Materials Science & Engineering A 627 (2015) 56–60

Contents lists available at ScienceDirect

Materials Science & Engineering A

journal homepage: www.elsevier.com/locate/msea

Improving the mechanical properties of pure magnesium through cold hydrostatic extrusion and low-temperature annealing

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ARTICLE INFO

Article history:
Received 19 September 2014
Received in revised form
17 December 2014
Accepted 24 December 2014
Available online 7 January 2015

Keywords:
Mechanical characterization
Magnesium
Bulk deformation
Thermal anomaly

ABSTRACT

A way to produce ultrafine grained structure with $d\ \sim 3\ \mu m$ and high plasticity in pure magnesium through the original technique of hydrostatic extrusion at room temperature has been demonstrated. The microstructure and the mechanical properties of the extruded magnesium rods have been investigated. The evolution of mechanical properties of the samples after treatments up to 450 °C has been analyzed. It has been shown that low-temperature treatments lead to some increase in strength and plasticity of pre-deformed magnesium. The annealing hardening phenomenon is explained by the thermally activated processes of rearrangement of the dislocation structure and blocking of dislocations.

1. Introduction

It is well known that at temperatures below 225 °C the workability of pure magnesium and magnesium alloys is limited or even not possible [1]. Poor room-temperature formability of magnesium is connected with its hexagonal close packed (hcp) crystalline structure in which the basal slip system dominates room-temperature deformation [2]. At high temperature formation, the prismatic and pyramidal slip systems are activated and thus the plasticity of magnesium increases.

Grain refinement and texture modification are both considered to contribute to the improvement of plasticity of magnesium. As a rule, different severe plastic deformation (SPD) techniques are used for this purpose [3,4]. A detailed review of studies, which used the well-known SPD method of equal channel angular extrusion (ECAE) for deformation of magnesium, was given in [4]. It was shown that the deformation of magnesium by the ECAE method is much impeded at lower temperatures. Nevertheless, over and over again, researchers have attempted to deform magnesium at room temperature in order to obtain ultrafine-grain structure (UFG) with high strength and ductility. A UFG material is stronger than one in a coarse grained state because of grain size hardening according to the Hall–Petch relationship [2,5]. In addition, UFG magnesium has a better ductility as well as a low ductile to brittle transition temperature; thus, their formability at room temperature can be improved [6].

Undesirable cracking of magnesium during deformation may be avoided even at room temperature if processing by high-pressure torsion is used [7]. In this case, formation of the magnesium sample occurs within the field of compression stresses. As a result, pores, defects, and cracks existing in the material cannot develop. Therefore, it is necessary to provide all-around compression stresses for the successful deformation of materials with poor plasticity. Of course, it was well known before [8].

Recently, we used this method for deformation of bulk magnesium workpiece at room temperature [9]. For this purpose a magnesium cylinder of 20 mm diameter and 30 mm height was placed in a thick-walled copper container. The deformation was carried out by squeezing this “assembly” at room temperature. Such a method of deformation generates high compression stresses on the side surfaces of the magnesium workpiece. They are caused by the response of the container walls to the extension of the sample during pressing.

At the final stage of the experiment the remains of the copper container were cut and removed. After processing by squeezing into the container the magnesium sample was significantly shorter and bigger in diameter, having the form of a puck. The degree of true (or logarithmic) deformation (ε) of magnesium reached $\varepsilon \sim 0.85$. No sign of destruction of the magnesium sample was observed. As the structure became virtually isotropic and the grain size decreased to 5 μm, the plasticity of magnesium increased. A very interesting phenomenon was revealed during these experiments. The yield strength of the pre-deformed magnesium samples does not drop but increases as a result of low-temperature annealing. Obviously, this phenomenon is of both fundamental and practical interest. In addition, obviously, this result needs verifying.

These experiments showed that the use of a container increases the formability of magnesium at room temperature.

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http://dx.doi.org/10.1016/j.msea.2014.12.104

This is caused by the formation of compression stresses around the sample during deformation. It seems interesting to use a container to increase the formability of magnesium with other methods of SPD. For example, magnesium placed in a shell can be deformed by hydroextrusion.

A great variety of sources did not produce any description of successful experiments of hydrostatic extrusion of pure magnesium at room temperature. Moreover, magnesium alloys are very difficult to be cold extruded, although they have better formability than pure magnesium. As shown in [10], hydrostatic extrusion of magnesium alloys at temperatures below 100 °C leads to the destruction of workpieces. The purpose of this study was to develop a hydrostatic extrusion technique for magnesium at room temperature, followed by an investigation of the structure and mechanical properties of the samples in the obtained structural state.

2. Material and method

As a material for experiment, an ingot of commercially pure magnesium (99.98%) was chosen. The investigation has shown that the ingot consists of very large grains (Fig. 1). The largest grains are disposed in the centre of the ingot and they are more than 30 mm in length and near 5 mm in width. Such coarse grained structure is typical for magnesium ingot (see, for example, [11]). Cylindrical workpieces of 10 mm in diameter and 70 mm in length for hydrostatic extrusion experiments were cut out of this ingot.

