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Changes in the structure, mechanical and corrosion properties of the Mg–Zn–Zr system alloy subjected to equal channel angular pressing

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Abstract: Magnesium alloys are considered promising materials for the production of bioresorbable implants. Their main disadvantages are low strength and corrosion resistance in biological environment. In the work, the authors studied the effect of severe plastic deformation using the equal channel angular pressing (ECAP) method on the structure, mechanical properties, and corrosion resistance of the Mg–8.6Zn–1.2Zr magnesium alloy. It was identified that one ECAP cycle at 400 °C leads to a substantial hardening of the Mg–8.6Zn–1.2Zr alloy by ~10 %, up to 330 MPa. Structural studies showed that dynamic recrystallisation plays a significant role in the structure transformation. ECAP leads to the formation of a bimodal structure with large deformed grains with an average transverse size of 20±4 μm and recrystallised grains with an average transverse size of 6±2 μm. It was found that with a decrease in the strain temperature up to 250 °C, the process of deformation-induced decay of the supersaturated solid solution takes place. Electrical conductivity of a sample after ECAP at 400 °C amounted 29±2 % according to the International Annealed Copper Standard (IACS), while second ECAP cycles lead to an increase in the electrical conductivity up to 32±2 % IACS. Using the electrochemical corrosion method, the authors found that one ECAP cycle at 400 °C leads to a slight decrease in the corrosion resistance of the alloy under study compared to the initial state. The study showed that the corrosion current increases from 24 to 32 μA/cm^2, while the subsequent ECAP cycle at 250 °C increases the corrosion current more than twice (up to 57 μA/cm^2).

Keywords: Mg–Zn–Zr system alloys; Mg–8.6Zn–1.2Zr; magnesium alloys; high strength of magnesium alloys; ECAP; corrosion resistance; electrical conductivity; dynamic recrystallisation during ECAP.

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INTRODUCTION

Currently, magnesium alloys are considered as promising materials for the development and production of biodegradable implants, to use in traumatology and surgery [1–3]. The attention was paid to these alloys for a good reason: magnesium has an elastic modulus close to human bone; it is non-toxic and biocompatible with the human body [4]. However, for the full use of these materials, it is necessary to eliminate a number of their demerits, first, to increase the strength characteristics. Alloying is one of the ways to solve this problem. Magnesium itself has a low capacity for strain hardening, but alloying can increase the hardening effect during thermomechanical processing. In particular, widespread magnesium systems with zinc and zirconium can be selected. Due to solid solution strengthening in Mg–Zn systems, the strength increases, and additional alloying with zirconium allows increasing ductility [5–7]. For magnesium alloys, processes of multi-cycle rolling with decreasing temperature are widespread [8]. As a result of this processing, products with high strength are produced due to the formation of a lineage and partially recrystallised structure. One should, note that the high extent of the boundaries of deformed grains, and the high dislocation density in this case can negatively affect the corrosion resistance of magnesium alloys [9–11].

Researchers have also drawn attention to methods based on the principles of severe plastic deformation. It is shown in [12–14] that high-pressure torsion can lead to

more efficient refinement of the structure of magnesium materials compared to rolling, however, this deformation method is not scalable. Another approach is equal channel angular pressing (ECAP). As a rule, to achieve strength above 300 MPa, from 2 to 4 deformation treatment cycles are carried out [15–17]. In this case, the effect of sliding along the main base plane has been identified, which reduces the ECAP effectiveness, and in some cases even leads to softening of the material with an increase in the number of deformation cycles [18–20]. However, thermomechanical processing, including ECAP and carried out according to special technological modes, can achieve the required set of properties.

The purpose of this work is to determine the influence of the deformation mode during equal channel angular pressing (ECAP) on the features of the structural state formation, corrosion resistance and mechanical characteristics of the Mg–Zn–Zr system alloy.

