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Compressive yield stress improvement using thermomechanical treatment of extruded Mg-Zn-Ca alloy

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ABSTRACT

Thermomechanical treatment consisting of pre-compression and a subsequent isothermal aging was used to improve the compressive properties of extruded Mg-Zn-Ca (ZX10) alloy. Isothermal aging at 150 °C and 200 °C was performed on as-extruded samples for various times to find peak aged conditions. To achieve a different twin volume fraction in the alloy, samples were pre-compressed up to 2%, 3% and 4% along the extrusion direction. Afterwards, the peak-aged conditions were applied to the pre-compressed samples to enhance their mechanical properties. It was found that after the 4 h@150 °C thermomechanical treatment, deformation curves exhibit a distinctive yield plateau independently on the pre-strain level. Using the 2 h@200 °C condition, the yield plateau occurs only after pre-strain of 3%. Higher compressive yield strength values were observed for the thermomechanically treated samples at the lower temperature.

1. Introduction

Thermomechanical treatment of extruded Mg alloys, consisting of pre-compression and a subsequent isothermal aging, is one of the ways to improve their mechanical properties. This metallurgical process can bring benefits from two different processing methods, whereas resulting mechanical properties cannot be estimated as a sum of contributions observed during separate processing routes.

An influence of temperature and alloying elements on the mechanical properties of Mg-Zn-Ca alloys subjected to the isothermal aging was intensively studied. It was shown in [1] that an addition of Zn in Mg-Ca alloy increases the hardness of the alloy. Precipitates in such Mg alloys are formed as basal plates, what with their fine distribution contributes to higher hardness and strengthening of the alloys [2–4]. However, strengthening via Mg_2Ca precipitates formed in basal planes is very low. A comprehensive review of precipitation and hardening in Mg alloys can be found in [5]. Higher content of Zn in the alloys contributes to a grain refinement, and thus enhances their mechanical properties [6–11]. Using Mg alloys with an ultrahigh purity can lead to a development of fine-grained high-strength alloy Mg [12].

Pre-twinning in textured Mg alloys chances the orientation of specific grains with respect to the loading direction [13] and sub-divides those grains. Thus, texture and refinement hardening enhance the strength of the alloys [14–16]. However, thermomechanical treatment is not very often used for Mg alloys, especially for those with a low content of alloying elements, and therefore, there is little literature evidence. An effect of heat treatment and pre-deformation on the damping capacity of cast binary Mg-Y alloys can be found in [17] and mechanical properties of an as-cast Mg-0.4Sm-1.0Ca alloy are presented in [18].

Wrought Mg alloys are commonly used in automotive industry where achieving proper mechanical properties is based on using a variety of alloying elements without any requirement on their biocompatibility. Nowadays, there is a high demand for fully biocompatible and biodegradable Mg alloys, which can be also used for biomedical applications. Based on that, Mg-Zn-Ca alloys are considered as promising biodegradable materials due to a presence of biocompatible Zn and Ca alloying elements [19–22].

The goal of the present study is to improve the compressive yield strength (CYS) of the Mg-Zn-Ca alloy using pre-compression and a subsequent isothermal aging. A low content of Zn and Ca in the alloy cannot adequately increase CYS only by heat treatment and thus, thermomechanical processing opens a window to enhanced compressive mechanical properties. To achieve the goal, both an appropriate level of pre-compression and optimized heat treatment conditions have to be found.

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2. Experimental procedure

The Mg-Zn-Ca (ZX10 - Mg + 0.9 wt% Zn + 0.25 wt% Ca) alloy was prepared by a modified gravity casting process with directional solidification by lowering the crucible into a water bath. After machining, the billets were solid-solution annealed for 20 h at 400 °C. Indirect extrusion to a round bar (extrusion ratio 1:25) with a final diameter of 10 mm was carried out at 400 °C with an extrusion speed of 0.5 mm/s (ram speed, corresponding profile exit speed 0.75 m/min).

Global texture information about the extruded bar was obtained by using X-ray diffraction (XRD). A Panalytical X-ray diffractometer setup using CuKα radiation was employed to measure pole figures on polished samples in reflection geometry to a sample tilt of 70°.