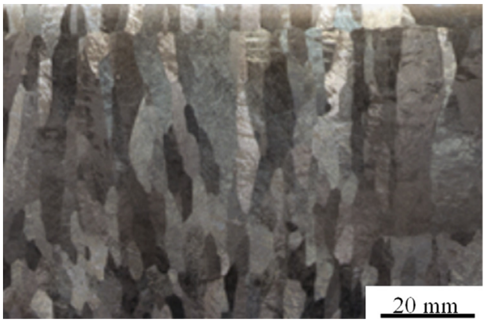

Fig. 1. Macrostructure of initial as-cast magnesium.

The result of our experiment using a conventional technique is shown in Fig. 2a. We are far from being the first who observed magnesium workpieces cracking after SPD at room temperature (see, for example, [10–13]). To prevent cracking of the magnesium during its flowing out from the die, we developed an original method (Fig. 2b). We placed a magnesium workpiece (1) of 10 mm in diameter in a shell (2) of 20 mm in diameter. Hydrostatic extrusion was made through a die (3) of 10 mm at room temperature. The distinction of this method consists in the material of the shell.

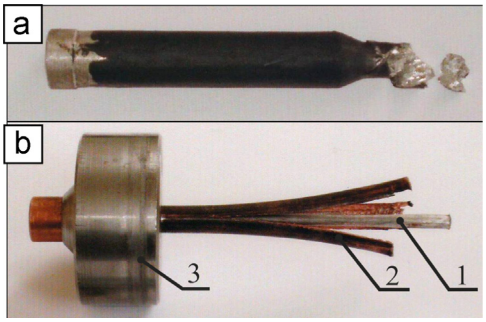

Fig. 2. Appearance of magnesium after hydrostatic extrusion at room temperature: (a) a destroyed workpiece after experiment using conventional technique and (b) a successful experiment with workpiece (1) which was placed in a self-destroying shell (2) and passed through the die (3).

As was described above, we carried out successful squeeze of magnesium workpiece placed in a copper container before [9]. Nevertheless, this method had a disadvantage. When the deformation process was finished, we had to cut the container to take the sample out of it. In our experiments on hydroextrusion, we replaced copper by another material. It is clearly seen in Fig. 2b that the shell (2) breaks down once hydrostatic extrusion is completed. It provides us an easy way to extract the sample out of the shell. The material of the shell is specialized knowledge ("know-how" of the Institute of Metal Physics of the Russian Academy of Sciences). As the result of the employment of this technique, a long magnesium rod of ~5 mm in diameter was formed without any flaws (Fig. 2b).

Microstructure observations were realized by means of a scanning electron microscopy (SEM) instrument QUANTA 200 FEI. Tensile mechanical tests were carried out at room temperature using an Instron test instrument. The length of the samples was 50 mm and the strain rate was $2\times10^{-3}\,s^{-1}$. Five samples were tested per each experimental point. All of the heat treatments were carried out in evacuated glass ampoules. Samples were subjected to annealing in the temperature interval from 100 to 450 °C for 6 h followed by cooling in water.

3. Results

3.1. Microstructure of the extruded rod

After the first step of hydroextrusion processing we observed grains of $d\approx3$–5 μm in the microstructure of Ø5 mm rod (Fig. 3a). The largest grains attain 15 μm in size; they are noted in the central part of this rod.

The magnesium rod in the obtained structural state has high plasticity. For example, samples for mechanical tests of 2 mm in diameter were made by hydroextrusion processing of magnesium rod Ø5 mm through two passes at room temperature without any shell. First, rods of 3 mm diameter were obtained. Then, they underwent hydrostatic extrusion again through a die of Ø2 mm. So, the total degree of true deformation (ε) of this magnesium sample was $\varepsilon\sim3.2$.

After deformation to Ø2 mm, the structure becomes homogenous with grain size $d\approx2$–3 μm (Fig. 3b). As it can be seen, in this case the refinement of the initial cast structure by more than three orders of magnitude takes place (we may compare Fig. 1 with Fig. 3b). Small grain formation in highly deformed magnesium is caused by dynamic recrystallization [1,13,14]. The values of grain sizes obtained can be compared to the minimal values cited in the literature [2–5]. An average grain size of 6–8 μm was achieved by ECAE of commercially pure magnesium at 250 °C after four passes [6]. As was found in [4], the minimum average grain size obtained after ECAE processing was not less than ~2 μm.

3.2. Mechanical properties

True stress (σ) versus elongation (δ) responses of magnesium samples with 2 mm in diameter are plotted in Fig. 4a. It is seen that magnesium samples after hydrostatic extrusion at $\varepsilon\sim3.2$ following the developed technique have a very high plasticity. For example, the elongation-to-failure of such samples is $\delta=19\%$ (curve 1 in Fig. 4a).

The mechanical properties of samples that we obtained by our technique can be compared to the mechanical properties of those reported in the literature. For example, in the rods of 2 mm in