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Enhanced age-hardening response and compression property of a Mg-7Sn-1Ca-1Ag (wt.%) alloy by extrusion combined with aging treatment

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ABSTRACT

The age-hardening response and compression property of a Mg-7Sn-1Ca-1Ag (wt.%) alloy are improved by extrusion combined with aging treatment (abbreviated as extruded-aged). The corresponding microstructure evolution was investigated and discussed in detail. The results show that the average grain size of alloy was refined obviously from ~54.6 μm to ~3.2 μm by extrusion at 320 °C and the refinement of CaMgSn phase is also exhibited. Extrusion process promotes the precipitation of Mg_2Sn and $Mg_{54}Ag_{17}$ phase and more precipitates of Mg_2Sn phase exists in the extruded-aged alloy. As a result, age-hardening response and compressive strength are significantly enhanced in the extruded-aged Mg-7Sn-1Ca-1Ag alloy. The peak-aged hardness and ultimate compressive strength (UCS) of extruded-aged Mg-7Sn-1Ca-1Ag alloy are 95.2 HV and 482 MPa, respectively, both of which are obviously higher than that of as-aged and as-extruded alloys. This is mainly attributed to the grain refinement, formation of $Mg_{54}Ag_{17}$ phase and the increased content of finer Mg_2Sn precipitates. Moreover, it was found that an obvious yield behavior occurs in compressive engineering stress versus strain of extruded and extruded-aged alloys, which is mainly attributed to the basal texture caused by extrusion and high value of Schmid factor, leading to the easy formation of {10−12} twinning in the extruded-aged alloy during compression along the extrusion direction.

1. Introduction

Magnesium alloys are among the lightest metallic materials used for applications in different fields, such as aerospace, automobile and armament applications [1]. Improving the strength of magnesium alloys has been one of the most important goals in recent years. Meanwhile, magnesium alloys exhibit a low ductility, which can be detrimental for metal forming operations. Therefore, the improvement of ductility for Mg alloys also should be focused on the research in future. Generally, the strengthening mechanism of Mg alloys is attributed to multiple aspects, including grain boundaries, texture, dislocations, solid-solution, and precipitates [2]. Heat-treatment and extrusion treatment are two major methods to improve the strength of magnesium alloys, which can produce significant precipitation hardening and finer grain strengthening effects.

At present, aged Mg-Sn alloy has obtained a noticeable mechanical property at different temperatures relying on dispersive Mg_2Sn precipitates [3–7]. Various elements (Zn [4,6–8], Al [6,9], Na [4], Ag [5]) addition is beneficial to improve the strength of Mg-Sn alloy through corresponding dispersive precipitates. It has been indicated that Ca addition can enhance the strength of Mg-Sn alloys due to the formation of CaMgSn and Mg_2Ca or Mg_2Sn phase, which is particularly beneficial to the strength at elevated temperatures [10,11]. Suresh et al. demonstrated that the ultimate compressive strength of as-cast Mg-3Sn-2Ca (wt.%) is the highest among all the ternary Mg-Sn-Ca alloys studied at 25 °C. Furthermore, the ultimate compressive strength of Mg-3Sn-2Ca (wt.%) alloy has been improved from ~200 MPa to ~230 MPa with Al + Zn addition due to the formation of Al_2Ca, CaMgZn phase and the refinement of CaMgSn phase [12]. However, the coarse CaMgSn phase in the Mg-Sn-Ca (wt.%) alloy will gives a detrimental effect on the mechanical properties [12,13]. Therefore, refining Mg_2Sn phase and CaMgSn phase is an important research aspect to further improve the strength of Mg-Sn-Ca alloys. Meanwhile, Huang et al. confirmed that Ag element is beneficial to form dispersive Mg_2Sn precipitates

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so that Ag addition obviously improves the age-hardening response of Mg-7Sn alloy [14]. Feng et al. reported that the Ag-containing extruded Mg-3Al-1Zn alloy also exhibited an enhancement in the tensile and compressive properties. But the strength of extruded Mg-3Al-1Zn alloy decreased when the Ag addition reached to 2 wt.% [2]. In addition, the increased content of Ag leads to the increased cost of alloys. According to the refined CaMgSn phase and dispersive Mg_2Sn precipitates, the present authors has designed a Mg-7Sn-1Ca-1Ag (wt.%) alloy and obtained a noticeable age-hardening response. The Vickers hardness of Mg-7Sn-1Ca-1Ag alloy reaches to peak-aged (~80HV) after aging at 200 °C for 140 h due to dispersive Mg_2Sn precipitates and refined CaMgSn phase [15].

It has been shown that extrusion treatment is an effective way to improve the strength of Mg-7Sn alloys. The reason can be attributed to the refinement of grain size by dynamic recrystallization and the dynamic precipitation of Mg_2Sn phase under extrusion deformation [8]. Extrusion treatment can refine the grain size so that a higher yield strength is obtained according to the Hall-Petch equation ($\sigma_y = \sigma_o + Kd^{-1/2}$) [16–18]. In addition, Huang et al. has reported that the extrusion and Ag or Ag + Zn addition in Mg-7Sn alloy can significantly improves the hardness due to the dynamic precipitation of Mg_2Sn phase. Furthermore, CaMgSn laths will be broken into finer phase during the extrusion process [19,20], which is probably beneficial to obtain a better mechanical property compared with coarse CaMgSn phase [21].

