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Electron back-scattering diffraction preliminary analysis of heterogeneous nuclei in magnesium alloy during solidification process under GPa high pressure☆

Zhibin Fan [a], Xiaoping Lin [a, *], Rui Xu [b], Yun Dong [a], Bin Wen [b], Lin Wang [a], Shengshi Zhao [a]

[a] School of Materials and Metallurgy, Northeast University, Shenyang 110819, China
[b] Key Laboratory of Metastable Materials Science and Technology, College of Materials Science and Engineering, Yanshan University, Qinhuangdao 066004, China

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ABSTRACT

The effective heterogeneous nuclei in Mg-7.92 Zn-1.70 Y alloy during solidification process under high pressure was investigated by EBSD. It is found that the orientation relationship between Mg matrix and MgY phase, $[111]_{MgY}//[1210]_{\alpha\text{-}Mg}$ and $(101)_{MgY}//(0001)_{\alpha\text{-}Mg}$, exists between Mg matrix and MgY phase. The MgY phase is the potential heterogeneous nuclei substrate for α-Mg solidified under high pressure. It is calculated by Bramfitt mode that the mismatch between Mg matrix and MgY phase is greater than 12% under atmospheric pressure. When solidified under high pressure of 4 GPa, the solidification pressure can increase the number of the α-Mg nucleus by reducing the size of the nucleus, decreasing interfacial energy of MgY/α-Mg, and changing the wettability between MgY phase and α-Mg from non-wetting under atmospheric pressure to wetting under high pressure. The MgY phase becomes a powerful heterogeneous nuclei for α-Mg crystal. Increasing the number of valid heterogeneous nuclei substrate increases nucleus number and refines the grain of the alloy.

1. Introduction

The strengthening of casting magnesium alloys is an important issue in the study of magnesium alloys. The constant k of Mg alloy in Hall–Petch formula is a big value ($k_{Mg} \approx 280–320$ MPa $\mu m^{1/2}$), compared with 4–5 times for aluminium alloy ($k_{Al} \approx 68$ MPa $\mu m^{1/2}$). Strengthening effect of grain refinement is significant.[1] Therefore, grain refinement has become a hotspot and the most effective way to improve properties of casting magnesium alloys.[2–4]

Controlling the metal solidification process is a most important method to refine the solidification microstructure. Adjusting the chemical composition and temperature is the traditional way to control the metal solidification process. However, another thermodynamic parameters, pressure, is always ignored. Pressure, temperature, and chemical composition, are all the important thermodynamic variables that have influence on the solidification process of metals or alloys. Especially when pressure is in the range of a few GPa, the thermodynamics and kinetics of the solidification process can be significantly different, as compared to solidification at ambient pressures.[5–8] According to the current high-pressure solidification theory, increasing the pressure reduces the activation energy for nucleation, thereby increasing the rate of nucleation, while at the same time, it also raises the activation energy for atomic diffusion, and thus restrains atomic diffusion. As a result, this increases the activation energy for crystal growth and reduces the crystal growth rate.[9–12] Li et al.[13] studied the effect of pressure on the formation of the microstructure of ZA27 alloy and have found that the dendritic morphology changed from coarse dendrites to cellular crystallites as the solidification pressure increased. Han et al.[14] researched the solidification microstructure of Al-5.4% Cu alloy, which was solidified under high pressure, and found that the dendrite size was reduced evidently. Zhao et al.[15] investigated the effect of high pressure on the solidification microstructure, and the corrosion resistance of Mg–30%Al alloy, and showed that high pressure solidification produced significantly refined magnesium alloy structures. Wang[16] analyzed the impact of high pressure on the solidification microstructure, and the mechanical property of

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* Corresponding author.
E-mail address: lxping3588@163.com (X.P. Lin).

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Mg–10Zn–21Y alloy, and showed that the secondary dendrite arm spacing decreased with increase of pressure during solidification, and the tensile strength increased from 80 to 139 MPa under 3 GPa.

Dong et al.[17,18] investigated the solidification microstructure of Mg–Zn–Y alloy solidified under GPa grade high pressure, and found that the number of nucleus per unit volume was about 40 times that of the conventional casting alloy. The relationship between the number of nucleus per unit volume (Z) and two dynamics parameters (nucleation rate (N), growth velocity (G)) is $Z = 0.9(N/G)^{3/4}$. The greater the nucleation rate is, the more the number of nucleus per unit volume and the finer the grain size is. In general, the nucleation of liquid metal is heterogeneous nucleation. The critical undercooling limit of heterogeneous nucleation is correlated with the wetting angle (θ). The smaller wetting angle is, the smaller the critical nucleus radius and the smaller the critical undercooling limit is. The maximum of nucleation rate increases with the increase of the valid heterogeneous nucleation substrate area. However, the effect of solidification pressure on the valid heterogeneous nucleation and wetting angle in magnesium alloy during the solidification process has not been described. Therefore, in the present work, the heterogeneous nucleation and the effect of solidification pressure on wetting angle in magnesium alloy during the high pressure solidification process were analyzed.

