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The formation of Al_6 (Fe, Mn) phase in die-cast Al-Mg alloys

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Abstract. In aluminium alloys, iron is a common impurity as it is unavoidably picked up in practice. The excessive Fe is strongly prone to form various intermetallic phases. These Fe-rich intermetallics are generally brittle and act as stress raisers to weaken the coherence with Al matrix, therefore decreasing elongation. However, Fe addition in Al-Mg alloys may be beneficial because of the improvement in the yield strength with the scarification of ductility of die-cast aluminium alloys. The morphology of intermetallic phases has a vital effect on the properties of aluminium alloys. In the present work, the 3D morphology of Al_6 (Fe, Mn) in die-cast Al-Mg-Mn alloys with different levels of Fe contents were revealed. The formation of Al_6 (Fe, Mn) was also studied through crystal features and solidification behaviours.

1. Introduction

In die-cast aluminium alloys, Fe is a common deleterious impurity for elongation and is not economically removed from the melt [1-3]. Therefore the accumulation of iron has been a critical concern in recycled materials materials. Up to now, several methods have been invented to diminish the detrimental effect of Fe, such as melt-superheating treatment, chemical modification and rapid solidification [4-7]. Among them, Mn modification is a popular method to transform needle-like β-AlFeSi to blocky α-AlFeMnSi in Al-Si alloys. While, α-AlFeMnSi phase would forms large primary crystal and resulting in unacceptable mechanical properties when the total amount of Mn and Fe is more than 1.5 wt.%. Therefore, the application of recycled Al alloy with high Fe content is still an important concern in industry.

The obstacle of the application of recycled high-Fe Al alloy is that Fe-rich intermetallics would decrease elongation obviously. Therefore, a soft alloy matrix (such as ductile Al-Mg matrix) is necessary for high-Fe alloy. On the other side, it was found that Fe-rich phases in aluminium alloys are helpful in improving the yield strength with the scarification of ductility [8]. Besides, the addition of Mn in Al-Mg alloys can modify Al_3Fe to Al_6(Fe, Mn), and the increased cooling rate can significantly refine the Al_6(Fe, Mn) phase. It further increases the upper limit of the Fe level in aluminium alloys. Therefore, it is potential to use Fe as a strengthening element to design an Al-Mg-Mn-Fe alloy prepared by high pressure die casting (HPDC) which has a high cooling rate among various common casting methods.

The mechanical properties (especially the fracture behavior) of alloys are closed with the morphology of intermetallic phases. However, the Al_6(Fe, Mn) morphology and growth mechanism in Al-Mg alloys are still unclear. Therefore, the present work aims to reveal the 3D morphology and relative growth mechanism of Al_6(Fe, Mn) phase in Al-Mg-Mn-Fe alloys.

2. Experimental methods

Commercial pure Al and Mg ingots, Al-20% Mn and Al-45% Fe master alloys (all compositions quoted in this paper are in wt.% unless otherwise stated) were melted in an electric resistance furnace at 730 °C to prepare a series of Al-5Mg-xFe-0.6Mn alloys (x = 0.5, 1.5 and 2.5). After a homogenization process for about 30 min, the melt was manually dosed and subsequently released into the shot sleeve of a FRECH 4500 kN cold chamber HPDC machine. The HPDC die and shot sleeve were preheated at about 150 °C and 100 °C, respectively. The pouring temperature was 720 °C. Al_6(Fe, Mn) particles were collected after deep-etching or completely removing the matrix of samples by 15 vol.% HCl–distilled water solution, and then observed by a scanning electron microscope (SEM, Zeiss-Supra 35VP, Germany) equipped with an energy-dispersive X-ray spectroscope (EDS) and electron back-scattered diffractor (EBSD).

