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# Structure and strength of Mg-Zn-Zr alloy subjected to high pressure torsion

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**ABSTRACT**

Magnesium alloys are considered a promising material for the production of biodegradable implants. However, their widespread adoption is hindered by their low strength and high corrosion rate. Deformation can enhance the strength characteristics; however, due to the limited number of slip planes in magnesium alloys, choosing the deformation processing regime for them is a non-trivial task. This study presents the results of research aimed at determining the influence of processing modes by high-pressure torsion on the transformation mechanisms of the structure and mechanical characteristics of the Mg-8.6Zn-1.2Zr alloy. It is shown that at room temperature, predominantly a twinning structure is formed, with microhardness values reaching 1200 MPa. However, it is demonstrated that such a structure leads to significant embrittlement of the Mg-8.6Zn-1.2Zr alloy. Deformation at a temperature of 250 °C leads to the formation of recrystallized grains with a size of 3–4 μm. After 5 revolutions of high-pressure torsion, the microhardness is 820 MPa, and the tensile strength is 335 MPa, with an elongation of 13 %.

**KEYWORDS**

magnesium alloys • biomedical applications • severe plastic deformation • bioresorbable materials • nanomaterials

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## Introduction

Magnesium is characterized by low density while exhibiting outstanding mechanical properties such as high ductility and specific strength. Therefore, magnesium alloys have received significant attention as lightweight structural materials. However, some magnesium alloys possess properties such as biocompatibility and an elastic modulus close to that of human bone tissue, making them suitable for medical implant materials [1–3]. Magnesium and its alloys are biodegradable, allowing for the production of temporary implants and thus reducing the number of surgical interventions, avoiding the need for repeat surgery procedures, and reducing the traumatic effect [4,5]. It is worth noting that magnesium is non-toxic and hypoallergenic, being essential for human metabolism and naturally present in bone tissues [4,6]. The Young's modulus of magnesium alloys, being close to the modulus of bone under loading, helps to reduce the effect of stress differences occurring in the "bone-implant" system [7]. To ensure adequate bone tissue restoration, a magnesium alloy implant must possess a certain corrosion rate and high levels of mechanical properties. This requirement is formulated


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based on the fact that a certain amount of time is needed for the complete fusion of bone fragments, during which the implant dissolves, leading to a corresponding decrease in the overall strength of the structure. Naturally, there is a gradual "transfer" of load from the implant to the bone. This aspect underscores the necessity of obtaining an implant with a specified dissolution rate and certain strength characteristics [5,6].

One of the effective methods for improving the comprehensive mechanical properties of metallic materials is deformation processing with the refinement of structural components down to an ultrafine-grained (UFG) state, enhancing their size uniformity. However, magnesium and its alloys poorly undergo deformation processing at room temperature due to their hexagonal close-packed (HCP) lattice, which limits the number of active slip systems [8]. Thus, the application of approaches to enhance the mechanical properties of magnesium alloys based on plastic deformation requires careful selection of processing regimes and the use of combined treatments, including heat treatment and pre-deformation [9,10].

Of particular interest is the application of thermo-mechanical processing based on severe plastic deformation (SPD), which is one of the most effective methods used to form UFG structures in metals today. Among the possible SPD methods, some of the main ones are equal-channel angular pressing (ECAP) and high-pressure torsion (HPT) [11–13]. Magnesium and its alloy processing by ECAP are carried out at relatively high temperatures, which helps avoid segmentation and cracking caused by low ductility at room temperature. However, during processing at high temperatures, one of the main structure transformation processes is recrystallization [14,15]. HPT processing has its advantages and disadvantages; however, from a scientific point of view, it allows processing under "critical" conditions for the material, thereby determining the real range of thermo-mechanical processing regimes available for the investigated material. For example, HPT for magnesium alloys allows avoiding undesirable cracking even at room temperature, due to high hydrostatic pressure [16]. Also, compared to ECAP, HPT has the advantage of forming a structure with smaller grain size and makes it possible to analyze structural and phase transformations under extreme conditions for the material [16–18]. The structure transformation processes of pure magnesium during HPT are discussed in the literature [19,20], however, regarding the Mg-Zn-Zr alloy system, there are not many studies [21–24], and this work is aimed at expanding the knowledge about changes in the structure and properties of the Mg-Zn-Zr alloy under high pressures. The Mg-Zn-Zr system is interesting because already in the annealed state, the samples have a tensile strength close to 300 MPa [25]. The alloying elements Zn and Zr are non-toxic and can lead to additional strengthening and refinement of the structure [26,27]. Based on the above, the aim of this study is to establish the influence of the high-pressure torsion (HPT) process on the structure and properties of the biodegradable magnesium alloy in the Mg-Zn-Zr system.

