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Microstructure and mechanical properties of Mg-Nd-Zn-Zr alloy processed by integrated extrusion and equal channel angular pressing

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ABSTRACT

The ultrafine-grained Mg-Nd-Zn-Zr alloy was obtained by an integrated extrusion and equal channel angular pressing at 350 °C with one pass. Microstructural observations showed a significant refinement of grain structure after a single pass extrusion. The average grain size was reduced to around 500 nm. Numerous β_1 phases and GP zones were observed in the extruded and subsequently aged alloy. A weak fiber texture with $\langle 10\bar{1}0\rangle$ tilting angle of ~30° from normal direction towards extrusion direction was found in the extruded alloy. The extruded and subsequently aged alloy presented similar texture to that of the extruded alloy. The extruded alloy exhibited the yield strength (YS), ultimate tensile strength (UTS) and elongation of 248 MPa, 288 MPa and 14.4%, respectively. The extruded alloy after aging treatment achieved higher strength and slightly lower ductility with the YS, UTS and elongation of 264 MPa, 307 MPa and 12.9%, respectively. Compared to the as-cast alloy, the mechanical properties of the extruded and subsequently aged alloy increased 158%, 65% and 34% in YS, UTS and elongation, respectively. The increase in the strength of the extruded and subsequently aged alloy was attributed to both the grain refinement and the precipitation strengthening. The fracture surfaces of the extruded alloy were composed of a lot of small dimples and some cleavage planes. After the aging treatment, the cleavage planes were increased resulting in a decline in the ductility.

1. Introduction

Mg alloys have attracted considerable attention in the past decade because of their high specific strength, good damping characteristics, and other advantages [1–3]. However, Mg alloys exhibit poor mechanical properties at room temperature, which is ascribed to the limited slip systems in the hexagonal close-packed structure [4,5]. Severe plastic deformation (SPD) is recognized as a promising technique to refine the microstructure and improve the mechanical properties [6]. Up to now, several different SPD processing techniques have been extensively studied, including high-pressure torsion (HPT) [7], multi-axial forging (MAF) [8], accumulative roll-bonding (ARB) [9] and equal channel angular pressing (ECAP) [10]. Among these SPD techniques, ECAP is regarded as the most efficient SPD processing technique for the production of bulk ultrafine-grained Mg alloys with grain sizes below 1 μm [11]. However, ECAP is not widely used in industrial practices due to some limitations. For instance, the plunger of the press has a limited travel distance, which limits the length of workpiece [12]. Particularly, ECAP usually requires 4–10 passes to obtain a stable uniform grain size. This suggests that ECAP is not a continuous process, making it difficult for industrial applications. Furthermore, the texture induced by ECAP promotes basal slip in Mg alloys, which reduces the strength.

To alleviate the above mentioned problems, the extrusion and equal channel angular pressing should be performed in one processing chain. Orlov [13–15] proposed a semi-continuous SPD technique that was characterized as an integrated process combining conventional extrusion and ECAP in a single processing step. This process was applied to the production of ZK60 Mg alloy workpiece. The strength, ductility and corrosion resistance of extruded alloy have been significantly improved. It was attributed to the reduced grain size of extruded alloy was around 1.6 μm. Nevertheless, the ultrafine-grained microstructure with grain size below 1 μm was not obtained in their experiments. In order to further refine the microstructure, a greater shear strain should be achieved by reducing the channel angle of the extrusion die. As an age-hardenable Mg alloy, the Mg-Nd-Zn-Zr alloy has great potential for industrial applications, especially in the field of biodegradable

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implant materials such as stents, bone fixtures, plates, and screws [16]. Up to now, the influences of the aging treatment on microstructure, textures and mechanical properties of the ultrafine-grained Mg-Nd-Zn-Zr alloy were not clear.

In this work, the process of the integrated extrusion and equal channel angular pressing (IEECAP) is modified to produce the ultrafine-grained Mg-Nd-Zn-Zr alloy with grain size close to 500 nm. For this purpose, several ECAP passes are reduced to one pass and the angle Φ between the channels is reduced to 90°. The effect of IEECAP and the subsequent aging treatment on microstructure, textures and mechanical properties of the Mg-Nd-Zn-Zr alloy is investigated. A systematical understanding the relationship between the microstructure and mechanical properties is provided.

