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EVALUATION OF ALLOYING EFFECTS ON THE MECHANICAL AND CORROSION PROPERTIES OF MAGNESIUM AND ITS ALLOYS FOR BIOMEDICAL APPLICATIONS

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Abstract: Magnesium alloys and their derivatives are considered promising materials for the future of implantology. The study investigates the influence of alloying elements on the mechanical and corrosion properties of magnesium alloys for materials intended for biomedical engineering. The research focuses on defining key concepts such as biocompatibility, biodegradability, and the criteria that determine the suitability of magnesium and its alloys as biomaterials for implants. The results demonstrate that the properties of magnesium alloys can be significantly improved by selecting appropriate alloying elements. In our case, we focused on alloying elements such as aluminium, zinc, and rare earth elements. Five samples from each main group of magnesium alloys, AZ (aluminum–zinc) and AE (aluminum–rare earth element), underwent a series of mechanical tests, including Vickers hardness measurements, as well as electrochemical analysis, specifically potentiodynamic polarization and electrochemical impedance spectroscopy. The behaviour of magnesium and its alloys was analysed in the work and experimentally verified in the simulated environment of the human body. Among the tested materials, the AZ91 alloy exhibited the most favorable mechanical and corrosion properties, confirmed by mechanical, potentiodynamic, and electrochemical tests. AZ91 showed a relatively positive corrosion potential, low corrosion current density, and a low corrosion rate. The aluminum alloying element in the AZ91 contributes to achieving higher strength of the alloy. However, the high percentage of aluminum remains a problem with the AZ91 alloy, which, in the long term, damages the functioning of processes in the organism. The findings also revealed that the addition of rare earth elements in AE alloys reduces their hardness. Future investigations will concentrate on finding alloying elements capable of improving the mechanical integrity and corrosion resistance of magnesium alloys while ensuring biocompatibility.

Keywords: biocompatibility, magnesium alloys, corrosion, electrochemical methods, mechanical tests

1. INTRODUCTION

Magnesium is an indispensable element in both nature and the human body. Due to its high chemical reactivity, it rarely occurs in its elemental form in nature. Instead, it is predominantly found in compound forms, most commonly as silicates, carbonates, and chlorides, in which it exhibits an oxidation state of Mg^{2+}. Furthermore, it represents an important component of seawater. As an element, magnesium is an integral part of the human body structure. It supports enzymatic activity, proper muscle and nerve function, and participates in numerous biochemical reactions within the organism.

Owing to its exceptional properties such as biocompatibility, low density, and light weight, magnesium has, for many years, attracted the attention of biomedical engineers and implant manufacturers as promising material to produce biodegradable intra-body implants.

However, magnesium also has several drawbacks that limit its medical applications. It shows low corrosion resistance, especially in environments containing Cl^- ions; therefore, its degradation in body fluids would occur too rapidly. During corrosion, a significant amount of hydrogen gas is released, which could adversely affect the surrounding environment of the biomaterial.

The improvement of magnesium and its alloys have been the subject of extensive research for many years. One of the approaches to tailoring material properties is alloying. Through the appropriate selection of alloying elements, both mechanical and corrosion properties of magnesium can be optimized. Before choosing suitable alloying elements, it is essential to evaluate the mechanical and corrosion behavior of magnesium alloys, for instance, in a simulated human body environment.

Mechanical behavior is commonly assessed through tensile testing and hardness measurements, which strongly correlate with each other. One of the most frequently used methods is the Vickers hardness test. For corrosion resistance evaluation, potentiodynamic polarization and electrochemical impedance spectroscopy (EIS) are typically applied. The latter technique is particularly useful for simulating the behavior of material in conductive environments, such as human body fluids. By combining mechanical and electrochemical testing methods, valuable insights into the corrosion behavior of magnesium can be obtained.

The study investigates both commercially pure magnesium and commonly used AZ and AE magnesium alloys. However, these alloys have not yet been sufficiently discussed as potential biomaterials. The aim of the study is to evaluate the influence of the type and content of alloying elements on the mechanical and corrosion behavior of magnesium in a simulated physiological environment and ultimately to identify the most suitable alloy for biomedical applications. The discovery of optimal combination of magnesium with other alloying metals could significantly enhance the use of this element in biomedicine and replace some less suitable materials used for manufacturing implants, stents, screws, and similar medical devices. (Palček, et al., 2015) (Davis, 2001) (Kennedy et al., 2022) (Thomas, et al., 2023)

2. METHODOLOGY OF RESEARCH

Five material samples were used for experimental investigations, commercially pure magnesium Mg 99.9 and the alloys AZ31, AZ63, AZ91, and AE21. The standardized chemical compositions of these alloys are presented in Table 1.