## Materials and Methods

The research material selected for the study is the biodegradable magnesium alloy Mg-8.6Zn-1.2Zr (grade MA14). Based on preliminary studies and literature data [28,29], the initial structural condition obtained through prolonged annealing process at a temperature of 430 ± 10°C for 24 hours in a Snol 8.2/1000 furnace has been adopted.

As stated above, high-pressure torsion (HPT) was chosen as the investigated method. The specimens were in the form of disks with a thickness of 2 mm and a diameter of 10 mm. HPT was conducted at a pressure of 6 GPa on flat anvils, at temperatures of 20 and 250 °C. Samples were obtained after 1 and 5 rotations of torsion, at a constant angular velocity of 1 rpm.

Structural analysis was performed using a Carl Zeiss Axio Observer A1m optical microscope. The linear density of twins was calculated using the equation [30]:

$$\rho = N/L, \quad (1)$$

where $N$ is the number of twins occurring within the segment, and $L$ is the length of the segment.

X-ray structural analysis was conducted using a Bruker D8 Advance diffractometer with CuKα radiation, with continuous sample rotation (20 degrees/min) and step scanning (step size 0.02°, exposure time 1 s) in the angular range of 2Θ = 30°–90°. To evaluate dislocations, an approach based on modified Williamson-Hall and Warren-Averbach methods as described in references [31,32] was implemented.

Microhardness was measured using a Micromet 5101 microhardness tester. A diamond indenter in the form of a tetrahedral pyramid with a square base under a load of 100 g was lowered onto the ground and held for 10 seconds. The measurements were carried out along the diagonal of the sample.

Tensile mechanical tests were conducted on "miniature" specimens (see Fig. 1), cut using an electroerosion machine ARTA 120 in such a way that the working part fell within the middle radius region of the sample (see Fig. 1). The length of the working part was 4 mm, thickness was 0.8 mm, and width was 0.6 mm. The tests were performed at a deformation rate of 0.24 mm/min at room temperature.

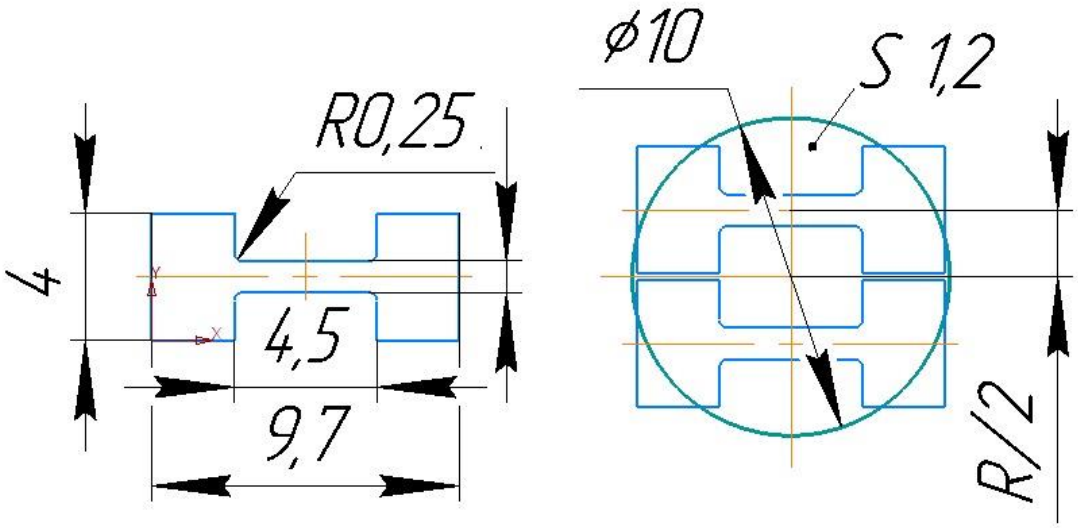


**Fig. 1.** Drawing of a "small" tension specimen and scheme of cutting a "small" tension specimen

## Results and Discussion

The initial state is characterized by a structure of a mixed nature (see Fig. 2). Large grains of 30 ± 10 μm and clusters of recrystallized grains along the boundaries of the strips with an average size of 4 ± 2 μm are observed.

