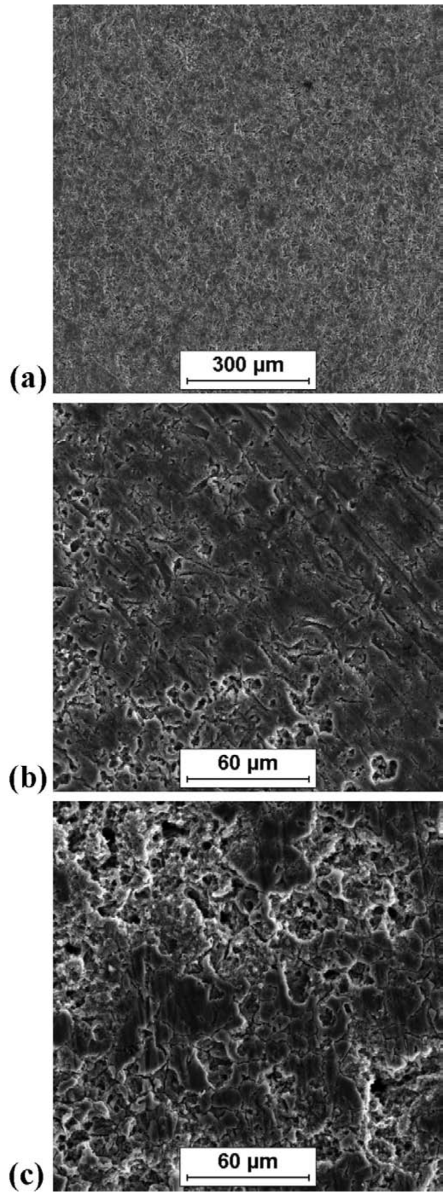


Figure 2: The surface of the alloy after 1 d exposure in the solution with a concentration of: a) 0.01 M, b) 0.6 M and c) 2 M NaCl
Slika 2: Površina zlitine po izpostavi 1 d v raztopini s koncentracijo: a) 0,01 M, b) 0,6 M in c) 2 M NaCl

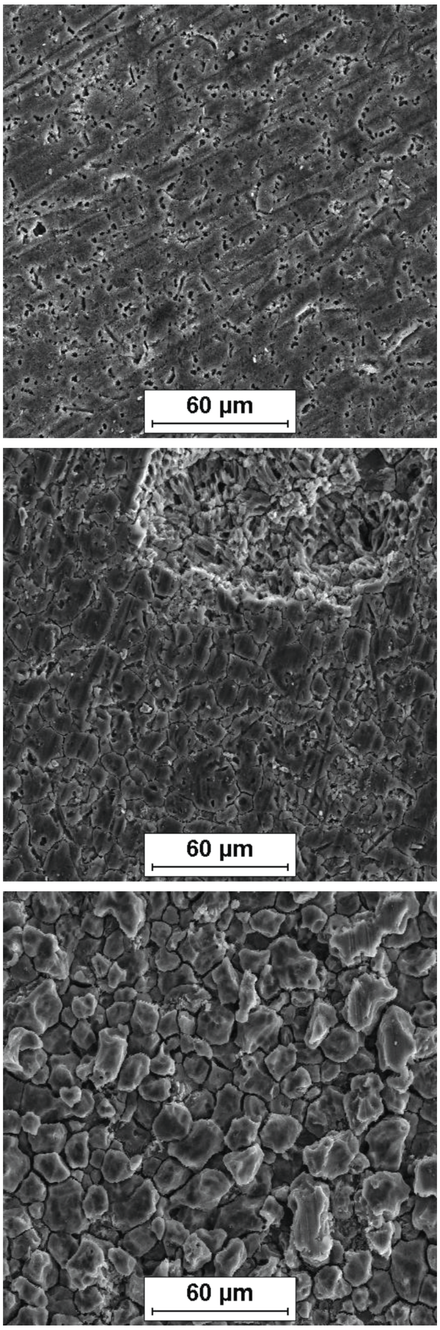
(a)
(b)
(c)


Figure 3: The surface of the alloy after 5 d exposure in the solution with a concentration of: a) 0.01 M, b) 0.6 M and c) 2 M NaCl
Slika 3: Površina zlitine po izpostavi 5 d v raztopini s koncentracijo: a) 0,01 M, b) 0,6 M in c) 2 M NaCl

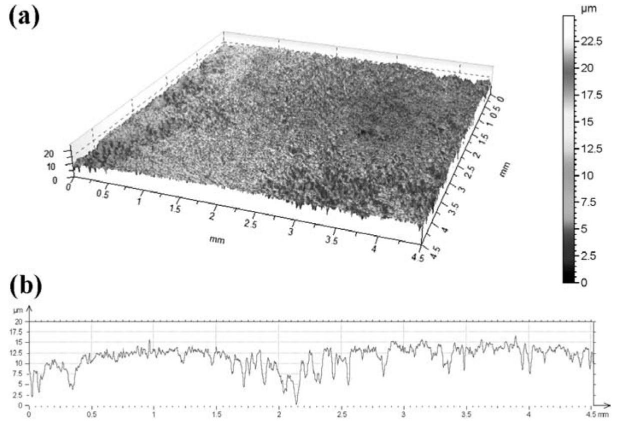


Figure 4: a) 3D image of the surface of the alloy and b) roughness distribution after 5 d exposure in 0.01 M NaCl solution
Slika 4: a) 3D posnetek površine zlitine in b) razporeditev hrapavosti po izpostavitvi 5 d v raztopini 0,01 M NaCl