Table 1
Chemical composition of experimental samples from magnesium alloys (wt.%)

Content [wt.%]				
Alloy	**Al**	**Zn**	**RE**	**Mn**
AZ31	3	1	-	0.3
AZ63	6	3	-	0.3
AZ91	9	1	-	0.3
AE21	2	-	1	0.3

Source: (Gu, et al., 2009)

Static Vickers hardness testing was performed on all samples. Prior to the hardness measurement, each of the five samples was ground using abrasive paper with grit the size of P4000, subsequently cleaned with isopropanol, and dried using an air stream. This surface preparation was carried out to remove undesirable oxides and corrosion products from the material surface and to ensure that the surface irregularities would not affect the accuracy of the measurement.

The hardness measurements were conducted using an INNOVATEST FALCON 450 tester. The indenter used was a square-based diamond pyramid with an apex angle of 136°. The samples were placed on the tester platform in a defined sequence, and the testing load and dwell time (30 seconds) were selected.

Once the measurement was initiated, the indenter approached the sample surface until contact was established, after which the full load was gradually applied. After maintaining the load for 30 seconds, the indenter was retracted. Using the built-in optical microscope, the diagonals of the indentation were measured, and the system automatically calculated the Vickers hardness value. (Skočovský, et al., 2013)

Before corrosion testing, all samples were ground using P1200 grit abrasive paper, rinsed with distilled water and ethanol, and subsequently dried. The 0.9% NaCl solution (physiological saline) was replaced before each measurement. The solution was prepared by dissolving 9 g NaCl in 1 L of distilled water.

The corrosion measurements were carried out using a BIOLOGIC SP300 laboratory potentiostat. A three-electrode configuration was employed, in which the tested material sample served as the working electrode (WE), a platinum mesh was used as the counter electrode (CE), and a saturated calomel electrode (SCE) functioned as the reference electrode. The configuration of electrodes is presented in Figure 1.

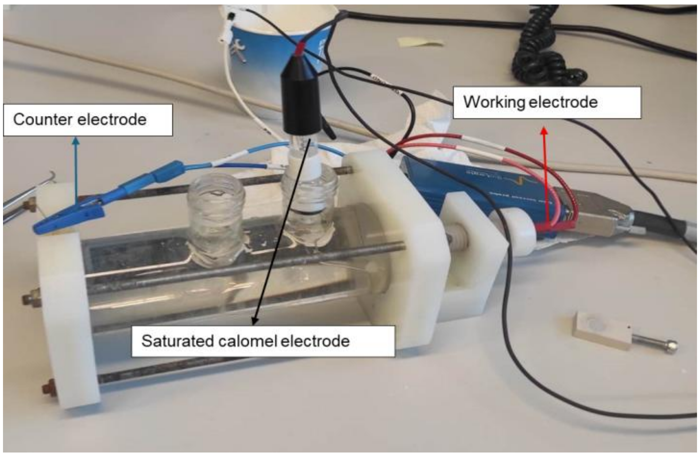

Fig. 1. Experimental setup for potentiodynamic polarization and electrochemical impedance spectroscopy (EIS)

Both electrochemical techniques were performed in a 0.9% NaCl solution at a laboratory temperature of 22 ± 2°C. The stabilization time of the open-circuit potential was set to 10 minutes prior to each measurement.
The sequence of the corrosion testing procedure was as follows: potential stabilization, electrochemical impedance spectroscopy (EIS) and potentiodynamic polarization tests.
The potential range during the potentiodynamic polarization tests was set from -200mV to +300 mV with respect to the corrosion potential. The scan rate was 1 mV.s^{-1}. The resulting potentiodynamic polarization curves were analyzed using Tafel extrapolation, as illustrated in Figure 2. From this analysis, the corrosion potential (E_{corr}), corrosion current density (i_{corr}), and the anodic (β_a) and cathodic (β_c) Tafel slopes were determined, and the corrosion rate (v_{corr}) was subsequently calculated. (Diaz-Ramos, et al., 2023) (Polmear, 2006)

