

Microstructural evolution of Al–0.66Mg–0.85Si alloy during homogenization

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Abstract: The microstructural evolution of Al–0.66Mg–0.85Si alloy was investigated by optical microscopy (OM), scanning electron microscopy (SEM), transmission electron microscopy (TEM) and differential scanning calorimetry (DSC). The as-cast microstructure is typical dendrital structure, consisting of α (Al), Al(FeMn)Si, Mg_2Si , AlCuMgSi and Si phases. The electron diffraction analyses indicate that the Al(FeMn)Si phase is $Al_{15}(FeMn)_3Si_2$ and the AlCuMgSi phase is $Q(Al_{1.9}CuMg_{4.1}Si_{3.3})$. There are two kinds of Mg_2Si phases in the as-cast microstructure. One is formed in the casting process, and the other is formed in the cooling process after casting process is finished. The phases have different crystal structures. After homogenization treatment at 545 °C for 20 h, Mg_2Si , Si and Q intermetallic compounds are dissolved into matrix completely, and the remaining phases are α (Al) and $Al_{15}(FeMn)_3Si_2$. The size of $Al_{15}(FeMn)_3Si_2$ phase is decreased, and the phase is spheroidized and distributes along grain boundary discontinuously. The Zn-containing phases are not found during solidification and homogenization process.

Key words: Al–Mg–Si alloy; microstructural evolution; homogenization; intermetallic compound

1 Introduction

In the automotive industry, aluminum alloy panels have increasingly substituted for steel panels as automotive industry standards are becoming increasingly strict with respect to fuel efficiency, air pollution, recycling and safety [1–3]. Progress toward saving energy and achieving harmony with environment has been gaining momentum [4–6]. The European Commission had announced a policy to reduce CO₂ emissions, in order to encourage losing weight of automobiles [7]. Weight reduction of automobiles has received attention as a promising method to attain these goals. While the use of aluminum alloys meets not only the needs, but also the desirable characteristics of such products, namely their light weight, corrosion resistance, recycling and high strength. 6000 series aluminum alloys have a combination of medium strength, good corrosion resistance, excellent formability, good weldability and easy recycling, especially good heat treatability. The alloys exhibit bake hardening response during the baking step after painting in the automotive manufacturing process [8,9]. Consequently, 6000 series aluminum

alloys constitute the mainstream of alloys used for automotive panels.

The basic elements of the alloy are Mg and Si, which combine to form Mg_2Si precipitate. The alloy with excessive amount of Si exhibits better mechanical properties. Manganese, zirconium and copper are usually added to alloys to improve the properties. WU et al [10] investigated the microstructural evolution of Al–Mg–Si–Mn–Cu–Ce alloy during homogenization by OM, SEM and EDS. The results indicated that the phase constituents in as-cast microstructure were Al(FeMn)₃Si₂, AlCuMgSi, AlCuSiCe and ternary eutectic α (Al)+AlCuMgSi+Si. After homogenization at 470 °C for 3 h, the low-melting point phase (AlCuMgSi) and ternary eutectic phase (α (Al)+AlCuMgSi+Si) were almost completely dissolved. The obvious dissolution of the Al(FeMn)₃Si₂ phase was started at 570 °C. BIROL [11] studied the effect of homogenization on the microstructure of 6063 billets by OM, DSC and electrical conductivity. The main result was that the transforming temperature of β -AlFeSi $\rightarrow\alpha$ -AlFeSi reaction was 580 °C, with low Mn content. CAI et al [12] simulated casting and homogenization of two 6xxx series alloys. The result indicated that Mg_2Si phase was dissolved into