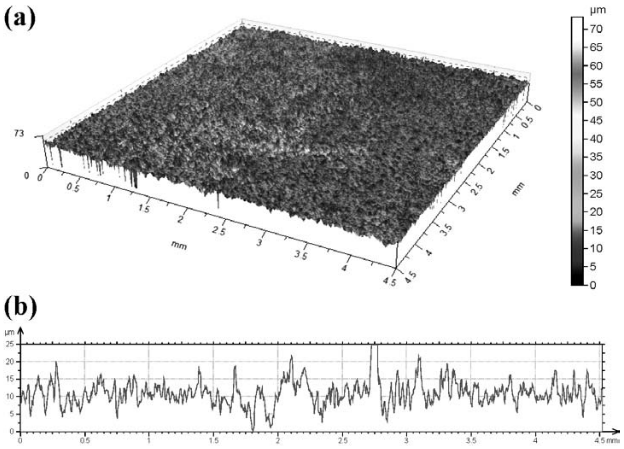


Figure 5: a) 3D image of the surface of the alloy and b) roughness distribution after 5 d exposure in 2 M NaCl solution
Slika 5: a) 3D posnetek površine zlitine in b) razporeditev hrapavosti po izpostavitvi 5 d v raztopini 2 M NaCl

Quantitative and qualitative analyses enabled us to identify the intermetallic phases present in the magnesium alloy AZ80. The presence of phases of the MgAl-, MgMnAl-, and MgAlSi-type was detected.

Figures 4 and **5** present the results of the tests performed with the optical surface analyser MicroProf by FRT, that are illustrated by selected 3D images of the AZ80 alloy and the distribution of the roughness.

Table 5: EIS analysis results
Tabela 5: Rezultati EIS-analize

NaCl concentration, M	R_s/ (kΩ cm^2)	R_f/ (kΩ cm^2)	C_f/ (µF cm^{-2})	CPE_f		C_{dl}/ (µF cm^{-2})	L/ (H cm^{-2})	R_{ct}/ (kΩ cm^2)	R_L/ (kΩ cm^2)
				Y_{01}/ (Ω^{-1} cm^{-2} s^{-n})	n_2				
0.01M	2.46	0.55	14.36	–	–	13.51	0.37e–7	0.21	0.20
0.2 M	0.49	1.10	19.03	–	–	7.99	0.14e–7	0.69	1.28
0.6 M	0.21	0.17	–	0.1572e–4	0.90	480.0	7.13	0.05	0.02
1 M	0.14	0.41	–	0.1475e–4	0.89	129.8	35.13	0.16	0.03
2 M	0.08	0.01	–	0.1652e–4	0.91	526.0	5.19	0.03	0.01

It was proved that with an increase of both the exposure time and the NaCl solution concentration the roughness parameters of the AZ80 deteriorated substantially. For instance, the average arithmetic deviation of the roughness profile R_a increases when the concentration is 0.01 M NaCl, from 0.745 μm (1 d) to 1.25 μm (5 d), and when the concentration is 2 M NaCl, from 0.944 μm (1 d) to 3.3 μm (5 d). The maximum height of the roughness profile R_z for the same concentrations 0.01 and 2 M increases from 4.89 μm (1 d) to 8.41 μm (5 d) and from 6.4 μm (1 d) even to 37 μm (5 d).

The results of the electrochemical impedance tests of the AZ80 alloy are presented in **Table 5**.

The obtained diagrams enabled us to match equivalent systems that are physical models depicting phenomena taking place in the respective object. It was proved that the best matching of the experimental impedance spectra is obtained with the application of an equivalent electrical system consisting of:

- For samples exposed to 0.01 M and 0.2 M NaCl solution from two consecutive parallel systems: within the range of low frequencies from the parallel capacitance system connected with the resistance of ion transition through phase boundary: metal – solution R_{ct}, resistance R_L together with coil (metallic conductor with electromagnetic induction) L representing the corrosion processes; within the range of medium frequencies from the parallel capacitance system C_f connected with the resistance of transition of the R_f ions placed on the surface of the alloy in the result of corrosion (the layer consisting of corrosion products) and resistance at high frequencies, which may be attributed to the resistance of the electrolyte R_s (**Table 5**).

In **Figure 6** R_f and C_f designate, respectively, the resistance and capacitance of the layer created as the result of corrosion (corrosion product layer). R_{ct} indicates the resistance of the charge transition and C_{dl} the capacitance of the double (porous) layer, then R_L and L – induction loop, implicating the initiation and development of the pitting corrosion process.