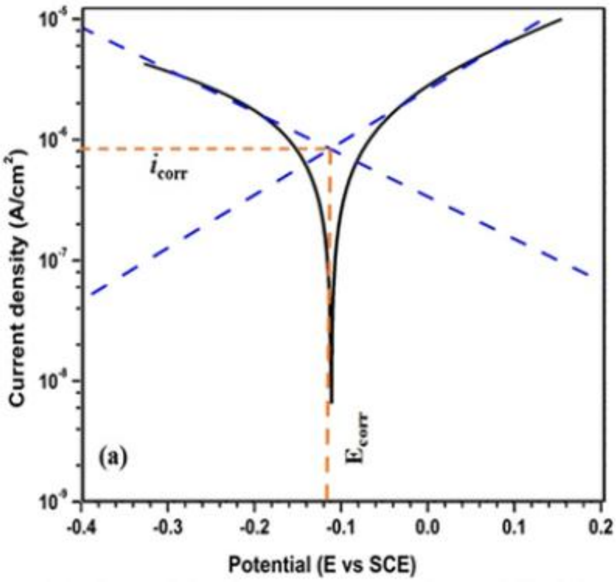

Fig. 2. Tafel analysis of potentiodynamic curve (Bansod, et al., 2018)

Electrochemical impedance spectroscopy (EIS) is a widely applied experimental technique used to determine the electrical properties of electrochemical systems. It is employed to describe the interfacial behavior between metals and various environments, particularly in the study of corrosion processes and material compatibility. In this experiment, constant potential was applied to the working electrode with a superimposed alternating voltage. The alternating component of the current response is measured next. The experiment was conducted over a defined frequency range, typically proceeding from high to low frequencies. The result of EIS is electrochemical impedance, representing the resistance of the system alternating current. Since the resistance varies with frequency, it is referred to as impedance (Z). Impedance characterizes the relationship between the amplitudes of voltage and current as well as their mutual phase shift.
The measured data can be represented in the form of Nyquist or Bode plots. In Nyquist plots, the data are plotted in coordinates of the real part of impedance$(+Z_{Re})$, against the negative imaginary part of impedance $(-Z_{Im})$.
For the analysis of the EIS results, the equivalent circuit method was implemented. The configuration of the equivalent circuit is shown in Figure 3. The simplest equivalent circuit is the Randles circuit, which was used for the AE 21 sample. The individual elements of

the circuit include: R_S, representing the resistance of the electrolyte and CPE (Constant phase element) which simulates surface inhomogeneity of the electrode.
The CPE element replaces the ideal capacitor in the circuit that characterizes the double-layer capacitance. The use of CPE instead of an optimal capacitor is justified by the non-homogeneous nature of the electrode surface and the double layer which causes deviations from ideal capacitive behavior. (Hadzima and Liptáková, 2008) (Kajánek, et al., 2019)

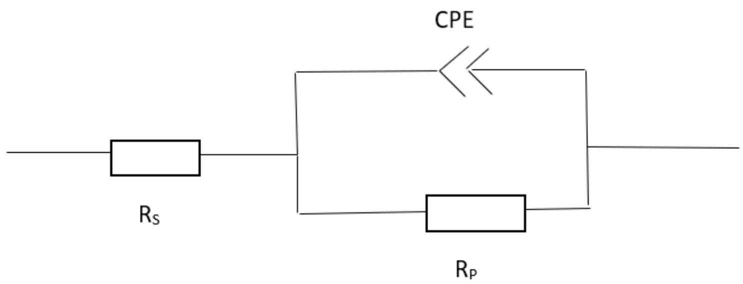

Fig. 1. The Randles equivalent circuit

The R_P element, also referred to as the polarization resistance, is the most significant parameter from the corrosion standpoint. It represents the charge transfer resistance at the sample-electrolyte interface. Its value corresponds to the diameter of the semicircle observed in the Nyquist plot.
A simple Randles equivalent circuit was used due to the presence of a single semicircle in the Nyquist diagram. Figure 4 shows the Nyquist plot with applied Randles equivalent circuit.