METHODS

The magnesium Mg–8.6Zn–1.2Zr (wt. %) alloy was chosen as the research material. Chemical analysis was carried out on a Thermo Scientific ARL Optim'X X-ray fluorescence spectrometer. The material as-delivered is a strip 20 mm thick, from which samples with a diameter of 20 mm, and a length of 100 mm were cut along the rolling direction on an ARTA-120 spark-cutting mill.

The initial state was taken to be after prolonged annealing at 400 °C for 24 h. The mode was chosen based on literature data [21–23] and state diagrams of the Mg–Zn and Mg–Zr systems. Annealing was carried out in air in a Snol 8.2/1000 furnace with air cooling.

Deformation processing using the ECAP method was carried out in 2 regimes.

1. Initial temperature of equipment and blanks is 400 °C, channel intersection angle is 120°, and deformation rate is 1 mm /s, 1 cycle.

2. Processing according to mode 1, then 1 ECAP cycle according to *Bc* route (the blank was turned around 90° relative to the longitudinal axis), at an initial temperature of the equipment and blanks of 250 °C, the channel intersection angle of 90°, the deformation rate of 1 mm/s, in copper shell 1.5 mm thick.

Structural studies were performed using an Olympus GX51 light microscope and a JSM6490 scanning electron microscope.

Mechanical tests were carried out in accordance with GOST 1497-84. For tensile tests, proportional cylindrical samples with a working part diameter of 3 mm and an initial gauge length of 15 mm were used. Tests were performed on an Instron 5982 electromechanical static test system, at room temperature, at a rate of 1 mm/min.

Electrical conductivity was determined using a VE-27NTs eddy-current meter, converting the obtained values into IACS (International Annealed Copper Standard).

Electrochemical corrosion tests were carried out using an Elins R-5X potentiostat-galvanostat-impedancemeter in Ringer's solution with pH=7.4 in a three-electrode 80 ml cell with a silver chloride reference electrode and a platinum counter electrode. The tests were carried out at a temperature of 37 °C for 12 h, while during the first 2 h the electrode free corrosion potential was measured until a steady state was established on the surface of the sample. To obtain polarisation curves after establishing a steady state, a potential sweep was performed in the range from −300 to +300 mV relative to the steady-state value of the electrode potential with a scanning velocity of 0.25 mV/s. The current and corrosion potential were calculated from polarisation curves using Tafel sections [24].

RESULTS

In the initial heat-treated state, the Mg–8.6Zn–1.2Zr alloy sample has a coarse-grained state with a bimodal grain size distribution (Fig. 1). Large grains with an average transverse size of 30±10 μm and small recrystallised grains with an average transverse size of 4±2 μm are observed. The initial state can be additionally characterised by such a structure-sensitive parameter as electrical conductivity, which helps to assess indirectly, the change in the solid solution concentration in the alloy during further deformation processing. In the initial state, the electrical conductivity was 29±2 % IACS.

As a result of deformation processing carried out in regimes 1 and 2, it was identified that the structure bimodal appearance was preserved in the samples (Fig. 2). After 1 ECAP cycle, the deformation texture is clearly pronounced, coarse grains are turned in the direction of shear action in the ECAP focus, their average transverse size decreased to 20±4 μm, recrystallised grains have an average transverse size of 6±2 μm and are located mainly along the boundaries of large deformed grains. This indicates the implementation of processes of both grain transformation and dynamic recrystallisation. The second cycle in regime 2 does not make significant changes to the nature of the structure. Coarse deformed grains with an average transverse size of 18±4 μm and recrystallised grains with an average transverse size of 5±2 μm are observed.

After deformation in regime 1, the electrical conductivity remained at the level of 29±2 % IACS. After deformation in regime 2, it was 32±2 % IACS. This change is most likely explained by temperature conditions.

The results of mechanical tensile tests indicate an increase in the Mg–8.6Zn–1.2Zr alloy strength after ECAP (Fig. 3). After the 1st ECAP cycle, the tensile strength increased from 300±7 to 330±5 MPa; the 2nd cycle did not lead to an increase in strength.