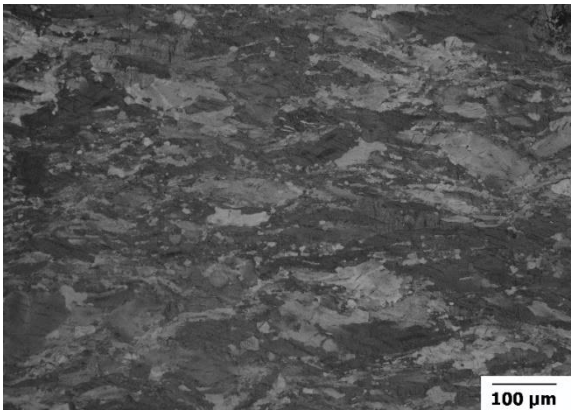


**Fig. 2.** The structure of the initial state of the Mg-8.6Zn-1.2Zr alloy

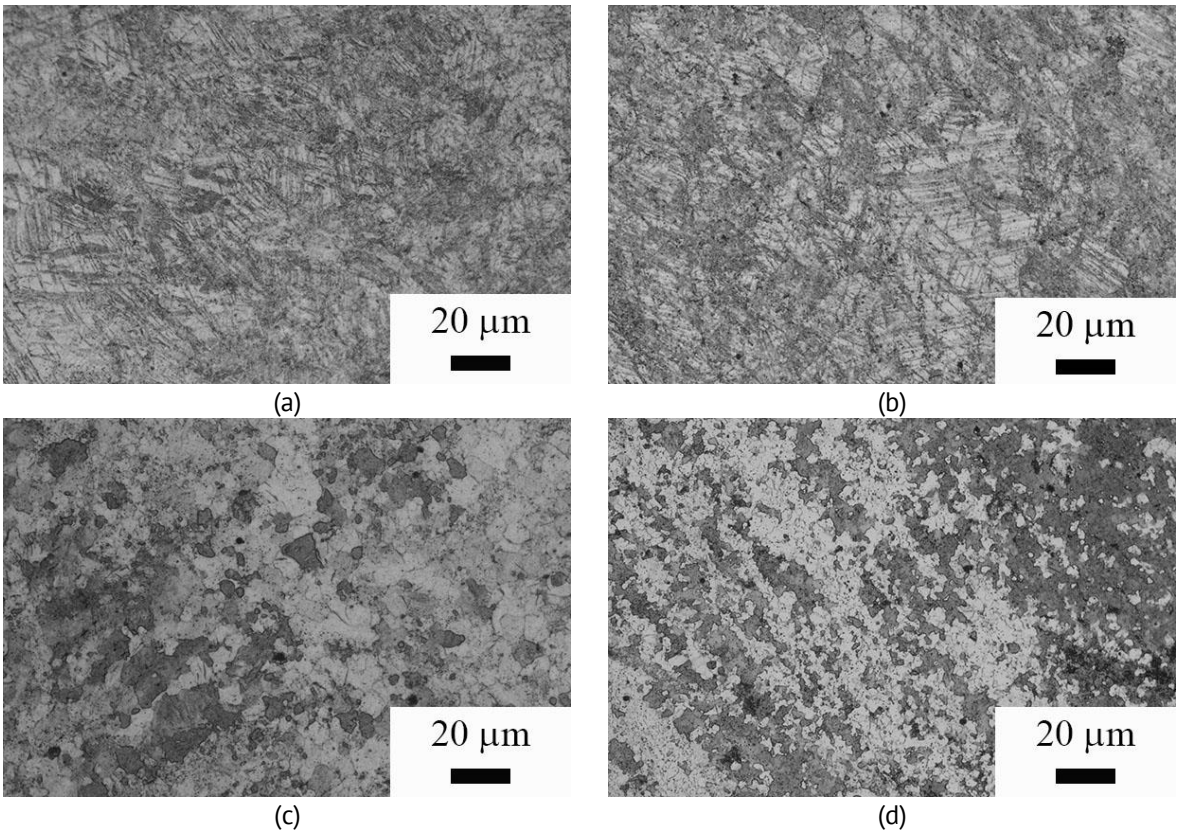


**Fig. 3.** The structure of Mg-8.6Zn-1.2Zr alloy specimens after high-pressure torsion (HPT) deformation: 1 rotation at 20 °C (a), 5 rotations at 20 °C (b), 1 rotation at 250 °C (c), and 5 rotations at 250 °C (d)

The analysis of the structure after HPT deformation indicates that at room temperature, one of the main mechanisms of structure transformation is twinning (see Fig. 3(a,b)). The calculation of twin linear density shows that after 1 rotation, the twin

density is 0.14 $\mu m^{-1}$, and after 5 rotations, the twin density decreases by 3 times to 0.05 $\mu m^{-1}$. The average twin thickness after 1 rotation is 2.6 ± 1.3 μm, and after 5 rotations, it is 2.1 ± 1.1 μm. The structure is highly twinned, and grain boundaries are poorly distinguishable; however, in the work [21], the formation of recrystallized grains with a size of ~1 μm and larger grains containing twins in their body is noted. It is worth noting that in the mentioned work, the applied pressure was only 2 GPa, which is three times less than in this study.

A temperature of 250 °C is considered high for magnesium alloys, so a large number of recrystallized grains are observed in the samples after HPT at this temperature (see Fig. 3(c,d)). After 1 rotation, the average size of recrystallized grains is 4.0 ± 1.3 μm, and larger grains containing twins in their body are also observed. After 5 rotations, twins are hardly observed, and the average size of recrystallized grains remains almost unchanged at 3.0 ± 1.4 μm.