3. Results and discussion

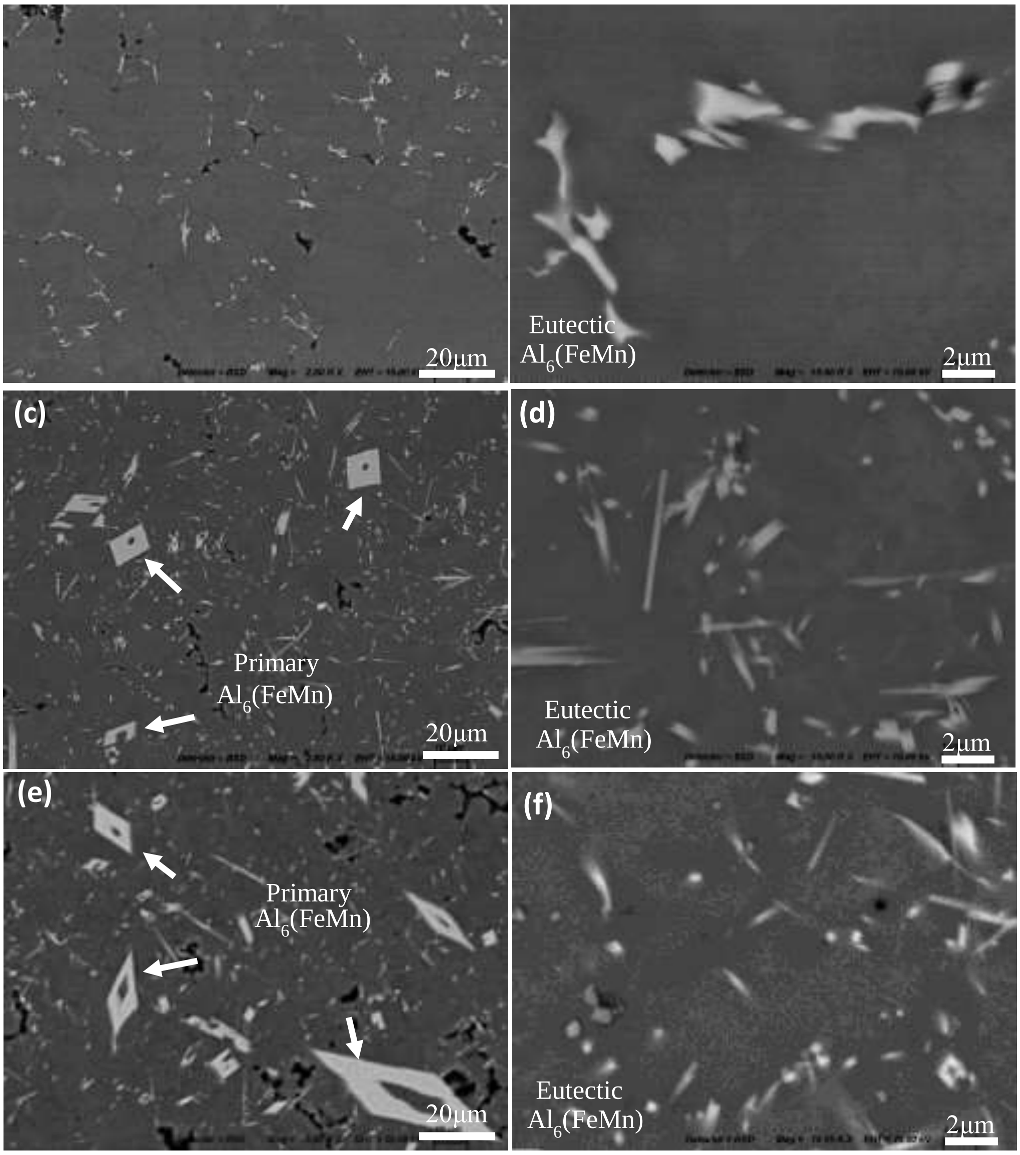

Figure 1. Backscattered SEM micrographs showing the microstructures of the Al-5Mg-0.6Mn-xFe alloys: (a, b) x=0.5, (c, d) x=1.5 and (e, f) x=2.5.

The as-cast microstructures of Al-5Mg-xFe-0.6Mn alloys (x = 0.5, 1.5 and 2.5) are shown in Figure 1. As observed, the morphology, size and amount of bright Al_6(Fe, Mn) phase varies obviously with the increase of the Fe level. In the alloy with 0.5 wt.% Fe, eutectic Al_6(Fe, Mn) were located at the grain boundaries and showed a lamellar morphology with some curved planes, as shown in Figure 1a and b. When the Fe level was at 1.5 wt.%, the rhombic or lath-like primary Al_6(Fe, Mn) phase was found (Figure 1c). Meanwhile, the eutectic Al_6(Fe, Mn) intermetallics were divorced from α-Al phase and exhibited as fine lath-like or rhombic morphologies with faceted surface (Figure 1d). When the Fe level was further increased to 2.5 wt%, both the amount and size of primary and eutectic Al_6(Fe, Mn) intermetallics were increased, while the morphology remains. It was noticed that the primary Al_6(Fe, Mn) intermetallics in the alloys with 2.5 wt.% Fe have two different size ranges. Similar with our previous result [3], the large and small primary Fe-rich intermetallics precipitated in the first solidification in shot sleeve and the secondary solidification in die cavity during cold-chamber HPDC process, respectively.

