

Effect of Cryogenic Treatment on Microstructure of Mg-2Zn-0.7Ce Alloy

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Abstract. In this paper, the microstructure observation of Mg-2Zn-0.7Ce alloy before and after cryogenic treatment is presented. The microstructure of as-extruded alloy is composed of α -Mg and W phase. After cryogenic treatment for 1min, there are some secondary phase particles precipitating. With increasing cryogenic duration, the volume fraction of precipitates increases as well. After cryogenic treatment for 24h, the volume fraction of W phase increases from 0.2% to 7.3%. The precipitation mechanism of W phase is due to the contraction of Mg lattice under cryogenic environment, since it needs extra space as nucleation kinetics.

Keywords: Magnesium alloy; cryogenic treatment; microstructure.

1. Introduction

Magnesium alloys are the lightest structural materials for the industry of aircraft, spacecraft and missiles. However, the applications of magnesium alloys are strongly limited by their low ductility, poor corrosion resistance and weak wear resistance. Thus, an efficient is urgently needed to overcome such shortcoming of magnesium alloy [1-2]. Cryogenic treatment means the treatment of material under cryogenic temperature (-230°C-190°C). CT is widely applied in the manufacture of steel since the hardness and wear resistance of steel are significantly improved after cryogenic treatment. The investigation of cryogenic treatment on magnesium alloy is very significant, while rare studies are reported. Therefore, the present work investigates the effect of cryogenic treatment on the microstructure and mechanical properties of Mg-2Zn-0.7Ce alloy. As a systematic study to the precipitating behavior, microstructure evolution and mechanical properties of Mg alloy under cryogenic treatment, this work has presents a new way for the research of Mg alloy. In addition, a new process has also been suggested for Mg alloy, which may be with great significance and application prospects [3-4].

2. Experiments

The raw materials are mainly commercial pure Mg and pure Zn, with a purity of 99.9%. Ce is added in the form of a Mg-25wt.% Ce intermediate alloy. The raw material is smelted in an electric resistance furnace at an atomic ratio of Mg-2Zn-0.7Ce (at. %) and then smelted in an electric resistance furnace at a temperature of 650°C. After the molten metal is cooled to 650°C, it is allowed to stand for 30



minutes and then poured into preheating. In the 200°C cylindrical metal mold, the cavity size is $\Phi 50\text{mm} \times 200\text{mm}$. It was then left to stand in the air and the ingot was allowed to cool to room temperature. Prior to hot extrusion, the Mg-2Zn-0.7Ce ingot was homogenized at 400°C for 12 h, the extrusion temperature was 300°C, the extrusion ratio was 16:1, and the bar with a diameter of 25 mm was extruded. The speed is 1-1.2 mm/s. After extruding the molding material, the bar is air-cooled to room temperature. The hot extruded Mg-2Zn-0.7Ce alloy sample was directly immersed in a metal tank filled with liquid nitrogen for cryogenic treatment. The cryogenic temperature was -196°C and the holding time was 1 min, 10 min, 1 h, 2 h, and 24 h, respectively. After the insulation is completed, the specimen is quickly removed using a jig, and the surface of the specimen is washed with alcohol and allowed to stand in the air to return to room temperature. The microstructure of the alloy before and after cryogenic treatment was observed by metallographic microscope and scanning electron microscope, and the phase composition of the crystal was identified by X-ray diffraction.