matrix during homogenization. Ji et al [13] investigated the microstructural characteristics of 6022 alloy by SEM; the as-cast microstructure of the alloy was $\alpha(\text{Al})$, Al_2Cu , Al_5FeSi , $\alpha\text{-Al}(\text{MnCrFe})\text{Si}$, $\text{Al}_3\text{Cu}_2\text{Mg}_8\text{Si}_6$, Mg_2Si and Si . After homogenization treatment, most of the Al_2Cu , $\text{Al}_3\text{Cu}_2\text{Mg}_8\text{Si}_6$ and Mg_2Si phases were dissolved. The remaining phases were Si , $\alpha\text{-Al}(\text{MnCrFe})\text{Si}$ and Al_5FeSi . Most of $\beta\text{-Al}_5\text{FeSi}$ phases transformed into $\alpha\text{-Al}(\text{MnCrFe})\text{Si}$ particles [13]. The microstructure of the alloy depended on alloy composition, cooling rate and homogenization treatment, and so on. In the present work, mole ratio of Mg to Si is different from above studied alloy. Zn element is also added, and the content is 0.6%; composition of the alloy is different from Al–Mg–Si alloy studied previously. Meanwhile, Mg and Zn could form strengthening phase during artificial ageing. However, the content of Zn element is low in usual Al–Mg–Si alloy, even uncontained Zn element. Investigation of microstructural evolution of Al–Mg–Si alloy adding Zn element is very necessary. The Al–Mg–Si alloy with Zn element by TEM was studied qualitatively with the unique selected area electron diffraction (SAED) pattern of the phase. Simultaneously, the effect of Zn element on as-cast and as-homogenized microstructure of the alloy was analyzed.

2 Experimental

The alloy used in this study was prepared from high-purity aluminum, magnesium, copper, zinc, Al–10Mn, Mg–30Zr and Al–14Si master alloys. The raw materials were melted in a graphite crucible in an electrical resistance furnace and then cast in a cast iron mould at 710 °C. The chemical composition of the ingots is listed in Table 1.

Table 1 Chemical composition of alloy (mass fraction, %)

Mg	Si	Cu	Fe	Mn	Zn	Zr	Al
0.66	0.85	0.20	<0.01	0.10	0.60	0.12	Bal.

The microstructure of the alloy was studied by differential scanning calorimetry (DSC), optical microscopy (OM) and scanning electron microscopy (SEM). The EXSTAR6220 differential scanning calorimeter was used for DSC analysis, with Ar atmosphere, at a heating rate of 10 °C/min. The as-cast samples were etched by Keller's reagent; as-homogenized samples were electro-polished and anode coated. Intermetallic compounds were investigated by JEM–2000FX transmission electron microscope (TEM) operated at 160 kV. Specimens for TEM were cut from bulk specimens followed by mechanical

wet-grinding to about 50 μm , and then dimpling to $d3\ \mu\text{m}$, and finally twin-jet electro-polished in a solution of 25% nitric acid–methanol at temperatures ranging from $-30\ ^\circ\text{C}$ to $-20\ ^\circ\text{C}$.

3 Results

3.1 As-cast microstructure of alloy

The as-cast microstructure of alloy is typical dendritical structure (Fig. 1(a)). The average grain size is 130 μm . The non-equilibrium constituents are distributed along grain boundary, and the phases nucleate near the grain boundary, owing to high energy of grain boundary. These phases are formed from non-equilibrium reactions during solidification process. While the spherical phases distribute within grains (Fig. 1(b)); these are equilibrium phases, which are produced from equilibrium reactions during solidification. Figure 2 shows the SEM image of the as-cast alloy; black lathy phases and white rod-shaped precipitates are distributed along grain boundary. The spherical phases are distributed within grains. The composition of the phases is summarized in Table 2. The premier phase precipitated from liquid is $\alpha(\text{Al})$, and it formed the matrix of material [14–16].