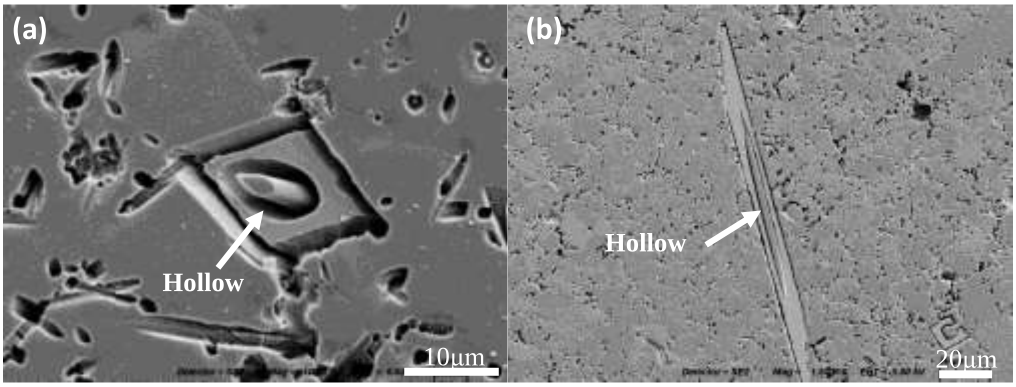

Figure 2. SEM micrographs showing (a) cross and (b) longitudinal sections of Al_6(Fe, Mn) phase.

It is interesting to note that both primary and eutectic phases have two morphologies: (1) lath / needle, (2) rhombus/dot. Besides, it was also found that almost all rhombic primary Al_6(Fe, Mn) crystals have a hollow. To further observe the morphology of the hollow primary Al_6(Fe,Mn) clearly, samples were deep-etched using a 15 vol% HCl-distilled water solution. Figure 2 indicates that the hollow is filled by α-Al phase and has a curved, smooth surface, although Al_6(Fe, Mn) is a strong faceted crystal. It means that these surface is not a certain exposed crystal surface, but the defects due to the incomplete crystal growth.

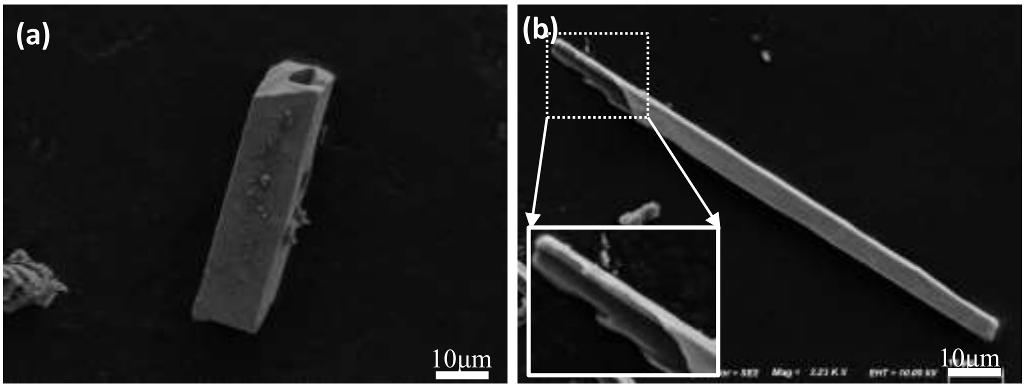

Figure 3. SEM micrographs showing 3D morphology of primary Al_6(Fe, Mn) phase: (a) a thick crystal and (b) a thin crystal with broken end.

To show the 3D morphologies of Al_6(Fe, Mn) crystals, a 15 vol% HCl water solution was applied to extract the intermetallic particles. The results shown in Figure 3 and 4 indicate that all primary and eutectic Al_6(Fe, Mn) crystals have one 3D morphology: quadrangular prism, and the lath/needle and rhombus/dot morphology in 2D sections are just the longitudinal and cross sections of Al_6(Fe, Mn) crystals, respectively. The difference is that the primary Al_6(Fe, Mn) crystals (shown in Figure 3) are coarse and hollow, while the eutectic Al_6(Fe, Mn) crystals (shown in Figure 4) is thin (200-500 nm in cross section) and solid.

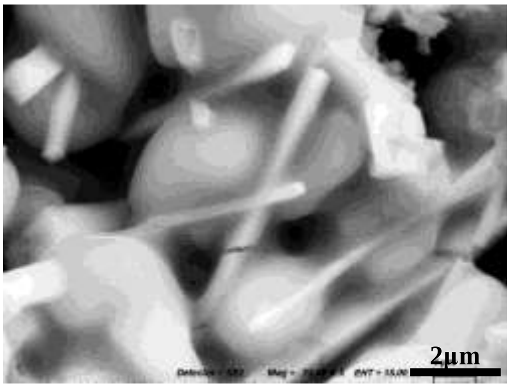

Figure 4. SEM micrographs showing 3D morphology of eutectic Al_6(Fe, Mn) phase.