3. Test results and analysis

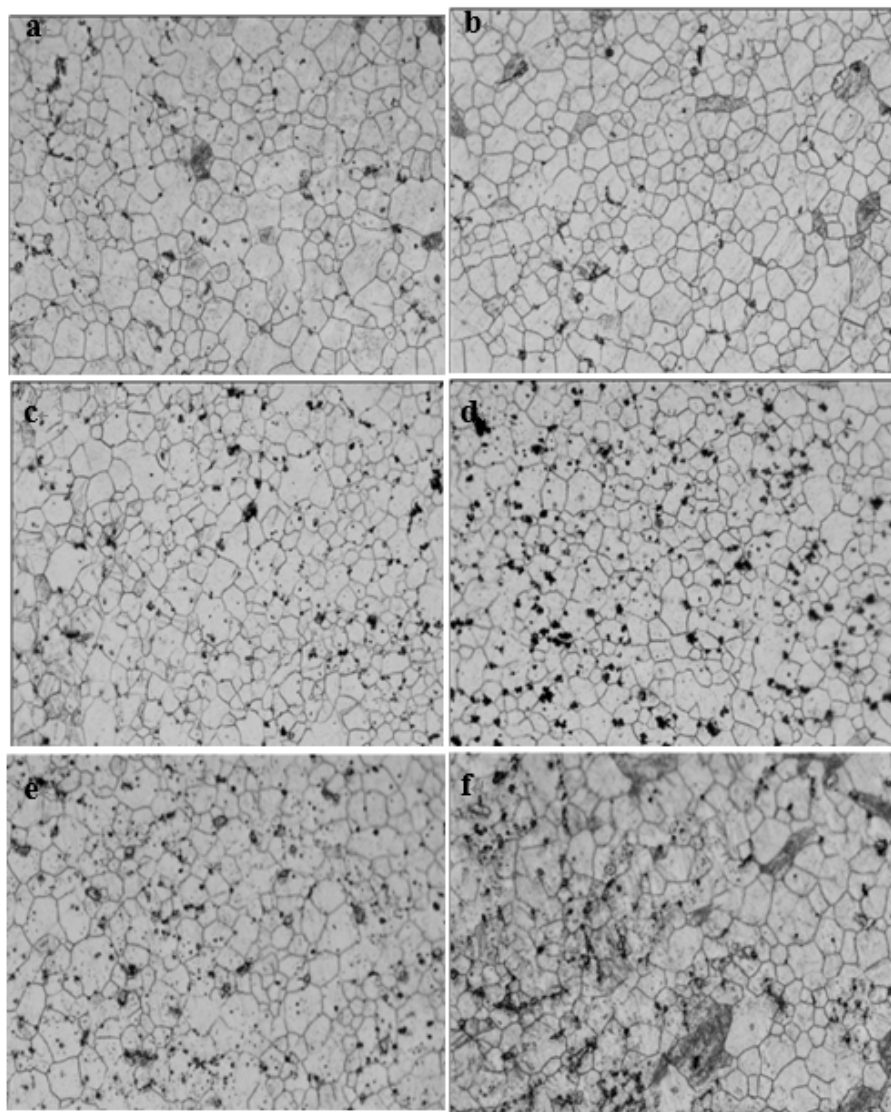


Fig 1. The optical microstructure of Mg-2Zn-0.7Ce alloy with different duration of CT: (a) as-extruded, (b) 1min, (c) 10min, (d) 1h, (e) 2h and (f) 24h

