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Effects of solid solution treatment and cooling on morphology of LPSO phase and precipitation hardening behavior of Mg−Dy−Ni alloy

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Abstract: Effects of solid solution treatment and cooling on the morphology of long period stacking order (LPSO) phase and precipitation hardening behavior of Mg−2Dy−0.5Ni (molar fraction, %) alloy were investigated. Microstructures of the as-cast alloy mainly consisted of α-Mg phase, bamboo-like $Mg_{12}DyNi$ phase with LPSO structure distributed between dendrites and small amounts of cubic Dy phases. During solid solution treatment at 565 °C for 12 h and subsequent different cooling conditions, dot-shaped, block, fine lamellar and rod-shaped LPSO phases precipitate in Mg matrix, respectively. For continuous cooling conditions (furnace and air cooling), the fine lamellar LPSO phase generally forms in grain interior and its volume fraction increases and block LPSO phase coarsens with increasing cooling time. For discontinuous cooling conditions (air cooling after furnace cooling to 415 and 265 °C), the dot-shaped LPSO grows into the rod-shaped phase, which results in an decrease of cooling hardening behavior of alloy.

Key words: Mg−Dy−Ni alloy; LPSO phase; microstructure; precipitation hardening

1 Introduction

Magnesium (Mg) alloys are one of the lowest density metal structure materials, and parts of them have been widely applied to the automotive fields due to their high specific stiffness, good damping capacity and machinability [1]. However, poor heat resistance of Mg alloy still limits their development in some industry fields. Thus, it is a significant issue to develop new kinds of heat resistance Mg alloys to expand their application [2−5].

Recently, Mg-based alloys with long-period stacking ordered (LPSO) structures have received considerable interest [6−12]. KAWAMURA et al [6] reported that the tensile yield strength and elongation of a $Mg_{97}Zn_1Y_2$ (molar fraction, %) alloy prepared by rapid solidification processing and hot extrusion can reach 610 MPa and ~5%, respectively. These excellent mechanical properties are manly ascribed to the precipitation of dispersed nano-scale LPSO phases [7]. The LPSO phase has a high thermal stability and good coherent interface with α-Mg matrix. Furthermore, the morphology, distribution, scale, volume fraction and structure of LPSO phase determine the heat resistance of alloy. At present, alloying, heat treatment and hot working could effectively modify the above properties of LPSO phase [13−16]. LIU et al [13] investigated that the addition of Zn effectively increased the volume fraction of LPSO phase in extruded Mg−5Y−4Gd−0.4Zr alloy. This phase plays an important role in improving the mechanical properties, especially for the elongation of alloy. WANG et al [14] found that the volume fraction of the LPSO phase of the $Mg_{94}Zn_{2.5}Y_{2.5}Mn_1$ alloy reached a peak (24.8%) with addition of 0.34% (molar fraction) Ca and the alloy exhibited an ultimate tensile strength of 231 MPa and a elongation of 8.6%, respectively. Additionally, ZHANG et al [15] reported that the block 18R-LPSO phase at the grain boundary transforms to 14H-LPSO phase in the grain interior for

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$Mg_{93.83}Zn_{1.5}Dy_{4.5}Zr_{0.17}$ alloy during aging-treatment and the tensile strength and elongation of the aging-treated alloy are 253 MPa and 10.5%, respectively. In addition, LU et al [16] demonstrated that the block, lamellar, and rod-shaped LPSO phase of $Mg_{95.2}Zn_2Y_{2.7}$ alloy could be obtained respectively by annealing heat treatment and the block LPSO phases are more conductive to the strength of the alloy. Additionally, hot working could refine LPSO phase and make the LPSO phase transform from 18R to 14H type, such as extrusion [17], hot rolling [18] and forging [19].

Previous literatures [16,20] have demonstrated that annealing heat treatment could obtain different morphology LPSO phases in Mg−Zn−Y alloys. However, the effect of continuous and discontinuous cooling on the morphology LPSO phase for Mg−Dy−Ni alloy has less been studied until now. At present, we have investigated the microstructure and mechanical properties of as-cast Mg−Dy−Ni alloy [21]. Therefore, in this work, the microstructure evolution and precipitation hardening behavior of the alloy during cooling were investigated. The transformation mechanism of morphology of LPSO phase during cooling was discussed.