The next important characteristic of the material for the production of implants is its corrosion resistance. From the analysis of the obtained results of corrosion tests presented in the form of polarisation curves, the values of the corrosion current (i_{corr}) and free corrosion potential (E_{corr}) were obtained (Table 1). Fig. 4 shows that Tafel regions are observed on the cathode branches of the samples. As a result of ECAP treatment in regimes 1 and 2, the surfaces of the samples are passivated, as evidenced by the lower value of the free corrosion potential E_{corr}. Based on the results presented in Table 1, the initial sample with the minimum corrosion current i_{corr} has the best corrosion properties. The displacement of the cathodic branches indicates a change in the surface area available for the cathodic reaction.

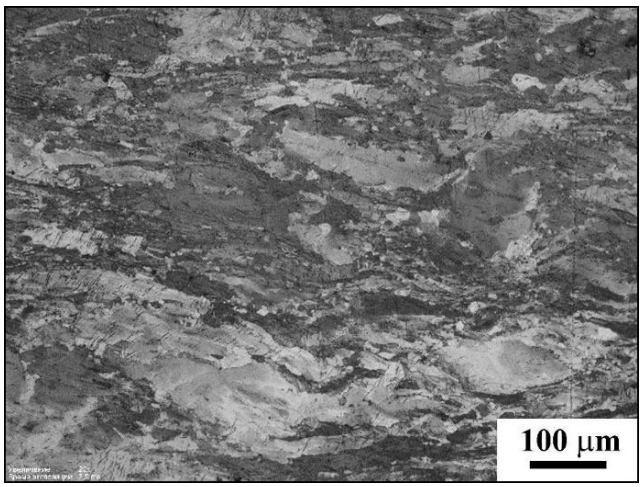
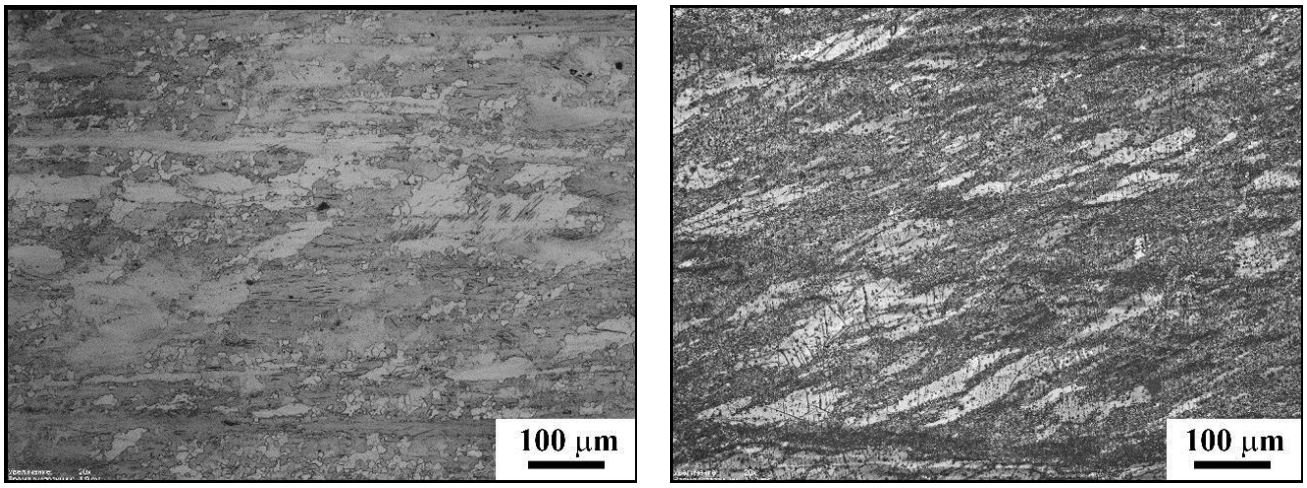