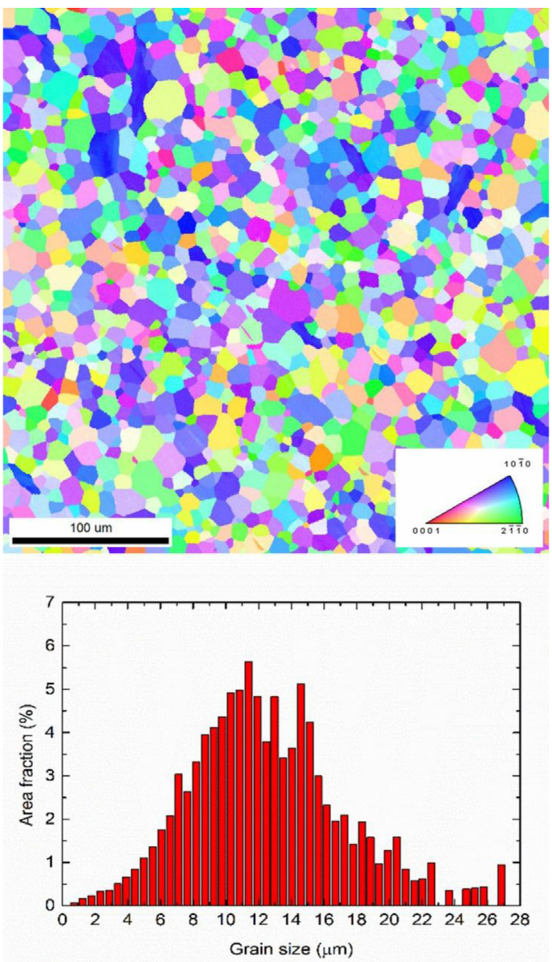
Specimens with a diameter of 9.5 mm and a gauge length of 14 mm were machined from the round extruded bar parallel to the extrusion direction (ED). Compression tests were carried out using an Instron 5882 universal testing machine at room temperature (RT) and an initial strain rate of 10^{-3} s^{-1}. From the compressive stress-strain diagram for the as-extruded sample, levels of pre-compression: 130 MPa (2% of pre-strain), 140 MPa (3% of pre-strain) and 155 MPa (4% of pre-strain), respectively, were revealed. The experiments were repeated to those strains in order to produce samples with a different volume fraction of twins in the alloy. In this way, a strengthening around the yield point based on pre-compression was studied by comparing the subsequent compression behavior with and without additional heat treatment on the samples. Particularly, for this study, isothermal aging at 150 °C or 200 °C was used.

Various isothermal aging times were applied for revealing the peak-aged condition. A fully automatic Qness Q10 microhardness tester enabling the Vickers hardness measurement (HV0.1) was used. The microhardness was determined on mechanically polished cross-section (perpendicular to ED) of the round bar. For samples in each condition, an average hardness value is based on 100 measurements (a grid of 10 × 10 points).

Electron backscatter diffraction (EBSD) was used to analyze the microstructure of the samples on their cross-sections with a field emission gun scanning electron microscope (SEM, Zeiss Auriga, EDAX/TSL EBSD system). The samples were ground using SiC paper and subsequently polished by diamond pastes down to 0.25 µm particle size. Finally, the surface of the samples was polished by Ag ion milling (Leica EM RES102). EBSD maps were measured with a step size of 0.3 µm. A software TSL OIM Analysis was used for the analysis of EBSD results: orientation and kernel average misorientation (KAM) maps. The identification of point-to-point misorientations was used to identify grain- and twin boundaries. A twin area analysis was carried out manually by selecting identified twins. In [23] it was shown that the KAM values represent local strain in materials. The KAM values were calculated as the average misorientation between the data point and all first nearest neighbors with a 5° maximum misorientation.

Transmission electron microscopy (TEM) was performed using JEOL JEM-2200FS operated at 200 kV. The foils were first mechanically polished and then the final thinning was performed using the ion milling system Leica EM RES102.

a



b

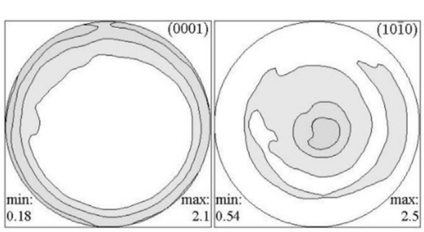


Fig. 1. Initial microstructure and texture of the extruded ZX10 alloy: a) orientation map with ED perpendicular to the image and the grain size distribution, b) pole figures with ED in the center.

3. Experimental results

The initial microstructure of the extruded ZX10 alloy (Fig. 1a) is homogeneous and fully recrystallized with an average grain size of (11 ± 1) µm. Pole figures obtained by X-ray diffraction (Fig. 1b) reveal an alignment of basal planes parallel to ED and weak prismatic fiber-texture, which is often found in extruded Mg alloys.

The evolution of the microhardness (HV0.1) as a function of aging temperature and time is presented in Fig. 2. Isothermal aging at 150 °C

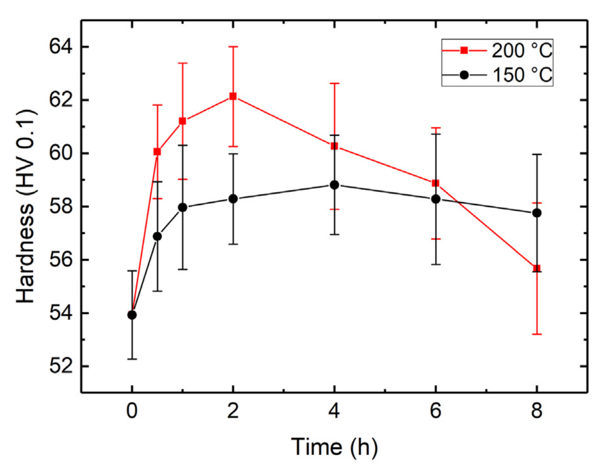


Fig. 2. Dependence of Vickers hardness on holding time for isothermal aging at 150 °C and 200 °C.