The mathematical model of the impedance for the system: AZ80 alloy – double layer –NaCl solution (0.01 M and 0.2 M) is presented by Equation (2):

$$Z = R_S + \frac{1}{1/R_f + (j\varpi)C_f} + \frac{1}{1/R_{ct} + (j\varpi)C_{dl} + 1/R_L - (j\varpi)L} \qquad (2)$$

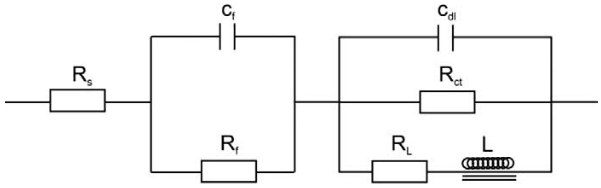


Figure 6: Physical model of equivalent electrical system
Slika 6: Fizikalni model enakovrednega električnega sistema

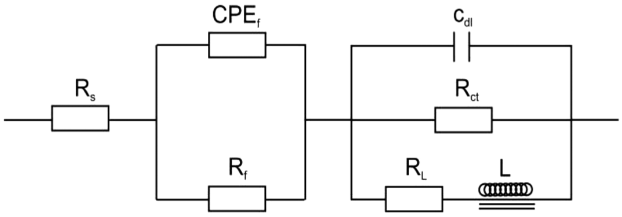


Figure 7: Physical model of equivalent electrical system of corrosion system metal – solution
Slika 7: Fizikalni model električnega sistema, enakovrednega korozijskemu sistemu kovina – raztopina

- For samples exposed to a solution from 0.6 M to 2 M NaCl within the range of low frequencies from a parallel capacitance system connected with the resistance of ion transition through the phase-boundary metal – solution R_{ct}, resistance R_L with the coil (metallic conductor with electromagnetic induction) L representing corrosion processes; within the range of medium frequencies from the parallel system of CPE_f connected with the resistance of transition of the R_f ions placed on the surface of the alloy as the result of corrosion (corrosion product layer) and the resistance at high frequencies that may be attributed to the resistance of electrolyte R_s (**Table 5**).

In **Figure 7** CPE_f indicates CPE depicting the character of the layer created as the result of the corrosion (corrosion product layer), R_f indicates, respectively, the resistance of that layer, whereas R_{ct} resistance of ion transition, and C_{dl} capacitance of double (porous) layer, when R_L and L – induction loop, implicating the initiation and development of the pitting corrosion process.

The mathematical model of the impedance for the system: AZ80 alloy – double layer –NaCl solution (0.6 M, 1 M and 2 M) is presented by Equation (3):

$$Z = R_S + \frac{1}{1/R_f + Y_0 (j\varpi)^n} + \frac{1}{1/R_{ct} + (j\varpi)C_{dl} + 1/R_L - (j\varpi)L} \qquad (3)$$

4 CONCLUSIONS

It is anticipated that the application of magnesium alloys in future years will be systematically increasing and more and more machine parts and units will be made from that group of materials. Their advantage is the fact that they can be formed with the application of casting as well as plastic working.

The application of the magnesium alloy AZ80 after plastic working is dependent to a large extent on its resistance to electrochemical corrosion. The results of the performed tests prove explicitly the deterioration of the corrosion characteristics of the alloy with an increase of the molar concentration of the NaCl solution. Potentiodynamic tests performed in solutions with concentrations of 0.01–2.00 M NaCl showed that with an increase in the chloride ions concentration, a decrease of the corrosion potential and polarisation resistance, as well as an increase of the corrosion current density of the alloy can be observed. The deterioration of the corrosion characteristics with an increase of the NaCl solution concentration was also confirmed by immersion tests and during the metallographic tests.

Microscopic tests of the samples made of the AZ80 alloy enable us to observe corrosion pits at each stage of the test. Within the early stage of pitting, pits were selectively located in the areas where non-metallic precipitates or inclusions were present (**Figures 2a** and **3a**). The effect of the internal galvanic corrosion was evidenced. The secondary-phase particles were preferentially and uniformly corroded, while the α phase was being obviously unattacked. During the development of corrosion, galvanic corrosion should thus have been less important and the increased corrosion attack of the whole structure was noticed. Significant degradation of the grain boundaries (**Figure 3c**) and the existence of crevices (**Figures 2c** and **3b**) were observed.

Deep anodic etching of the micrstructure and corresponding roughness of the samples were observed with an increase of the exposure time.

The performed EIS tests enabled a direct comparison of the behaviour of the real object with its equivalent system, i.e., an electrical model related to physically realised impedance.

To sum up, it must be highlighted that pitting corrosion is present in the tested magnesium alloy. This proves that the extruded magnesium alloy AZ80 is not resistant to that type of corrosion. The prospects for its application in the aircraft industry trigger the need for covering elements made of the tested alloy with protective coatings.

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