The phases were confirmed by TEM analysis. $\text{Al}_{15}(\text{FeMn})_3\text{Si}_2$, Mg_2Si and Si precipitates mostly distribute along grain boundary. The cooling rate is very fast in the casting process, the energy of grain boundary

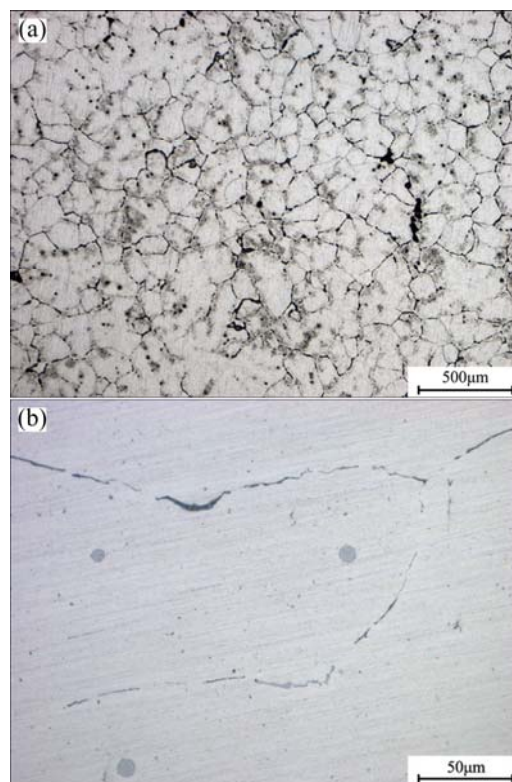


Fig. 1 Optical micrographs of as-cast alloy with (a) and without (b) etching

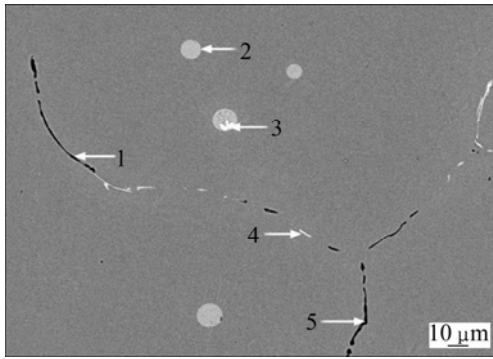


Fig. 2 SEM image of as-cast alloy

Table 2 Composition of second phase marked in Fig. 2 measured by EDS

Area No.	$x(\text{Al})/\%$	$x(\text{Si})/\%$	$x(\text{Mg})/\%$	$x(\text{Cu})/\%$	$x(\text{Mn})/\%$	$x(\text{Fe})/\%$
1	89.59	10.41	—	—	—	—
2	57.36	16.58	20.51	5.55	—	—
3	69.23	12.05	2.85	3.69	5.76	6.42
4	65.91	21.36	02.26	—	5.71	4.76
5	33.29	21.91	44.80	—	—	—

is high, and grain boundary provides the nucleation site for these phases. So non-equilibrium phases form from non-equilibrium reactions. The precipitation temperature of $\text{Al}_{15}(\text{FeMn})_3\text{Si}_2$ phase during casting process is higher than that of Mg_2Si or Si phase. Consequently, Mg_2Si and Si could be dissolved into matrix during homogenization, while $\text{Al}_{15}(\text{FeMn})_3\text{Si}_2$ phase is hardly dissolved, which is detrimental to material performance. The circular shaped phase is AlCuMgSi phase, which is easily dissolved into matrix during homogenization process. Figure 3 shows TEM image and SAED pattern of AlCuMgSi precipitate. The phase is eutectic and the crystal structure is hexagonal. The SAED analysis indicates that the Q phase is $\text{Al}_{1.9}\text{CuMg}_{4.1}\text{Si}_{3.3}$. It was reported that the phase precipitated from matrix at 530 °C during solidification [17]. TEM image and SAED pattern of $\text{Al}_{15}(\text{FeMn})_3\text{Si}_2$ phase are given in Fig. 4. The crystal structure of the phase is cubic. The phase is rod-shaped, and can be dissolved rarely during homogenization process, which is harmful to performance of the alloy. The precipitation temperature from matrix was above 600 °C during solidification [17]. Therefore, in order to improve the material performance, the amount of $\text{Al}_{15}(\text{FeMn})_3\text{Si}_2$ phase should be controlled initially, especially the content of Fe element.