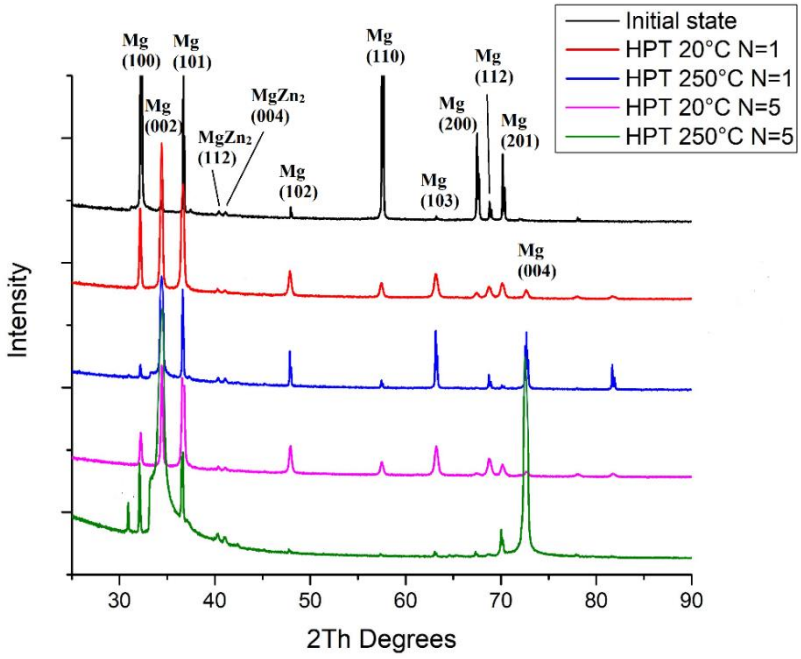


**Fig. 4.** X-ray diffraction patterns of the Mg-8.6Zn-1.2Zr alloy in the initial state and after high-pressure torsion

The conducted phase X-ray structural analysis indicates the presence of 0.3 % by weight of the $MgZn_2$ phase in the initial state (Fig. 4). Deformation by high-pressure torsion (HPT), regardless of the regime, leads to an increase in the content of this phase to 1.2–1.3 % by weight, indicating the decomposition of the solid solution during deformation. Analysis of the diffraction patterns allowed to establish a substantial refinement of the structure, as evidenced by the data on the coherent scattering region (CSR) (Table 1). The main refinement occurs during the first rotation, and no significant changes are observed with an increase in the number of rotations up to 5. Moreover, deformation at room temperature results in more substantial refinement compared to deformation at 250°C. CSR reduces approximately 7 times to 38 ± 4 nm after 1 rotation and reaches 32 ± 3 nm after 5 rotations. Deformation at a higher temperature leads to a

3-fold reduction in CSR to 81 ± 6 nm after 1 rotation, and this value remains unchanged after 5 rotations. The magnitude of microstrains in the samples subjected to HPT at 250 °C is three times smaller than in the samples after HPT at room temperature.