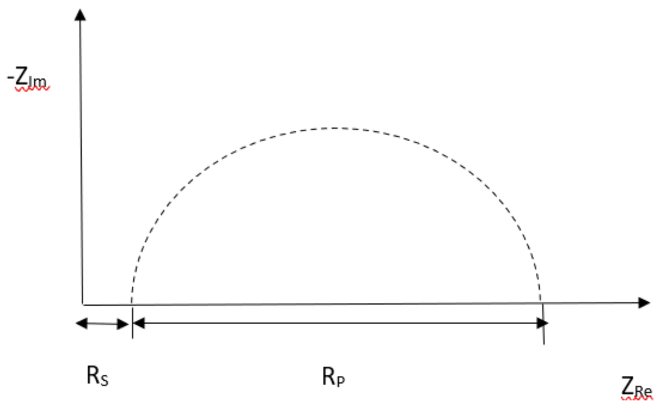

Fig. 4. The Nyquist diagram with Randles circuit

In the experiment, the resulting resistance R_P was also referred to as R_{Total}. With increasing R_P values, the corrosion resistance of the tested metal also increases.
For the analysis of most samples, a more complex equivalent circuit was required, as shown in Figure 5. This circuit consisted of the R_P element, two CPE elements, and the R_{P1} and R_{P2} elements the sum of which represents R_{Total} (R_P). The R_{P1} element can be interpreted as the resistance of the corrosion product layer formed at the metal-physiological solution interface, while R_{P2} represents the charge transfer resistance of the remaining double layer.

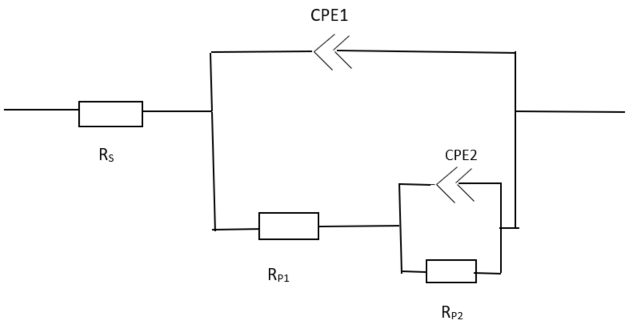

Fig.5. Modified Randles equivalent circuit

A more complex equivalent circuit was applied due to the presence of two semicircles in the Nyquist plots. The representation of the Nyquist plot with complex equivalent circuit is shown in Figure 6. (Kajánek, et al., 2019)

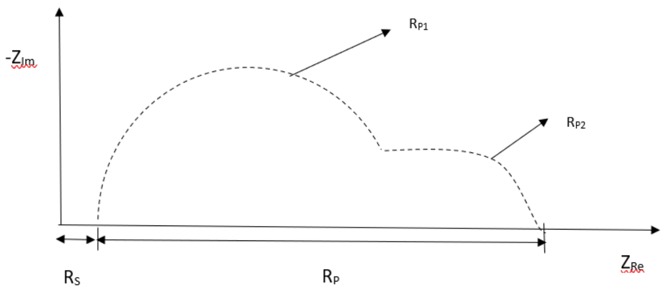

Fig.6. The Nyquist diagram with complex equivalent circuit

The frequency range measured during the EIS method was from 100 kHz to 10 mHz. The frequency varied ten times per decade, and the amplitude of the sinusoidal signal was 10 mV. The obtained Nyquist plots were analyzed using the equivalent circuit method in the EC-Lab v11.02 software.

3. RESULTS AND DISCUSSION

The measured hardness values of the individual samples are summarized in Table 2.

Table 2
Measured hardness values with average hardness

Material	Test load/dwell time [kp/s]	Measured hardness values [HV]	Average hardness [HV]
Mg 99.9	5/30	22.9 HV	23.8 HV
		26.7 HV	
		21.8 HV	
AZ31	5/30	42.7 HV	43.7 HV
		43.6 HV	
		44.7 HV	
AZ63	5/30	52.9 HV	56.4 HV
		59.9 HV	

		56.4 HV	
AZ91	5/30	59.01 HV	61.1 HV
		61.5 HV	
		62.8 HV	
AE21	5/30	56.9 HV	57.06 HV
		57.3 HV	
		56.98 HV	

Based on the measured hardness values and their averages, it can be observed that the highest hardness was observed for the AZ91 alloy, whereas the lowest value was recorded for the commercially pure magnesium (Mg 99.9) sample. A relatively high hardness value was also achieved by the AE21 alloy.