2. Experimental materials and methods

2.1. Experimental material and processing

The alloy with target compositions of Mg-3.0Nd-0.4Zn-0.5Zr (wt.%) was prepared using commercially pure Mg (>99.95 wt.%), pure Zn (>99.9 wt.%), Mg-25 wt.%Nd and Mg-30 wt.%Zr master alloy ingots. The materials were melted at ~780 °C in an electric resistance furnace under a mixed atmosphere of CO_2 and SF_6 with a volume ratio of 200:1. The liquid melt was stirred to ensure the homogeneity and then cast into a steel permanent mold with preheated temperature at 200 °C. The as-cast alloy ingots were solution-treated at 530 °C for 8 h covering with graphite powder. The solution-treated alloy ingots were cut into rectangular billets with dimensions of 30 mm × 30 mm × 100 mm. In order to minimize the effect of grain growth during IEECAP process and to minimize the load on the plunger, the solution-treated billets were extruded at 350 °C. The rectangular billets were extruded to bars with dimensions of 10 mm × 10 mm. Graphite powders were used as the lubricant. The plunger speed is 3 mm s^{-1}. The extruded specimens were aged at 200 °C for 4 h. The solution-treated specimens without extrusion were also aged at 200 °C for 4 h for comparisons. The schematics of IEECAP die and the corresponding workpiece extracted from the die after a single pass extrusion are shown in Fig. 1. The iso-view and side-view of IEECAP die present in Fig. 1(a) and (b), respectively. The extrusion, normal and transverse directions are abbreviated as ED, ND and TD, respectively. The angle Φ of the IEECAP die is 90°. As revealed in Fig. 1(c), the IEECAPed bar with smooth surfaces was achieved.

2.2. Characterization of microstructures, textures and mechanical properties

The specimens for the microstructure characterization were cut along the ND-ED plane. To analyze the microstructures of the as-cast, the solution-treated and the aged alloys, the specimens were polished and etched with a solution of 2.5 g picric acid, 2.5 ml acetic acid, 50 ml ethanol and 50 ml water, and observed by an optical microscope (OLYMPUS GX71). The TEM (JEM-2100, 200 kV) experiments were conducted to observe the precipitated morphologies. The TEM specimens were mechanically grounded to ~40 μm, and then prepared by ion milling operating at ~3.6 kV ion gun energy and ~4° milling angle. The fracture surface morphologies of specimens after tensile tests were observed by a scanning electron microscope (FEI Quanta 200). The average grain size was determined by the linear intercept method. For measurements of grain sizes, at least 10 images, including TEM and optical images, were repeated for each condition to improve precision.

The texture measurements were conducted using a X-ray diffractometer (X'Pert-PRO) equipped with Cu K_α at 40 kV and 40 mA. The specimens for the texture characterization were cut along the ND-ED plane. The texture measurements were done over the specimens tilting from 0° to 70° and azimuthal rotating from 2.5° to 357.5° with 5° steps in both directions. The {0002} and $\{10\bar{1}0\}$ pole figures were analyzed by X'Pert texture software.

The mechanical properties were characterized by tensile tests, which were carried out at room temperature with a strain rate of

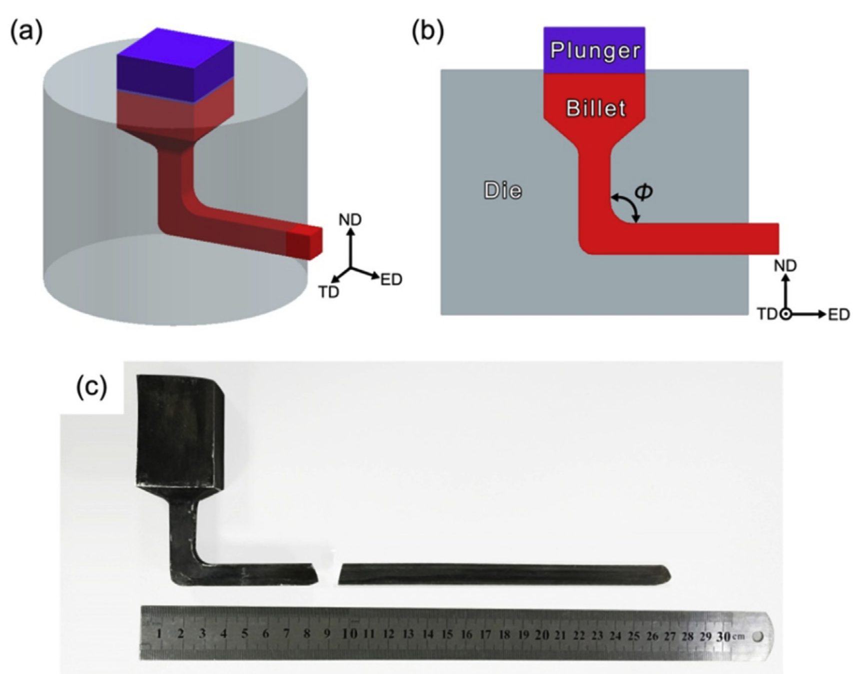

Fig. 1. Schematics of IEECAP die and the corresponding workpiece extracted from the die after a single pass extrusion, (a) iso-view, (b) side-view and (c) macrograph of the workpiece processed by IEECAP.