TEM images and SAED patterns of Mg_2Si phase are shown in Fig. 5. There are two kinds of Mg_2Si phases in as-cast alloy. One is formed in the casting process and distributed along grain boundary (Fig. 1(b) and

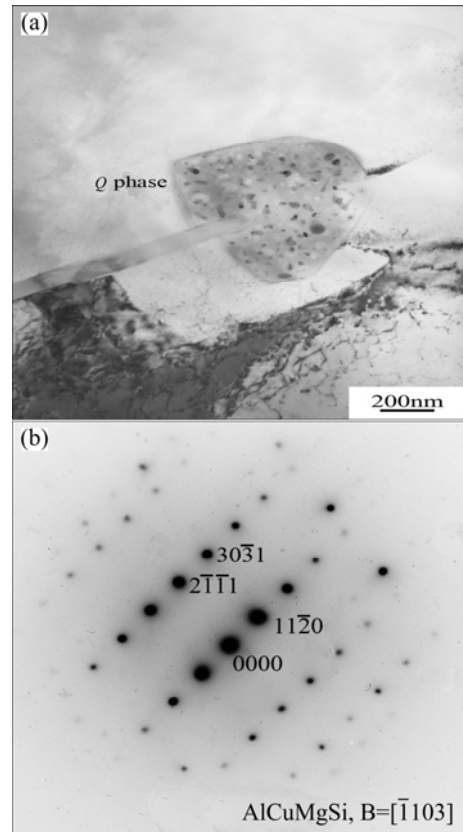


Fig. 3 TEM image (a) and SAED pattern (b) of AlCuMgSi phase in as-cast alloy

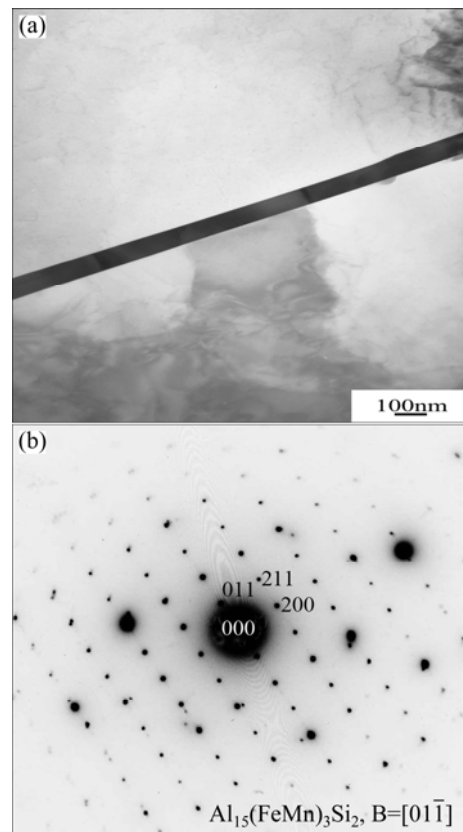


Fig. 4 TEM image (a) and SAED pattern (b) of $\text{Al}_{15}(\text{FeMn})_3\text{Si}_2$ phase in as-cast alloy