Analysis of dislocation density allowed establishing that during high-pressure torsion (HPT) at room temperature, the density increases by ~ 2 times, reaching values of $6.7 \times 10^{14}$ $m^{-2}$ after 5 rotations. This density value is 1.5 times lower than at 250 °C. The main increase in the number of dislocations for both conditions occurs after 1 rotation. HPT for 5 rotations at 250°C leads to an increase in defect density to $9.9 \times 10^{14}$ $m^{-2}$.

**Table 1.** X-ray diffraction analysis results

| State | Lattice parameter, nm | | CSR*, nm | Microstrain | Dislocation density, $10^{14}$ $m^{-2}$ |
|---|---|---|---|---|---|
| | *a* | *c* | | | |
| Initial | 0.3202 | 0.5200 | 257±14 | 0.1271±0.0014 | 3.5 |
| HPT 20 °C 1 rev. | 0.3200 | 0.5197 | 38±4 | 0.5394±0.0079 | 6.1 |
| HPT 20 °C 5 rev. | 0.3203 | 0.5200 | 32±3 | 0.5472±0.0066 | 6.7 |
| HPT 250 °C 1 rev. | 0.3203 | 0.5200 | 81±6 | 0.1703±0.0054 | 9.7 |
| HPT 250 °C 1 rev. | 0.3203 | 0.5200 | 78±5 | 0.1781±0.0060 | 9.9 |

The investigation of microhardness (Fig. 5) revealed that the main strengthening occurs after 1 rotation of high-pressure torsion (HPT) both at room temperature and at an elevated temperature of 250 °C. At room temperature, the microhardness reaches approximately 1220 ± 60 MPa, and its distribution across the sample diameter is shown in Fig. 4. After 5 rotations, no changes in microhardness are observed. HPT at 250 °C leads to an increase in microhardness after 1 rotation to the level of 780 ± 50 MPa, and after 5 rotations, no significant change in microhardness (820 ± 50 MPa) is observed. It should be noted that for the magnesium alloy, there is no heterogeneity in the distribution of microhardness across the sample diameter, as also noted in the study [21].