Figure 1(a) is a photomicrograph of the microstructure of the original extruded Mg-2Zn-0.7Ce alloy. The alloy consists of Mg grains and second phase grains. The average grain size in the as-extruded state is 21 μm . The second phase particles have a volume fraction of 0.2%. For Mg-Zn-Re-based alloys, two types of ternary equilibrium phases are generally formed, one is the I phase and the quasicrystal structure, and the other is the W phase and the cubic structure. The type of ternary equilibrium phase mainly depends on the Zn/Re ratio (atomic ratio) in the alloy composition. In the Mg-Zn-Ce system, the W phase appears in the alloy when the atomic ratio of Zn/Ce is 0.25-10. When the atomic ratio of Zn/Ce is 1.5-40, I phase appears in the alloy. In addition, when the atomic percentage of Zn in the alloy is higher than 3%, the Zn/Ce atomic ratio is in the range of 10-25 to form I phase. For the Mg-2Zn-0.7Ce (at. %) alloy, the atomic ratio of Zn/Ce is 15, and Zn accounts for less than 3% of the alloy atomic percentage. Therefore, these second phases may be phase I, phase W, or both. In previous studies, Phase I has a high thermal stability and can be tightly pinned on the Mg matrix, an effective reinforcing phase. However, the W phase and the Mg matrix do not bond tightly, and their enhancement is limited. Figures 1(b)-(f) are photomicrographs of the alloys after cryogenic treatment at different times. After cryogenic treatment for 1 min, the microstructure of the alloy did not change significantly. When the cryogenic treatment time increased to 10 min, a small and diffuse second phase was observed, mainly distributed on the grain boundaries, and a few evenly distributed in the grains. At this point, the volume fraction of the second phase particles surged to 2.3%. As the cryogenic time increases, the second phase particles continue to precipitate. When the cryogenic time increased to 1h and 2h, the volume fraction of the second phase reached 3.5% and 3.8%, respectively, showing little difference. When the cryogenic time was increased to 24 h, a large number of second phases had appeared inside some of the grains, and the second phase volume fraction was 4.8% (Fig. 1(f)).

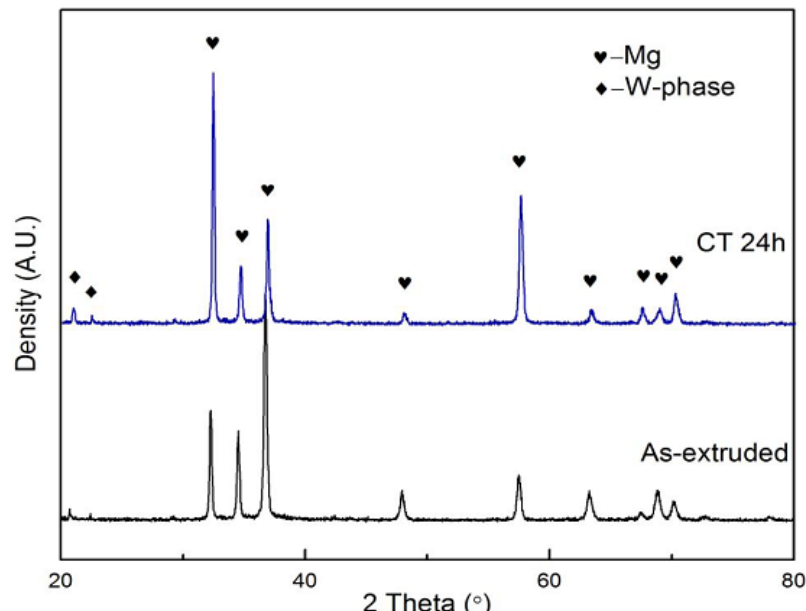


Fig. 2. The XRD curves of Mg-2Zn-0.7Ce alloy before and after CT

Figure 2 shows the XRD curves of Mg-2Zn-0.7Ce alloys after 24 hours in the as-extruded and cryogenic conditions. After the diffraction peak of the XRD curve was calibrated, it was found that the as-extruded alloy consisted only of Mg and W phases, but the characteristic peak of phase I could not be found. Therefore, it can be determined that the second phase appearing in the metallographic structure is mainly W phase. In addition, the diffraction peaks and peaks of Mg changed after cryogenic treatment for 24 h, in which the diffraction peaks of the (101_0) and (112_0) crystal planes

were enhanced, while the (0002) basal plane, the diffraction peaks of (101₁) and (101₂) were weakened. The change of Mg diffraction peak indicates that the grain rotation has occurred after cryogenic treatment. Other researchers have found similar phenomena in the cryogenic treatment of aluminum alloys and copper alloys. This shows that in the cryogenic treatment of non-ferrous alloys, the diffraction peak of crystal planes the change in intensity is a common phenomenon, which may be due to shrinkage of the volume of the crystal in the chilled environment of the material. According to the calculation of the linear expansion coefficient of polycrystalline Mg, when the polycrystalline Mg drops from room temperature (20°C) to cryogenic temperature (-196°C), its volume shrinks approximately 1.75%. The shrinkage of the crystals causes internal stresses in the material to initiate the rotation of the grains, causing their texture to change.