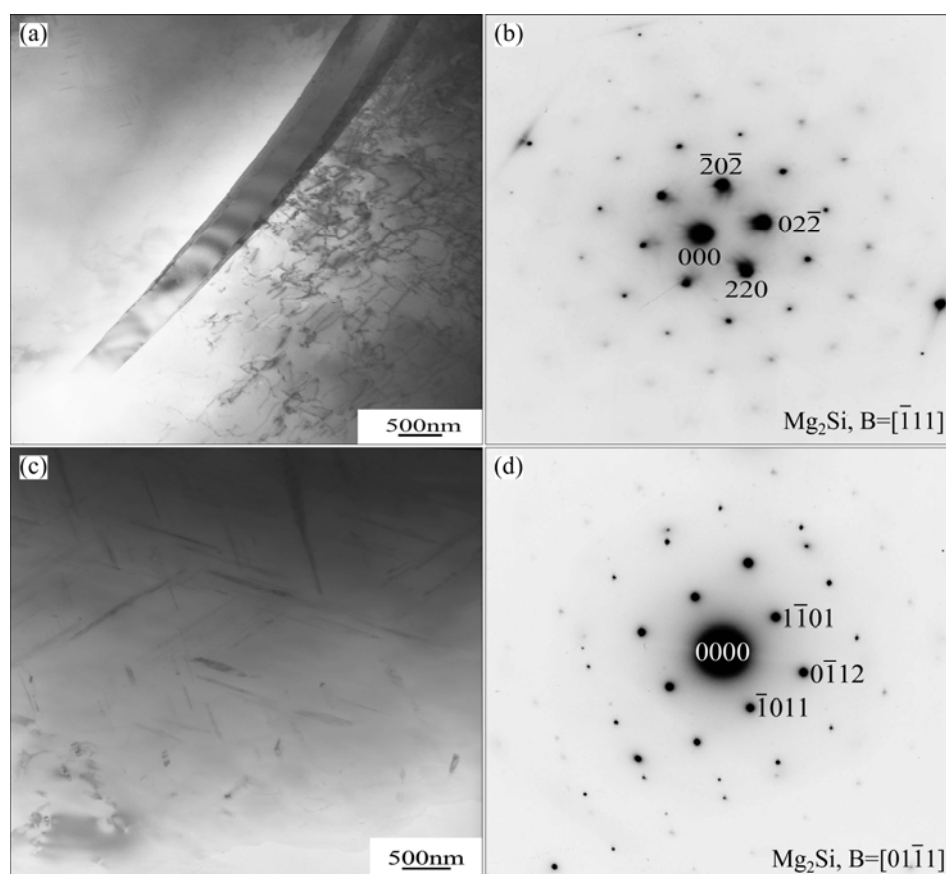


Fig. 5 TEM images and SAED patterns of Mg_2Si phase formed during solidification (a, b) and cooling process (c, d)

Fig. 5(a)), it is rod-like, and the size is large. The crystal structure of the phase is face-center cubic (Fig. 5(b)). The precipitation temperature from matrix was 580°C during solidification [14,15]. The other kind of Mg_2Si phase is shown in Figs. 5(c) and 5(d), which is formed in the cooling process after the casting process finished. As cooling rate is very fast during casting process, it is too late for phase to grow up. The size of the constituent is small (Fig. 5(c)), and it can also be seen in Fig. 1(b) and Fig. 2. The phase is hexagonal and needle-shaped. Zn-containing phases were not found during solidification process.

3.2 As-homogenized microstructure of alloy

In order to eliminate the constituent segregation and non-equilibrium phases formed during casting process, homogenization treatment is needed. DSC analysis is used to confirm the melting temperature of the secondary phases, which is useful to guide the homogenization treatment of the alloy. According to the DSC curve (see Fig. 6), homogenization treatment at 545°C for 20 h was chosen for the alloy. Figure 7(a) shows one phase remaining after homogenization; the phases are distributed along grain boundary and within grains, which are gray precipitates. Compared with as-cast alloy, the constituents were dissolved into matrix more

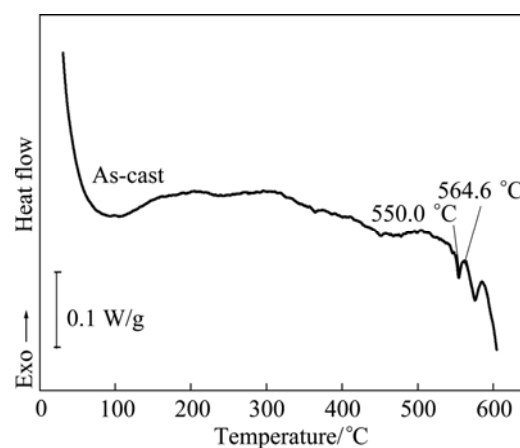


Fig. 6 DSC thermogram of as-cast alloy

sufficiently, while grey phases were dissolved hardly. The grains grow up obviously after homogenization treatment (Fig. 7(b)). The grain boundary is very thin, and becomes straight. The remaining intermetallic compound has two different shapes: circular-shaped phase distributed within grains, and small rod-shaped phase distributed along grain boundary (Fig. 8). The composition of the phase remaining after homogenization treatment is shown in Table 3. The results indicate that the remaining precipitate is Fe-containing phase.

