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Microstructure characterization on Mg−2Nd−4Zn−1Zr alloy during heat treatment

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Abstract: The microstructures of Mg−2Nd−4Zn−1Zr alloy in the as-cast state and after heat treatment were investigated. Several kinds of secondary phases were found and characterized by transmission electron microscopy (TEM), scanning electron microscopy (SEM) and X-ray diffraction (XRD). In the as-cast alloy, the existing eutectic compounds are Mg−Nd−Zn ternary phases: T phases and W phases. After the heat treatment, with increasing the temperature or time, it was found that T phase almost dissolved into the α-Mg matrix, while a large amount of W phase remained in the matrix. On the other hand, with prolonging the time, the morphology of the phase changed from continuous network to the spherical shape along the grain boundary. The density of the W phase gradually decreased and finally it was coarsened and stabilized in the treatment process.

Key words: Mg–Nd–Zn alloy; heat treatment; microstructure; T phase; W phase

1 Introduction

The addition of rare-earth metals can improve the corrosion resistance, as well as the mechanical properties, especially the mechanical properties of magnesium alloys at elevated temperatures [1−5]. Many fundamental studies on developing new magnesium alloys containing RE elements have been conducted. Mg−RE−Zn−Zr alloys are widely used for their high strength, good plasticity and corrosion resistance [6,7]. Nd is considered an effective additional rare earth element. Addition of rational neodymium (Nd) to the Mg–Zn–Zr alloy can effectively improve the yield strength, hardness and ultimate tensile strength of the alloy at higher temperatures as a result of grain refinement and the formation of the $Mg_{12}Nd$ phase [8−10], which could make it possible to develop a high-strength and low-cost magnesium alloy.

NUTTALL et al [2] examined the metallography and kinetics of precipitation in Mg−2.8Nd−1.3Zn alloy and found the equilibrium γ phase. Recently, FU et al [11,12] studied in detail the microstructure and mechanical properties of gravity cast Mg−2.75Nd−xZn−Zr (x=0−2.0%) alloys and found that Mg−Nd alloy with less Zn contains $Mg_{12}Nd$ phase, while the Mg−Nd alloy with more Zn up to 2.0% contains a new Mg−Nd−Zn ternary phases (γ-phase) with FCC structure. Microstructure of Mg−Nd−Zn ternary phases and mechanical properties of Mg−Nd−Zn alloy with Zn content more than 4 were reported [13−15]. However, there is still lack of investigation on phase transformation of Zn-containing Mg−Nd alloy, especially when the alloy is heat treated at a higher temperature.

In the present work, attention is given to the phase constitution and microstructure of secondary phases in the as-cast and heat-treated Mg−Nd−Zn alloy. Considering the effects of Zn element, in order to investigate phase evolution the Zn was added into the Mg−2Nd−1Zr alloy. Specifically, the objective is to identify the secondary phases, to study their structure, morphology and composition in Mg−2Nd−4Zn−1Zr alloy subjected to different heat treated time at 500 °C, at different heat treated temperatures of 480−520 °C for 10 h. The structure of phases is examined using transmission electron microscopy (TEM).

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2 Experimental

The Mg–2Nd–4Zn–1Zr (mass fraction, %) alloy was prepared in an electrical resistance furnace using steel crucible with a protective gas consisting of SF_6 (1% in volume fraction) and CO_2 (Bal.) in order to prevent burning of the melts. Pure magnesium was melt in a steel crucible, with pure Zn (99.99%) added at temperature of 730−740 °C and then Nd and Zr were added as Mg−25%Nd and Mg−30%Zr master alloys at 760 °C. After stirring, the melt was held at 780 °C for 30 min to make sure that Zr was completely dissolved. JDMJ refine flux was used to reduce the loss of Nd element. The alloy was poured into a steel moulds holding at (200±5) °C. By inductively coupled plasma (ICP), the actual chemical composition of the alloy was determined as 1.95Nd−3.91Zn−0.92Zr- Bal Mg (mass fraction, %). The samples were annealed at 500 °C for 6−48 h and at 480−520 °C for 6 h followed by quenching in cold water.