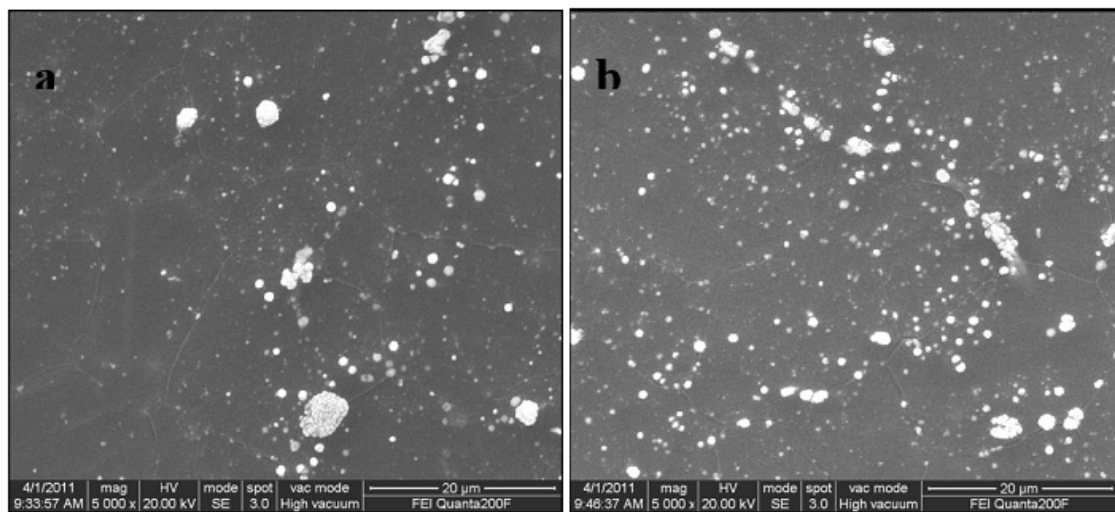


Fig.3. The SEM images of Mg-2Zn-0.7Ce alloy before (a) and after CT24 (b)

The size and morphology of the precipitated W phase after cryogenic treatment were further observed by SEM. Figure 3 shows the SEM images of the alloys before and after cryogenic cooling. From Figure 3(a), it can be seen that the as-extruded W-phase is spherical particles, mainly distributed along the grain boundaries, and the particle diameters are mainly distributed between 1 μm and 2.5 μm . Its average diameter is 2.1 μm . After 24 hours of cryogenic treatment, a large amount of fine W-phase particles precipitated within the grains (Fig.3(b)), so that the size distribution of the W-phase particles changed. The grain diameters at the grain boundaries are mainly distributed between 0.5 μm and 1.5 μm , and the average diameter is 1.4 μm , which is a reduction of 33%. This shows that cryogenic treatment can not only promote the precipitation of W-phase particles, but also refine the W-phase particles, thereby refining the alloy structure and improving the mechanical properties of the alloy.

4. Conclusion

The as-extruded Mg-2Zn-0.7Ce alloy consists of Mg matrix and W phase. After cryogenic treatment for 10 min, a large amount of nano-sized W-phase particles precipitated inside the alloy. As the cryogenic treatment time increases, the W phase continues to precipitate, but the precipitation rate gradually slows down. Cryogenic treatment refines W-phase particles and improves their distribution. This refines the microstructure of the extruded Mg-2Zn-0.7Ce alloy. The phenomenon of grain rotation occurred in the as-extruded Mg-2Zn-0.7Ce alloy after cryogenic treatment.

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