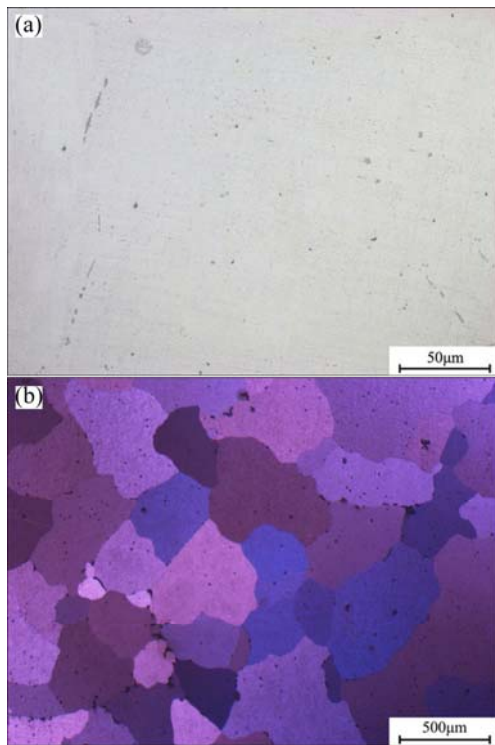


Fig. 7 Optical micrographs of alloy after homogenization: (a) Without etching; (b) Electro-polished and anode coated

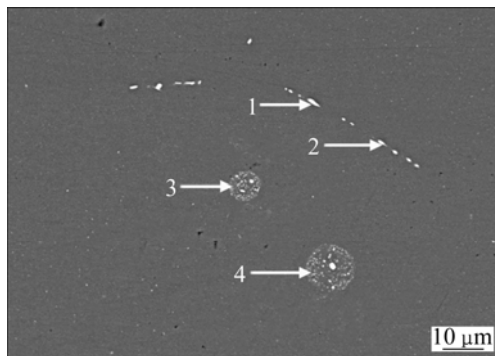


Fig. 8 SEM image of alloy after homogenization treatment

To confirm the phases after homogenization treatment, TEM analysis was used. The results indicate that after homogenization treatment at 545 °C for 20 h, AlCuMgSi, Mg₂Si and Si precipitates are dissolved into matrix completely. The electron diffraction analysis indicates that the remaining phase is Al₁₅(FeMn)₃Si₂ (Fig. 9), which is the same as that literature reported [10]. Studies had shown that low-melting point AlCuMgSi phase was dissolved after homogenization treatment at 470 °C for 3 h [10]. During homogenization treatment, AlCuMgSi phase un-containing Fe and Mn elements, dissolved completely. However, AlCuMgSi precipitate with Fe and Mn elements (Table 2); after homogenization treatment, the remaining phase was Al₁₅(FeMn)₃Si₂. The shape of phase is circular, as seen in Fig. 8. Studies had proved that Mg₂Si and Si phases

started to dissolve at 530 °C [18,19]. So, Mg₂Si and Si phases were dissolved into matrix after homogenization at 545 °C for 20 h. It was reported that the melting temperature of Al₁₅(FeMn)₃Si₂ was about 570 °C [10]. Temperature has more important influence than time during homogenization process. Consequently, it is difficult for Al₁₅(FeMn)₃Si₂ phase to dissolve at 545 °C, as seen in Fig. 9(a).