2. Experimental methods

The experimental Mg–Zn–Y alloy was prepared by pure fusing together a magnesium ingot (99.9 wt % purity), a pure zinc ingot (99.9 wt% purity) and an ingot of the Mg–30Y master alloy in a 2RRL-M8 vacuum resistance furnace at the temperature of 740 °C. The molten alloy was then poured into a preheated metal mold, to produce the alloy ingots. Plasma spectroscopy was done using an ICAP6300 ICP spectrometer, to analyze the chemical compositions of the alloy ingot. The results show a composition containing, in atom fractions, 7.92% Zn, 1.70% Y, with the rest being Mg. After the full homogenizing annealing process, the as-cast alloy was cut into samples with a diameter of 6 mm and a length of 8 mm for the experiments of high-pressure solidification. A CS-1B high-pressure cubic press was used to carry out the high-pressure solidification experiments. The solidification pressure used was 4 GPa. The heating temperatures were set to 700, 860, 900, 960, 1050 and 1100 °C, separately. As per the Clausius–Clapeyron equation,[19] the melting point of magnesium alloys increases with the increase of the pressure. Measurements show that the started melting temperature of the experimental alloy under 4 GPa is nearly 920 °C. Each sample was inserted into the assembly sleeve, and the assembled graphite sleeve was placed in the cavity position of the high-pressure cubic press. After the pressure acting on the alloy inside the cubic press increased to the preset pressure, the temperature-measuring device was turned on.[20] The samples were heated rapidly to the heating temperature and held under the preset pressure for 20 min until they were solidified and cooled. When the samples naturally cooled down to room temperature, the pressure was released and the samples were removed from the press for further testing and analysis.

The microstructure and the component were observed and analyzed using a LEO JSM 5400 scanning electron microscope (SEM) with energy disperse spectroscopy (EDS). Phase identification was performed using X-ray diffraction (XRD). The volume fraction of the second phase was calculated by using the Image-Pro-Plus (IPP) software. For preparing the samples for the EBSD analysis, the surface of the samples sectioned parallel to the compression axis was mechanically ground with SiC papers (800–5000#) and then electro-polished (electrolyte: 30% nitric acid alcohol, operating voltage: 1.5 V). The EBSD data were processed using the HKL Channel 5 analysis software.

3. Results and discussion

3.1. Microstructure changes of the Mg-7.92Zn-1.70Y alloy during heating process under high pressure

Fig. 1 shows the microstructure changes of the Mg alloy during heating process under high pressure. As shown in Fig. 1(a), the microstructure of the Mg alloy solidified under the atmospheric pressure showed that the α-Mg matrix consisted of equiaxed crystal. There were many secondary phases with a continuous net-like lamellar eutectic structure distributed along the interdendritic regions. The volume fraction of the second phases was 23%, and the highest solubility of Zn in matrix was 2.85 wt.%. The XRD results suggested that the microstructure of Mg alloy solidified under the

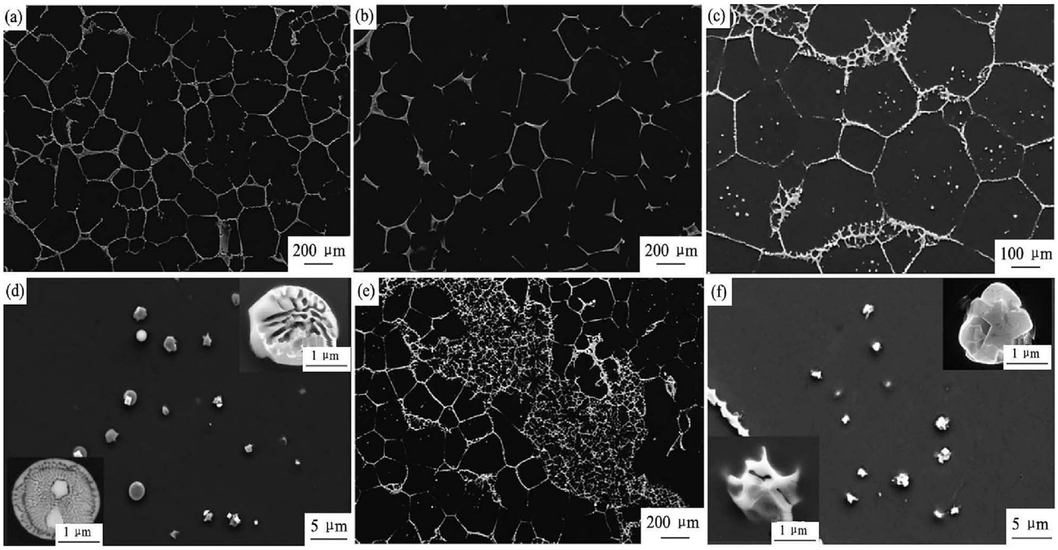

Fig. 1. Microstructures of alloy under different conditions, (a) atmospheric pressure; (b) 4 GPa-700 °C; (c,d) 4 GPa-860 °C (e,f) 4 GPa-900 °C.