The Bravais-Friedel-Donnay-Harker (BFDH) law is a well-accepted method to predict the possible exposed crystal faces. Al_6(Fe, Mn) has a Cmcm (63) space group and an orthorhombic structure (a = 0.7498 nm, b = 0.6495 nm, c = 0.8837 nm). According to the BFDH law, the 2 most possible exposed face are closed packed {100} and {002} faces. For orthorhombic crystal, the {110} only contains 4 (-110), (1-10) and (-1-10) faces, which form four side faces of an enclosed geometrical rhombic prism. While, {002} contains (112) and (00-2) faces, forming the top and bottom face of rhombic prism. Therefore, it can be speculated that the rhombic Al_6(Fe, Mn) is bounded by four {110} and two {002} faces.

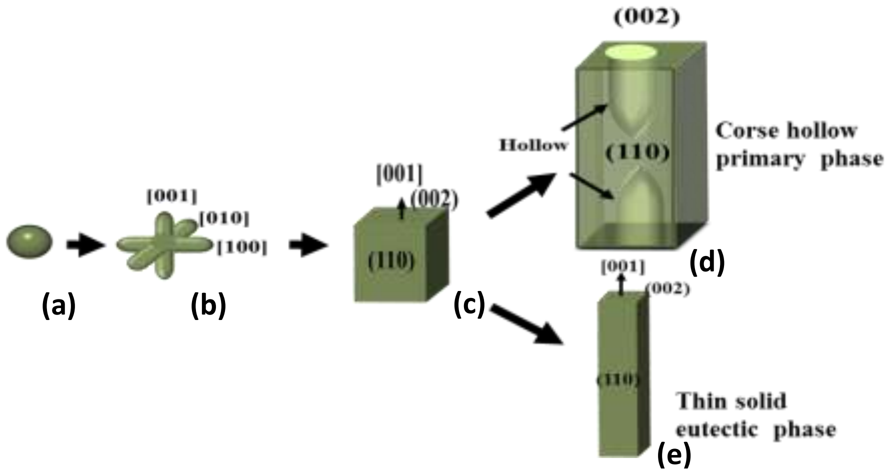

Figure 5. Schematic of the growth process of prism Al_6(Fe, Mn) crystal bounded by {110} and {002}: (a) spherical seed, (b) dentrite, (c) small solid rhombic prism, (d, e) coarse hollow primary phase and thin solid eutectic prism.

Figure 5 shows the growth process of prism Al_6(Fe, Mn) crystal in Al-Mg-Mn-Fe melt. As the decrease of melt temperature, the micro-segregation of atomic cluster becomes obvious and forms a spherical morphology with minimum free energy [10]. When the seed crystal grows and exceeds a critical size, it becomes unstable and grows rapidly along its first preferential growth directions ([001], [100] and [010]). At almost same time, new secondary branches generate on the first branches and grow between the first branches and finally fill these interspaces, making Al_6(Fe, Mn) crystal have a small prism morphology ({110}+{002}) with a strong intrinsic faceting feature. In the following growth stage, the rapid growth of {002}, which has a higher growth rate than {110} according to BFDH law, would elongate Al_6(Fe, Mn) prism, making it have a lath-like (primary phase) or needle-like (eutectic phase) morphology. The hollows inside Al_6(Fe, Mn) crystal also formed in the following process due to volume-diffusion. Compared with the diffusion in the side {110} faces, the diffusion in the central area of the faster growing {002} faces is relatively difficult. Therefore, the supplement of solute atoms and the ejection of impurities expelled from {002} faces are relatively slow. It will retard the growth in the central areas of the {002} faces, leading to the formation of hollows inside the primary Al_6(Fe, Mn) prism. While, the volume diffusion in small eutectic Al_6(Fe, Mn) crystal is not obvious, so eutectic Al_6(Fe,Mn) crystals remain solid.

4. Conclusions

In the present work, the 3D morphology and formation mechanism of Al_6(Fe, Mn) phase in HPDC Al-Mg-Mn-Fe alloys were revealed. It was found that Al_6(Fe, Mn) is the only eutectic Fe-rich phase and has an irregular morphology. As the Fe level increased to 1.5 wt.% and 2.5 wt.%, both primary and eutectic Al_6(Fe, Mn) have a rhombic prism morphology which is bonded by four {110} and two {002} faces. While, primary Al_6(Fe, Mn) phase has inside hollows, the eutectic phase is small and solid. In the late stage of crystal growth, volume-diffusion restrains the growth of the central areas of two {002} faces, leading to the formation of hollows inside the primary Al_6(Fe, Mn) phase. The small eutectic Al_6(Fe, Mn) phase is not affected by volume diffusion and remains solid.

5. References

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