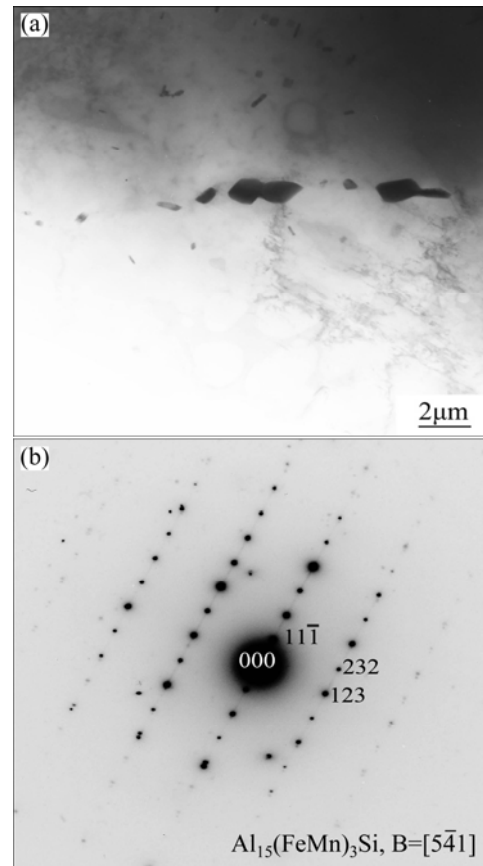


Fig. 9 TEM image and SAED pattern of Al₁₅(FeMn)₃Si₂ phase in as-homogenized alloy: (a) BF image; (b) SAED pattern

Table 3 Composition of intermetallic compounds marked in Fig.8 measured by EDS

Area No.	x(Al)/%	x(Si)/%	x(Mg)/%	x(Mn)/%	x(Fe)/%
1	82.07	6.24	0.87	6.69	4.13
2	80.16	6.20	1.14	7.88	4.62
3	93.32	2.32	1.02	1.96	1.38
4	89.22	3.08	1.03	3.93	2.74

In consequence, TEM image and SAED pattern indicated that the remaining phase was Al₁₅(FeMn)₃Si₂, and other constituents, such as Mg₂Si and Si were not found. After homogenization treatment, AlCuMgSi, Mg₂Si and Si phases were dissolved, and the remaining phases were α(Al) and Al₁₅(FeMn)₃Si₂. Zn-containing phases were not found during the homogenization process.

4 Discussion

The main alloying elements of Al–Mg–Si alloy are Mg and Si. Copper and manganese are usually added to the alloys to improve properties, sometimes titanium, zirconium and chromium are also added. Consequently, the as-cast microstructure of Al–Mg–Si alloy is complex; the possible phases are β -AlFeSi, α -AlFeSi, δ -AlFeSi, π (Al₈Si₆Mg₃Fe), AlCuMgSi, Mg₂Si, Si, θ (Al₂Cu), Al₆Mn and Al₆Fe, and so on [16,19–21]. The as-cast microstructure depends on composition of the alloy and the casting process. Fe has very low solubility in the Al. The Fe-containing phases exist in almost all of as-cast microstructure of Al–Mg–Si alloys, and the types of these phases depend mainly upon the cooling rate and the mole ratio of Fe to Si in the alloy. The size of Fe-containing phase is large and it is rarely dissolved during homogenization treatment, which is harmful to the performance of the alloy, such as surface quality, ductility and mechanical properties [22]. The Fe-containing phase should be possible to reduce and eliminate during casting and heat treatment process.

During casting process, the non-equilibrium solidification led to the non-uniform and non-equilibrium solidification effect (non-equilibrium eutectic phase, coarse precipitates, quenching effect). Therefore, the performance of material is non-uniform, and plasticity is low, deforming resistance is high and corrosion resistance becomes poor. The as-cast microstructure of the alloy was α (Al), Al₁₅(FeMn)₃Si₂, Mg₂Si, AlCuMgSi and Si, exhibiting dendrital structure. The Al₁₅(FeMn)₃Si₂, Mg₂Si, and Si were distributed along grain boundary, while AlCuMgSi precipitate was distributed within grains. The matrix of the alloy was α (Al). In order to make the alloy deform, homogenization treatment is needed. A large number of constituents are dissolved into matrix during homogenization process. The main solid transformation of homogenization process is atomic diffusion, with the secondary phases coarsening, dissolving and precipitating, and grain growing up, which directs microstructure to equilibrium state.

