Evolution of the structure and mechanical properties of the Mg-1%Zn-0.2%Ca alloy during ECAP

O B Kulyasova, I A Evdokimova and R K Islamgaliev

Institute of Physics of Advanced Materials, Ufa State Aviation Technical University, 12 K. Marx Str., Ufa, 450008, Russia

E-mail: elokbox@mail.ru

Abstract. Microstructure of the Mg-1%Zn-0.2%Ca magnesium alloy after equal-channel angular pressing (ECAP) has been investigated. It was shown that an increase of the shear strain leads to significant grain refinement, as well as to the improved uniformity of microhardness. As a result of grain refinement by ECAP processing, the microhardness was increased to a value of 45.5, which is 28% higher than the initial state. Tensile tests demonstrated the enhanced ultimate tensile strength of 248.1 MPa, yield stress of 153.2 MPa, ductility of 19.4% whereas in the initial coarse-grained state these parameters were 124 MPa, 45.3 MPa and 8.7%, accordingly.

1. Introduction

Recently, the treatment of bone fractures has become widespread in traumatology [1]. For their treatment in traumatology various implants are used, mainly from bioinert metals and alloys [2]. The requirements for materials for the manufacture of implants are constantly being tightened, in particular, this concerns the increase in biochemical and biomechanical compatibility with body tissues, reduction of toxicity [3]. Bioreorbable Mg alloys are of great interest, since implants from the completely disappear when the bone has healed sufficiently, and the device becomes no longer useful [4-7]. Magnesium is a unique material for medical use also because it can gradually resorb [8]. Magnesium alloys are non-toxic, non-carcinogenic, their mechanical properties are more similar to the structure of the cortical bone [9]. But the analysis of the experiments allowed us to identify the main problems, namely, the problems of increasing strength [10]. It is known that a promising method for increasing the strength of magnesium alloys is grain refinement by the equal-channel angular pressing (ECAP) method [11-16]. Therefore, there is a special interest to study the influence of the structure evolution in the process of ECAP on the mechanical properties of the Mg-1% Zn-0.2% Ca alloy.

2. Experimental

The cast Mg-1%Zn-0.2%Ca alloy was obtained as an experimental material for the studies. The initial cast samples with a length of 100 mm and a diameter of 20 mm were subjected to homogenization annealing at a temperature of 723 K for 24 hours with cooling into water using a Nabertherm muffle furnace. Equal-channel angular pressing was carried out through a die-set with an angle of channels intersection of 120° at a speed of 6 mm/min. Samples were pressed by 1, 2 and 4 passes through route Bc at a temperature of 673 K. The equivalent strain in the case of the number of passes 1, 2, and 4 was 0.9, 1.8 and 3.6, respectively.
The microstructure was examined after etching in 2.5 mg. picric acid; 2.5 ml. acetic acid; 5 ml. distilled water; 50 ml. ethyl alcohol using an Olympus GX51 optical microscope. The microhardness (Hᵥ) was measured by the Vickers method on a Micromet 5101 measurement tool with a load of 50 g and a dwell time of 10 s. Tensile tests were performed on an Instron 5982 testing machine at room temperature and an initial strain rate of 10⁻³ s⁻¹ using specimens with a gage dimension of 0.6×1×4 mm³. For each condition, a minimum of 5 samples was tested.

3. Result and discussion

3.1. Microstructure

Metallographic analysis showed that the structure of the initial material consists of equiaxed grains with a mean grain size of about 280 μm (figure 1a). The heterogeneous structure was observed in the sample after 1 pass ECAP (figure 1b). Undeformed grains up to 280 μm in size occupied 65% of the scanned area. The remaining surface area consists of fine equiaxed grains with an average size of 4.5 μm. After 2 passes of deformation, slight changes in the microstructure were found. The size of large grains decreased to 230 μm and their fraction was only 31%, so the rest of the area was occupied by small grains of 4.3 μm in size (figure 1c). Further deformation up to 4 passes of ECAP leads to the most...
uniform structure with a mean grain size of 2.7 μm. However, large grains with a size of 120 μm existed in about 17% of the investigated area (figure 1d). It should be noted that the formation of an inhomogeneous structure after deformation by ECAP on the similar magnesium alloy Mg-1% Zn-0.3% Ca was observed also in [15].

3.2. Mechanical properties

The microhardness of the investigated alloy increased with an increase in the number of passes of ECAP due to the grain refinement. Thus, the microhardness of the initial samples was 35.5 Hv and it increased to 40, 42 and 45.5 Hv after 1, 2 and 4 passes of ECAP, respectively (figure 2a, table 1). The alloy in the initial coarse-grained state has rather poor mechanical properties, namely: yield stress (YS) 45.3 MPa, ultimate tensile strength (UTS) 124 MPa and ductility 8.7 % (figure 2b, table 1). An increase in the mechanical properties was found in all samples after deformation. As a result of deformation of the Mg-1% Zn-0.2% Ca alloy by 1 pass of ECAP, YS and UTS increased up to 60.7 and 181.2 MPa, respectively. An increase in strength occurred due to the grain refinement in the structure and growth of the dislocation density during severe plastic deformation. The ductility remained at the same level. After 2 passes of deformation, YS and UTS also slightly increased up to 68.7 and 195.3 MPa, respectively, in accordance with growth of the area, which was occupied by small grains (figure 1c).

The most fine-grained structure provides the highest level of YS and UTS, which were 153.2 and 248.1 MPa, respectively (figure 2b, table 1). The elongation to failure changed a lot after deformation by 4 passes and amounted to 19.4%. This result can be caused by both special texture formation and additional twinning during the ECAP processing. An increase in the strength of Mg-Zn-Ca alloys as a result of grain refinement during severe plastic deformation by ECAP was already observed in [15, 16]. In the case of the Mg-4%Zn-0.56%Ca alloy [15], the ultimate tensile strength (UTS) as a result of ECAP increased by factor 1.1 up to 215 MPa. In [16], due to the grain refinement by ECAP, UTS of the Mg-4%Zn-0.56%Ca alloy also increased up to 276 MPa that was 1.6 times higher in comparison with the initial undeformed sample. Both studies also revealed an increase in ductility as a result of the formation of an inhomogeneous (bi-modal) structure. It is noted that the increase in strength occurred due to the formation of small grains, which led to the formation of a greater length of grain boundaries. Large grains are responsible for an increase in ductility [17, 18].

Figure 2. (a) Microhardness, (b) tensile tests of the magnesium alloy after ECAP processing.
Table 1. Mechanical characteristics of the samples in different conditions.

| Samples          | Microhardness/Hv | YS/MPa | UTS/MPa  | Elongation (\(\varepsilon\)), % |
|------------------|------------------|--------|----------|-------------------------------|
| Initial state    | 35.5±3.5         | 45.3±2.3 | 124.0±6.2 | 8.7±1.2                       |
| ECAP 1 pass      | 40.0±2.2         | 60.7±3.1 | 181.2±7.1 | 10.0±1.1                      |
| ECAP 2 passes    | 42.0±1.5         | 68.7±3.4 | 195.3±7.7 | 10.6±1.2                      |
| ECAP 4 passes    | 45.5±0.7         | 153.2±5.7 | 248.1±7.4 | 19.4±1.3                      |

4. Conclusions
An increase in the equivalent strain during the ECAP processing leads both to the grain refinement and to an increase in the homogeneity of microstructure in the Mg-1%Zn-0.2%Ca alloy. The increase in microhardness and strength occurring during ECAP can be explained by the grain refinement and enhanced dislocation density. The finest grain structure provides the highest level of Hv, YS and UTS.

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