Fig. 1. *The structure of the Mg–Zn–Zr system alloy after annealing at 400 °C for 24 h. Light microscopy*
Рис. 1. *Структура сплава системы Mg–Zn–Zr после отжига при 400 °С в течение 24 ч. Световая микроскопия*



a ***b***

Fig. 2. *The structure of the Mg–Zn–Zr system alloy after ECAP according to regimes 1 (**a**) and 2 (**b**)*
Рис. 2. *Структура сплава системы Mg–Zn–Zr после РКУП по режиму 1 (**а**) и 2 (**b**)*

DISCUSSION

The strength properties, and corrosion resistance of a magnesium alloy are closely related to its structural state. Structure refinement can have a dual effect on the corrosion resistance of magnesium alloys. With the Mg–Al system, grinding will lead to an increase in corrosion resistance [25], while for a Mg–Zn–Zr system alloy, the nature of the corrosion behaviour may be different, due to the type and distribution of the second phase dispersed particles along the grain boundaries, and therefore an increase in the corrosion rate is possible [26]. The results of measuring electrical conductivity can indirectly prove the change in the solid solution concentration and the process of precipitation of the second phase particles. It is known that at temperatures above 300 °C, the solubility of both Zn and Zr increases. Thus, ECAP at 250 °C leads to the decomposition of the solid solution of alloying elements. It was found that in regime 1, when the temperature was 400 °C, the electrical conductivity corresponded to the value of the annealed state of 29 % IACS. Regime 2 at a temperature of 250 °C leads to an increase in the material electrical conductivity up to 32 % IACS. Thus, lowering the deformation processing temperature to 250 °C will lead to deformation-stimulated decomposition of the solid solution.

Electrochemical tests for corrosion resistance indicate that the magnesium alloy sample in the annealed state is of greatest importance, which is caused by the equilibrium state of the grain boundaries [27]. A decrease in the average transverse size of coarse and recrystallised grains, texturing, and the possible increase in the number of crystal lattice defects, as well as a change in the solid solution state, lead to an increase in corrosion currents. Moreover, after high-temperature deformation processing in regime 1,

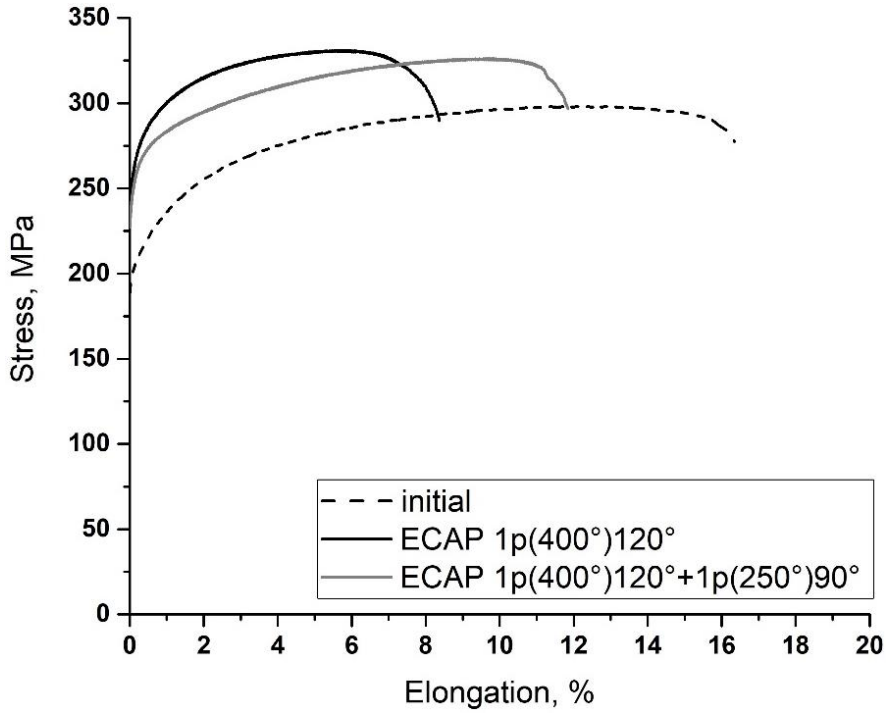


Fig. 3. *Curves of mechanical tests of the Mg–Zn–Zr system alloy after annealing at 400 °C, ECAP according to regimes 1 and 2*
Рис. 3. *Кривые механических испытаний сплава системы Mg–Zn–Zr после отжига при 400 °C, РКУП по режимам 1 и 2*