Homogenization annealing has important influence on performance of material. During homogenization process, non-uniform and non-equilibrium microstructure transforms into uniform and balanced microstructure. Dendritic segregation is eliminated, producing a uniform microstructure. Low-melting point non-equilibrium phases disappear, and the transformation of phases leads the microstructure to balance, causing metastable precipitates to vanish. Soluble and rapidly diffusing elements such as magnesium and silicon are firstly diffused into solid solution, and the process is

quick. After homogenization treatment at 540 °C for 20 h, Mg₂Si, AlCuMgSi and Si phases had been dissolved into matrix completely, although Al₁₅(FeMn)₃Si₂ was dissolved rarely, the phase was spheroidized and the size of it was smaller. Meanwhile, homogenization also promotes the precipitation of other fine dispersoids. The grains grow up during homogenization process, which could be hindered by fine dispersoids precipitated during homogenization process. The plasticity of material is improved, resistance to deformation is decreased, the anisotropy of materials is weakened, and microstructures of the material tend to stabilize after homogenization process.

In the present work, Zn was added in the alloy, the Zn-containing phases were not found during solidification process and homogenization process, owing to high solubility of Zn element in Al.

It was clear from the above analysis that microstructural evolution of Al–Mg–Si alloy was that, AlCuMgSi intermetallic compound was firstly dissolved into matrix, and the remaining phase was Al₁₅(FeMn)₃Si₂. Mg₂Si and Si were dissolved completely. Al₁₅(FeMn)₃Si₂ phase became small and round after homogenization process.

5 Conclusions

1) As-cast microstructure of the alloy is typical dendrital, including α (Al), Al₁₅(FeMn)₃Si₂, Mg₂Si, AlCuMgSi and Si phases. According to the DSC curve of as-cast alloy, the ideal single-stage homogenization parameters of the alloy are 545 °C and 20 h.

2) After homogenization treatment at 545 °C for 20 h, AlCuMgSi, Mg₂Si and Si phases were dissolved completely. The remaining phase is Al₁₅(FeMn)₃Si₂. In order to improve material performance, the number of Al₁₅(FeMn)₃Si₂ constituents should be controlled strictly. The Zn-containing phases were not found during solidification process and homogenization process. During the homogenization process, the phases should be dissolved into matrix, under the condition accepted.

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Al–0.66Mg–0.85Si 合金均匀化过程中的组织演变

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摘 要: 采用金相显微镜、扫描电子显微镜、透射电子显微镜和差示扫描量热法研究 Al–0.66Mg–0.85Si 合金在均匀化过程中的组织演变。合金铸态组织呈典型的枝晶形貌, 存在 α (Al)、 $\text{Al}_{15}(\text{FeMn})_3\text{Si}_2$ 、 Mg_2Si 、 $Q(\text{Al}_{1.9}\text{CuMg}_{4.1}\text{Si}_{3.3})$ 和 Si 相。铸态组织中存在 2 种不同晶体结构的 Mg_2Si 相, 一种是在铸造过程中形成的, 另一种是在铸造完成后的空冷过程中形成的。经过 545 °C、20 h 均匀化处理之后, 组织中的 Q 、 Mg_2Si 和 Si 相已完全溶入基体, 残留的第二相主要是 $\text{Al}_{15}(\text{FeMn})_3\text{Si}_2$ 相。 $\text{Al}_{15}(\text{FeMn})_3\text{Si}_2$ 相的尺寸减小、球化并且在晶界上断续分布。在铸态和均匀化热处理状态中, 均未发现含 Zn 相。

关键词: Al–Mg–Si 合金; 组织演变; 均匀化; 金属间化合物

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