2 Experimental

Mg−2Dy−0.5Ni (molar fraction, %) alloy was prepared from high pure Mg and Ni and Mg−20Dy (mass fraction, %) master alloy. Melting was conducted by using a graphite crucible in an electric resistance furnace at about 750 °C under the protection of antioxidant flux. The melts were homogenized at 720 °C for 0.5 h, and then cast into a steel mould with size of 70 mm × 40 mm × 13 mm. The specimens for solid-solution treatment were cut from the bulk as-cast ingot with size of 10 mm × 10 mm × 5 mm. The specimens were solution treated at 565 °C for 12 h in box resistance furnace of SX2-4-10 style and then quenched into water at room temperature. Then, the specimens after solid-solution treatment at 565 °C for 12 h were cooled at different cooling conditions which consist of air cooling after solid solution heat-treated state (S+A), furnace cooling after solid solution heat-treated state (S+F), air cooling after furnace cooling to 415 °C (S+415F+A) and cooling after furnace cooling to 265 °C (S+265F+A).

Microstructure, phase structure and phase composition of the alloy were characterized using optical microscopy (Olympus GX71), X-ray diffractometry (XRD) (Rigaku D/max 2500 PC), scanning electron microscopy (EDS) and transmission electronic microscopy (TEM) (JEM-2100F). Calorimetric response of the as-cast alloy was measured using differential scanning calorimetry (DSC). Specimens for microstructure observations were firstly ground on 400#, 600#, 800# and 1000# silicon carbide papers and etched after polishing in a solution of picric and 2% nitric acid. Phase analysis was conducted using X-ray diffractometer (XRD) with a copper target at scanning angles from 20° to 80° and scanning speed of 1.2 (°)/min. The thin foils with a diameter of 3 mm for the TEM observation were prepared using ion polishing system (RES101). The volume fraction of LPSO phase was measured by using Image-Pro Plus 6.0 and at least five OM/SEM images were analyzed for each state. Vickers hardness was measured by hardness tester (HV-100) with a load of 1 N, a dwelling time of 15 s and 10 measurements were collected for each sample.

3 Results and discussion

3.1 Microstructure of alloy in as-cast and solution heat-treated states

Figure 1 shows the DSC curve of as-cast alloy. The DSC heating curve contains two endothermic peaks. One peak is 570.2 °C which is close to the temperature of formation of the second phases. The other is at 600.2 °C which is close to the melting temperature of α-Mg according to the equilibrium phase diagram of the Mg−Dy alloy [22]. Therefore, the solution heat treatment of alloy was selected as 565 °C.

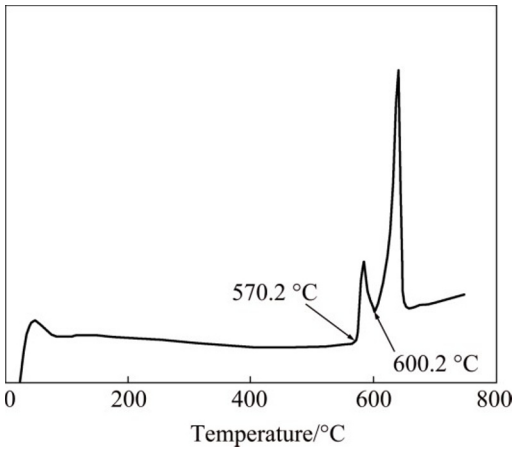

Fig. 1 DSC curve of alloy

The as-cast alloy is mainly composed of black α-Mg dendrites and second phases between dendrites, as shown in Fig. 2(a). These second phases are mainly composed of bamboo-like phase and a small amount of the square particle phases. EDS analysis results in Table 1 reveal that chemical compositions of black dendrites (point *A* in Fig. 2(a)), bamboo-like phase (point *B* in Fig. 2(a)) and cubic particle phase (point *C* in Fig. 2(a)) are Mg99.3−Dy0.7, Mg92.6−Dy4.4−Ni3.0 and Mg31.2−Dy66.5−Ni2.3 (molar fraction, %), respectively. According to previous investigation [21] and XRD results (Fig. 3),