Table 1. *Values of corrosion current and free corrosion potential*
Таблица 1. *Значения тока коррозии и потенциала свободной коррозии*

State	I_{corr}, μA /cm^2	E_{corr}, V
Initial state (400 °C, 24 h)	24.05±5.46	−1.428±0.013
ECAP, regime 1	32.06±16.20	−1.382±0.030
ECAP, regime 2	57.00±6.22	−1.391±0.085

the increase is ~25 %, and after the 2nd cycle of deformation at a lower temperature (250 °C) in accordance with regime 2, the corrosion current increases by more than 2 times relative to the initial state.One should note that on the Mg–8.6Zn–1.2Zr alloy, it was possible to achieve high values of ultimate strength and offset yield strength within just one ECAP cycle. Table 2 presents a comparison of the achieved strength characteristics with the results of other studies on the alloys of similar chemical composition.

CONCLUSIONS

It was identified that during ECAP deformation processing of the Mg–8.6Zn–1.2Zr alloy, temperature plays a significant role. Thus, at 400 °C, the main mechanism of structure formation during deformation is dynamic recrystallisation, while the solid solution state is maintained.

Deformation processing of the Mg–8.6Zn–1.2Zr alloy by ECAP, 1 cycle, channel intersection angle 120°, 400 °C (mode 1), leads to the formation of a structure with recrystallised grains with an average transverse size of 6±2 μm, and larger deformed grains directed along the shear direction, with an average transverse size of 20±4 μm. Such structural state provides an increase in tensile strength relative to the initial one annealed by ~10 %, up to 330 MPa.

Electrical conductivity measurements, indirectly indicate that lowering the temperature of ECAP deformation processing to 250 °C leads to deformation-induced decomposition of the supersaturated solid solution. The electrical conductivity after deformation in regime 1 was 29±2 % IACS, while the final electrical conductivity after deformation of the Mg–8.6Zn–1.2Zr alloy in regime 2 differs from the initial state by 3 % and amounts to 32±2 % IACS.

Table 2. *Mechanical characteristics of the Mg–Zn–Zr system alloy after ECAP*
Таблица 2. *Механические характеристики сплава системы Mg–Zn–Zr после РКУП*

Work	Mode	UTS, MPa	YTS, MPa	Percentage elongation, %
Current work	ECAP, regime 1	330	267	8
	ECAP, regime 2	325	245	12
[16]	4 ECAP cycles, *Bc* route, 220 °C, channel intersection angle 90°	290	231	27
[28]	2 ECAP cycles, *Bc* route, channel intersection angle 90°	341	264	23
	4 ECAP cycles, *Bc* route, 220 °C, channel intersection angle 90°	334	277	21
[29]	2 ECAP cycles, 250 °C, channel intersection angle 90°	326	175	25