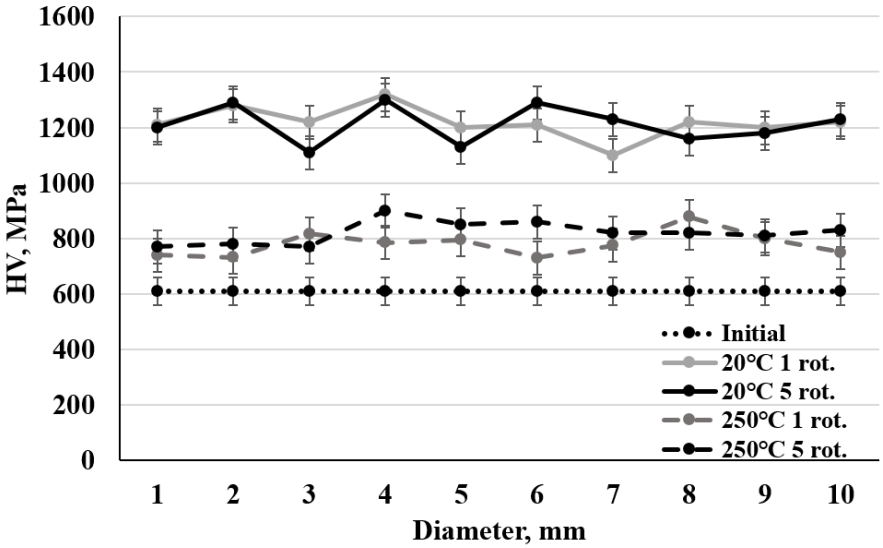


**Fig. 5.** The microhardness of the Mg-8.6Zn-1.2Zr alloy in the initial state and after high-pressure torsion

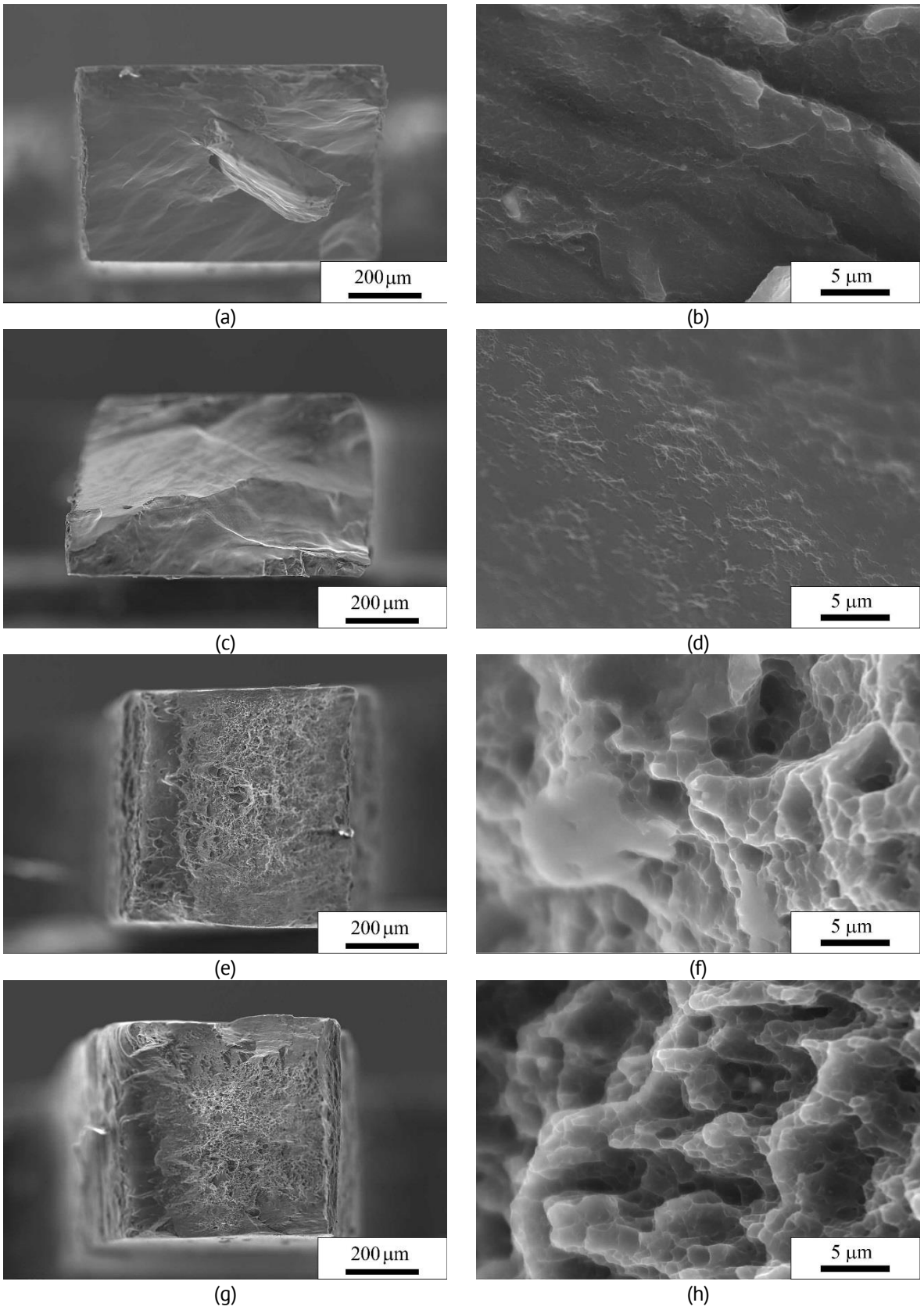


**Fig. 6.** The images of the fractures at magnifications of ×100 and ×4000 of the Mg-8.6Zn-1.2Zr alloy after high-pressure torsion (HPT) at temperatures of 20 °C for 1 (a,b) and 5 (c,d) rotations, and 250 °C for 1 (e,f) and 5 (g,h) rotations