According to the results of the potentiodynamic tests, the most positive corrosion potential (E_{corr}) was observed for the AZ63 alloy. The corrosion potential characterizes the thermodynamic tendency of corrosion, and more positive values indicate a higher nobility of the metal. The lowest corrosion current density (i_{corr}) was recorded for the AZ 91 alloy. The corrosion current density reflects the kinetics of the corrosion process. Furthermore, in evaluating the corrosion resistance of the material, attention is also paid to the corrosion rate (v_{corr}). The lowest corrosion rate was obtained for the AZ91 sample. The results from the potentiodynamic polarization are presented in Table 3.

Table 3
Measured results from the potentiodynamic polarization

Material	E_{corr} [mV]	i_{corr} [mA/cm^2]	β_a [mV]	β_c [mV]	v_{corr} [mm/year]
Mg 99.9	-1744	12.924	142	206	0.295
AZ31	-1584	9.034	64	139	0.206
AZ63	-1496	7.6	46	174	0.17
AZ91	-1520	7.041	52	188	0.161
AE21	-1533	20.550	72	139	0.407

When assessing the overall corrosion performance using the potentiodynamic polarization method, all the above-mentioned parameters must be considered. Based on the results, the AZ91 alloy can be identified as the most corrosion-resistant material, whereas the AE21 alloy exhibited the poorest corrosion behavior. For clarity, a graphical representation of the measured results is shown in Figure 7.

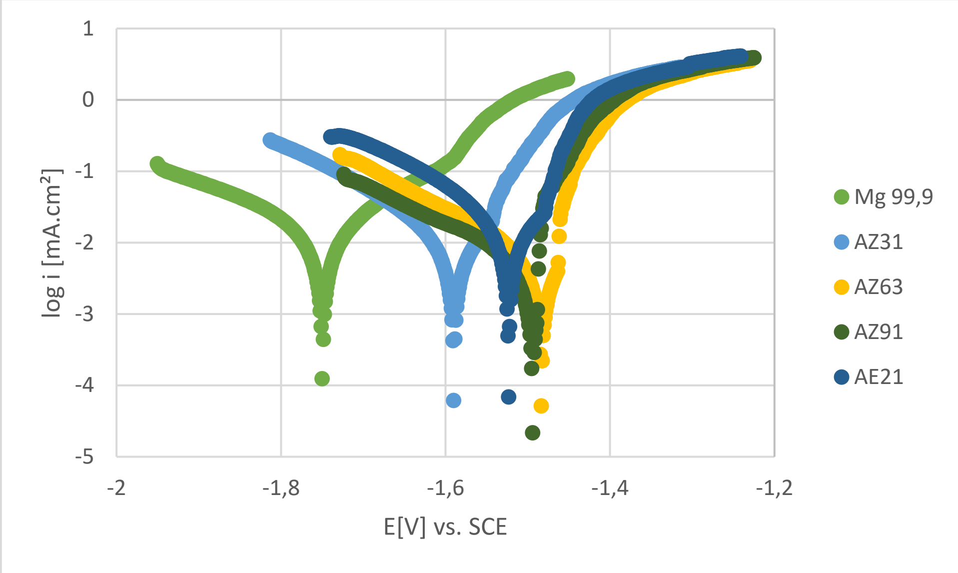

Fig.7. The potentiodynamic curves of the magnesium samples

Table 4 represents the parameters of the equivalent circuit elements in the EIS method. The parameters are: R_S, R_{P1}, R_{P2}, CPE_1, CPE_2. In the table are also presented exponential coefficients n_1 and n_2. The exponential coefficients describe the depression of the semicircle in the Nyquist plots and take values between 0 and 1. The closer the coefficient is to 1, the more the curve resembles a perfect semicircle.

Table 4
Measured parameters of the equivalent circuit in EIS

Material	R_S [$\Omega.cm^2$]	R_{P1} [$\Omega.cm^2$]	R_{P2} [$\Omega.cm^2$]	R_{Total} (R_P) [$\Omega.cm^2$]	$CPE_1 . 10^{-6}$ [F. sn^{-1}]	$CPE_1 . 10^{-3}$ [F. sn^{-1}]	n_1 [-]	n_2 [-]
Mg 99,9	74	1425	920	2345	12	1	0.9	0.7
AZ31	77	1434	543	1977	7.6	1.4	0.9	0.8
AZ63	79	2403	1710	4113	7.1	0.7	0.9	0.7
AZ91	74	2970	2478	5448	7.2	0.6	0.9	0.7
AE21	76	762	-	762	10.2	-	0.9	-

When evaluating the experimental results, the focus was placed on the comparison of the R_{total} values. A higher R_p value indicates a greater resistance to charge transfer and therefore, better corrosion resistance. The AZ91 alloy exhibited the highest R_p, confirming its superior corrosion resistance. It was closely followed by the AZ63 alloys which also demonstrated high polarization resistance. The AE21 alloy showed the lowest performance, with total resistance of only 762 $\Omega.cm^2$, indicating the poorest resistance among the tested samples. In addition to the table, Nyquist plots were used for better visualization of the EIS results. Nyquist plots of samples are shown in Figure 8.