To increase the number density of Mg_2Sn precipitates in the extruded Mg-7Sn alloy, a reasonable aging treatment is necessary to perform on the extruded Mg-7Sn alloy. More nucleation sites for dispersive Mg_2Sn precipitates can be provided by a large amount of dislocations and twins produced during extrusion process [22]. Hence, Mg_2Sn precipitates are easier to form after extrusion treatment. More and finer Mg_2Sn precipitates probably have a better effect on preventing dislocations slipping, resulting in the improvement of strength. Therefore, it can be expected that a noticeable comprehensive property will be obtained for as-extruded Mg-7Sn-1Ca-1Ag alloy after aging treatment.

According to the strengthening mechanism of magnesium alloys, the second phase formation and grain refinement are two important methods to achieve noticeable mechanical properties. Especially, dispersive precipitates play significant roles in improving the strength of magnesium alloys, such as Mg_2Sn [23], Mg_2Zn [7], $Mg_{17}Al_{12}$ [24]. The effect of extrusion combined aging treatment on the mechanical properties and microstructure of Mg-7Sn-1Ca-1Ag alloy has not been investigated. Therefore, the present study investigates the microstructure characteristics and aging behaviors of Mg-7Sn-1Ca-1Ag alloy processed by extrusion and extrusion combined with aging treatment. Furthermore, the compressive properties of alloys also are examined. It is believed that the microstructure and properties of Mg-7Sn-1Ca-1Ag alloy after extrusion or extrusion combined aging treatment can be further understood from the present work.

2. Experiment

The alloy with a nominal composition of Mg-7Sn-1Ca-1Ag (wt.%) was investigated in this study. The alloy was melted from high pure Mg (99.99%), Ag (99.99%), Sn (99.95%) and Mg-25%Ca (wt.%) master alloys at 750 °C in the electrical resistance furnace and then held at 720 °C for 20 min with the protection of CO_2 (99%) and SF_6 (1%) mixture gas. The melt was stirred well and poured into a steel mold preheated to 200 °C. The cast alloys were homogenized at 400 °C for 16 h and then increased to 460 °C for 4 h to be solution treated with the protection of a Ar atmosphere followed by water quenching. Then, the solution-treated alloy was extruded with a extrusion ratio of 36 at 320 °C into a bar with a diameter of 4.2 mm, which is called as-extruded state. Previous report indicated that the solution-treated alloy was aged at 200 °C for 140 h to reach peak-aged state [15]. Therefore, the solution-treated alloy and as-extruded alloy were aged at 200 °C for various times to investigate the age-hardening response. Micro-hardness measurements were carried out on a micro-Vickers testing machine under a load of 200 g (1.96 N) for 15 s. The average hardness value (HV) was calculated by 10 individual indentations. The as-cast, as-aged, as-extruded and extruded-aged alloys were machined into cylindrical specimens with a diameter of 4 mm and a length of 8 mm to investigate the compressive properties. The compressive tests were carried at a constant cross-head speed corresponding to an initial strain rate of about 1×10^{-3} s^{-1} to obtain compressive properties, including ultimate compressive strength (UCS), compressive yield strength (CYS) and compressive elongation (CEL), which is defined as fracture elongation and the quantity of CLE is measured by the value of strain corresponding to ultimate strength, and compression curves in the universal test machine (DDL-100). The 0.2% offset strain was used as a measurement of the yield stress of the investigated alloys [25].

The microstructure of experiment alloys was characterized using optical microscope (OM), scanning electron-microscope (SEM) equipped with energy dispersive spectroscope (EDS), Electron Backscatter Diffraction (EBSD) and transmission electron microscopy (TEM). The phase constituents of the investigated alloys were determined by the X-ray diffraction (XRD) and EDS results. The grain size was measured by the linear intercept method according to E112-96 following the ASTM standard. The scanning step of EBSD was 0.5 μm and the examined specimens of EBSD were taken from as-aged Mg-7Sn-1Ca-1Ag alloy with 7% engineering strain and extruded-aged Mg-7Sn-1Ca-1Ag alloy both with 0% and 7% engineering strain during compression, which depended on the completion of the yield behavior. TEM results were used to analyze the dispersive precipitates and the precipitates morphology in as-aged Mg-7Sn-1Ca-1Ag alloy and extruded-aged Mg-7Sn-1Ca-1Ag alloy. TEM specimens were prepared by twin-jet electropolishing at (−40°) at a voltage of 95 Vin a mixed solution of 250 ml methanol, 50 ml ethylene glycol monobutyl ether, 5.6 g $Mg(ClO_4)_2$ and 2.6 g LiCl.

3. Results and discussions

3.1. Microstructure and phase constituents of the investigated alloys

The microstructure images of as-cast, solution-treated, as-aged, as-extruded and extruded-aged Mg-7Sn-1Ca-1Ag alloys are given in Fig. 1. Fig. 1a shows that the as-cast Mg-7Sn-1Ca-1Ag alloy composes of α-Mg, Mg_2Sn phase and CaMgSn phase. It was observed that black Mg_2Sn phase exists along the grain boundaries and lath-CaMgSn phases are distributed both in grains and along boundaries in the as-cast Mg-7Sn-1Ca-1Ag alloy. After solutioned at 460 °C for 4 h, the coarse Mg_2Sn phases in the grain boundaries disappear absolutely due to its entire solution at 460 °C in α-Mg matrix in Mg-7Sn (wt.%) alloy according to the Mg-Sn binary diagram [26]. But the CaMgSn phases still exist due to its higher thermal stability and still keep the same morphology as that in as-cast alloy, as shown in Fig. 1b. After extrusion treatment, the optical microstructure of extruded alloys (parallel planes to extrusion direction (ED) and radial direction (RD)) shows that both grains and CaMgSn phases are refined. The average grain size decreases from ~54.6 μm to ~3.2 μm (Table 1). The grain refinement of α-Mg matrix is mainly attributed to dynamical recrystallization (DRX) and grain pinning effect of precipitates during hot deformation [9,27]. In