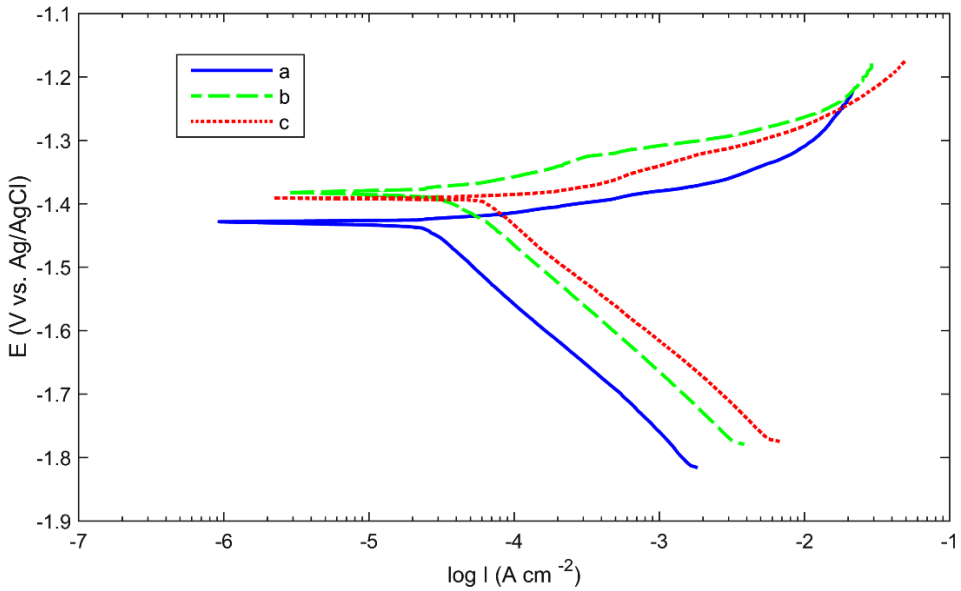


Fig. 4. *Polarisation curves: a – initial state; b – regime 1; c – regime 2*
Рис. 4. *Поляризационные кривые: a – исходное состояние; b – режим 1; c – режим 2*

The results of electrochemical corrosion tests indicate that the magnesium alloy sample in the initial annealed state, has the greatest resistance to corrosion. 1 ECAP cycle at 400 °C leads to an increase in the corrosion current by 25 % (32.06±16.20 μA/cm^2), while subsequent deformation at 250 °C leads to an increase in the corrosion current by more than 2 times (57.00± ±6.22 μA/cm^2).

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Изменение структуры, механических и коррозионных свойств сплава системы Mg–Zn–Zr, подвергнутого равноканальному угловому прессованию

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Аннотация: Магниевые сплавы считаются перспективными материалами для изготовления биорезорбируемых имплантатов. Их основные недостатки – низкая прочность и коррозионная стойкость в биологических средах. В работе изучалось влияние интенсивной пластической деформации методом равноканального углового прессования (РКУП) на структуру, механические свойства и коррозионную стойкость магниевого сплава Mg–8,6Zn–1,2Zr. Установлено, что 1 цикл РКУП при 400 °C ведет к заметному упрочнению сплава Mg–8,6Zn–1,2Zr на ~10 %, до 330 МПа. Структурные исследования показали, что в трансформации структуры существенную роль играет динамическая рекристаллизация. РКУП ведет к формированию структуры бимодального вида с крупными деформированными зернами со средним поперечным размером 20±4 мкм и рекристаллизованными зернами со средним поперечным размером 6±2 мкм. Установлено, что с понижением температуры деформации до 250 °C происходит процесс деформационно-индуцированного распада пересыщенного твердого раствора. Электропроводность образца после РКУП при 400 °C составляла 29±2 % согласно International Annealed Copper Standard (IACS), в то время как 2 цикла РКУП при 250 °C ведут к повышению электропроводности до 32±2 % IACS. Методом электрохимической коррозии установлено, что 1 цикл РКУП при 400 °C приводит к незначительному снижению коррозионной стойкости исследуемого сплава по сравнению с исходным состоянием. Показано, что ток коррозии увеличивается с 24 до 32 мкА/см^2, в то время как последующий цикл РКУП при 250 °C увеличивает ток коррозии более чем в 2 раза (до 57 мкА/см^2).

Ключевые слова: сплавы системы Mg–Zn–Zr; Mg–8,6Zn–1,2Zr; магниевые сплавы; высокая прочность магниевых сплавов; РКУП; коррозионная стойкость; электропроводность; динамическая рекристаллизация при РКУП.

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