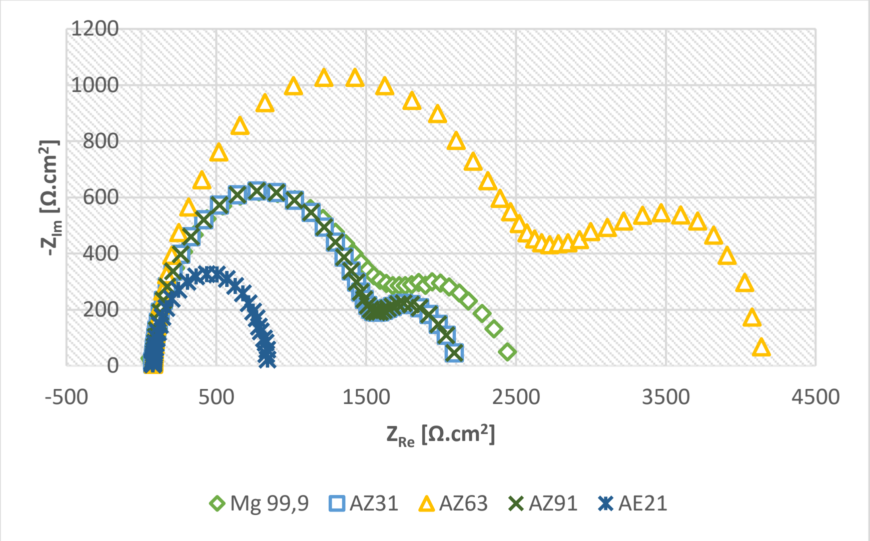

Fig.8. The Nyquist plots of the magnesium samples

4. CONCLUSION

Based on the obtained measurement results and their analysis, the following conclusions were drawn:

- The addition of aluminum in the AZ91 alloy contributes to achieving higher mechanical strength.
- The AZ91 alloy exhibited the highest hardness among all tested samples (61.1 HV).
- The presence of rare earth elements (REEs) decreases the hardness of magnesium alloys.
- Commercially pure magnesium (Mg 99.9) is not suitable for biomedical use due to its low hardness.
- The corrosion potential (E_{corr}) of the AZ91 alloy (-1520mV) is more positive than potential of the AE21 alloy (-1533mV), indicating that AZ91 is significantly more corrosion resistant than AE21.
- The corrosion current density showed the best result for AZ91 and the worst result for AE21 alloy, confirming the markedly better corrosion resistance of AZ91.
- From the EIS measurements, the polarization resistance was highest for AZ91 and lowest for AE21, again demonstrating that AZ91 is more resistant alloy.
- Overall, the AZ91 alloy exhibited the best corrosion resistance and mechanical properties among all tested materials.
- The AZ91 alloy, after further studies on its biocompatibility, due to its higher aluminum content, could potentially be used for biomedical applications.

The overall conclusion of this study is that the most suitable magnesium alloying elements for biomedical applications, in terms of improving both mechanical strength and corrosion resistance, are aluminum and zinc. Among the tested alloys, AZ91 achieved the most favorable results in all experiments, making it the most promising candidate for biomedical use. In contrast AE21 alloy achieved the poorest performance in all three experiments due to its low mechanical strength and corrosion resistance.

Although the AZ91 exhibited the best performance in the experiments the problem with biocompatibility remains. Currently, implant manufacturers tend to avoid the use of aluminum metal in the human body. The reason is that the aluminum metal when exposed to human bloodstream and in acidic environments has been found to cause disruptions in cellular metabolism. Prolonged exposure to this element may even lead to the development of neurodegenerative diseases. In the case of magnesium alloys where aluminum is used only as an alloying element, Al exists in the solid solution and metallic forms. Whether aluminum in such forms has a similarly negative effect on the human organism is not yet clearly established.

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