Tensile tests indicate that effective strengthening occurs during hot deformation. The high-pressure torsion (HPT) regime at 250 °C for 5 rotations substantially increases both the tensile strength (335 ± 8 MPa) and the yield strength (275 ± 5 MPa) in the Mg-8.6Zn-1.2Zr alloy (Table 2). One rotation of HPT at 250 °C leads to an increase in tensile strength to 305 ± 7 MPa, however, the yield strength remains at the initial level of 185 ± 5 MPa. Deformation with 1 and 5 rotations of HPT at room temperature does not result in strengthening of the Mg-8.6Zn-1.2Zr magnesium alloy. The plasticity of the samples after HPT at 250 °C is explained by the presence of recrystallized grains.

**Table 2.** Mechanical properties

| State | UTS, MPa | YTS, MPa | Elongation, % |
|---|---|---|---|
| Initial | 270±5 | 180±5 | 15±1 |
| HPT 20 °C N=1 | 285±5 | - | <2% |
| HPT 20 °C N=5 | 290±5 | - | <2% |
| HPT 250 °C N=1 | 305±7 | 185±5 | 15±1 |
| HPT 250 °C N=5 | 335±8 | 275±5 | 13±1 |

To further understand the reasons behind such mechanical behavior, a fractographic analysis of the fractures was conducted (Fig. 6). At high magnification, it is observed that the fractures of the samples after room temperature high-pressure torsion (HPT) have a cellular relief. The fracture occurs by shear, which is typical for cast magnesium materials [33]. Fracture usually initiates from the surface. Twins act as barriers to dislocation propagation, leading to deformation localization and crack propagation in a shear-limited mode. Therefore, the obtained data on the ultimate tensile strength cannot be considered as final values.

In the case of the condition after hot HPT with recrystallized grains, a ductile fracture appearance is observed. Despite fracture initiation from the surface, deformation localization does not occur. In the central part of the samples after 1 and 5 rotations, elongated pits aligned with the fracture direction are observed. The average size of the pits is 1.3 ± 0.5 μm and 1.1 ± 0.4 μm for 1 and 5 rotations, respectively, indicating a similar fracture behavior for both conditions. A dimple region is observed on the opposite side of the fracture initiation. Such a characteristic fracture appearance has been observed multiple times in magnesium samples after deformation processing [34].

## Conclusions

1. Deformation of the Mg-8.6Zn-1.2Zr alloy by High-Pressure Torsion at room temperature results predominantly in a twin structure. The main strengthening occurs after 1 rotation. In this state, the average twin thickness is 2.6 ± 1.3 μm, and the linear density is 0.15 $\mu m^{-1}$. The state exhibits high microhardness at the level of 1200 MPa, and the specimens demonstrate brittleness, as evidenced by mechanical tests and fractographic studies. After 5 rotations, there is a decrease in the twin linear density to 0.05 $\mu m^{-1}$, while the twin thickness is 2.1 ± 1.1 μm. Significant changes in mechanical properties and fracture characteristics during tensile testing relative to specimens after 1 rotation are not observed for this state.

2. High-Pressure Torsion at 250 °C leads to the formation of predominantly recrystallized structure. After 1 rotation, the average grain size is 4.0 ± 1.3 μm. This state is characterized by an average microhardness across the diameter of 780 MPa, increased strength of 305 ± 7 MPa, and ductility of 15 %. For this state, a ductile fracture mode is observed during tensile testing. After 5 rotations at 250 °C, recrystallized grains with a size of 3.0 ± 1.4 μm are observed in the structure. The state is characterized by an average microhardness of 820 MPa, high strength of 335 ± 8 MPa, yield strength of 275 ± 5 MPa, and ductility of 13 %.
3. During high-pressure torsion of the Mg-8.6Zn-1.2Zr alloy, an active process of deformation-induced decomposition of the supersaturated solid solution occurs. Regardless of the deformation temperature and the number of rotations, there is an increase in the proportion of the $MgZn_2$ phase from 0.3 to 1.2–1.3 wt. %.

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