The microstructures of the alloy were observed by using an optical microscope (OM) and a JEOL JSM−6460 scanning electron microscope (SEM) equipped with X-ray energy dispersive spectrometer (EDS). The phases in the as-cast and heat treated alloys were examined by D/Max 2550VL/PC X-ray diffraction (XRD). The detailed investigations were also performed on a TECNAI G^2 20F transmission electron microscope (TEM) operated at 200 kV. The TEM foils were prepared by a twin-jet electron polisher in a solution of ethanol with 1% perchloric acid at −30 °C, followed by ion beam thinning on a Gatan precision ion polisher for about 0.5 h with an incidence angle of 5°.

3 Results and discussion

3.1 As-cast microstructure and phase constitutions

The optical micrograph (OM) and scanning electron microscopy (SEM) image of the as-cast Mg−2Nd−4Zn−1Zr alloy are shown in Fig. 1. A typical dendritic solidification microstructure can be seen, and is comprised of interdendritic phases formed by the eutectic reaction and α-Mg matrix. A large number of intermetallic compounds distribute along the grain boundaries, and some of them appear as particles and exist inside the grains, as shown in Fig. 1(a). Figure 1(b) shows the magnified microstructure of secondary phases, which appear in a bone-like precipitates along the boundaries and are thickened.

XRD analysis on the as-cast alloy is given in Fig. 2. The peaks from the as-cast Mg−2Nd−4Zn−1Zr alloy can be indexed as α-Mg, and the others are unknown peaks. But the result shows that no distinct peaks belong to Mg12Nd phases, and the localization of others small diffraction peaks are well close to the T phase determined by WEI et al [6,7] and W phase reported by PADEZHNOVA et al [16] using XRD in Mg−Zn−Y alloy. In the as-cast Mg−2Nd−4Zn−1Zr alloy, combining XRD and EDS results intermetallic compounds here may be regarded as the T phase and W phase with both different kinds of Mg−Nd−Zn ternary phases. WEI et al [7] indicated that for the Mg−4Zn−1.5RE alloy, only T phase was found after solidification, and YANG et al [13] observed no W phases emerging in the Mg−2Nd−4.5Zn alloy. However, in this paper, it is interesting that W phase was found and cannot completely dissolve into the matrix after high temperature treatment in the as-cast

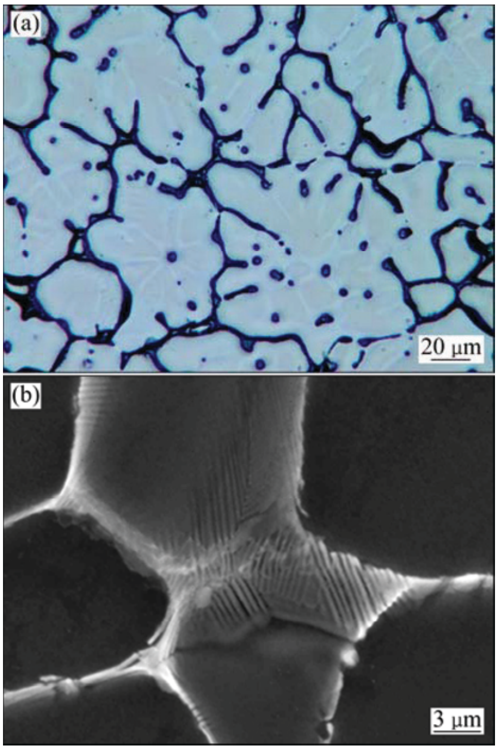

Fig. 1 Optical micrograph (OM) (a) and scanning electron microscopy image (b) of as-cast Mg−2Nd−4Zn−1Zr alloy etched with HNO_3−ethanol solution

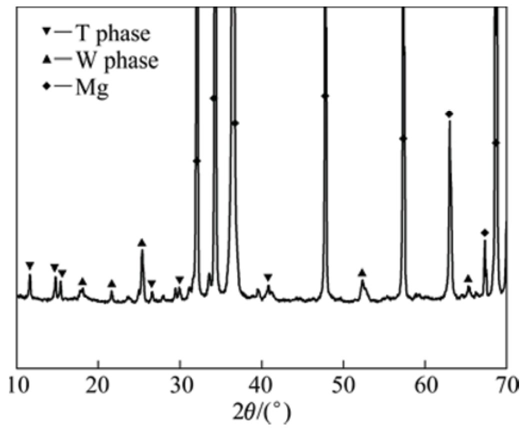

Fig. 2 XRD pattern of as-cast Mg−2Nd−4Zn−1Zr alloy