Improvement of Mechanical Properties in a High Silicon Al-Si-Mg Alloy through Effective Grain Refinement in Sand Casting

1. Introduction

Al-Si-Mg cast alloys have been widely used for making high integrity castings with a combination of good castability, strength, ductility and corrosion resistance [1-3], which are necessary for transport manufacturing to provide light weighting components. Sand casting is widely applied for the casting of Al-Si-Mg cast alloys. The most commonly used Al-Si-Mg cast alloy is A356/A357 [4]. However, sand castings usually provide inferior mechanical properties because of the existence of defects formed during solidification with a relatively low cooling rate.

Grain refinement has been proved as an important melt treatment during casting aluminium alloys in order to reduce or eliminate the casting defects, which can improve
the toughness, strength, formability and machinability [2, 5-9]. Al-Ti-B master alloys, in particular Al5Ti1B, have been widely used as grain refiners over the past several decades [10,11]. The Al5Ti1B master alloy essentially consist of TiB2 particles with extra Al3Ti and capable of offering remarkable grain refinement and performance improvement in the casting of wrought alloys. It is generally believed that a layer of Al3Ti is formed on the surface of TiB2 particles to induce grain refinement. However, the grain refinement using Al5Ti1B is hard to meet the expectations in cast Al-Si alloys, especially with a content of Si higher than 3.5 wt.% [12-15]. The reason is that Si in the melt reacts with Ti to form Ti-Si phases, which poison the TiB2 nucleation sites and consumes the Ti dissolved in the melt for grain growth restriction [16, 17]. Thus the decrease of grain refining efficiency of Al5Ti1B is a severe problem for high silicon cast aluminium alloys.

In order to increase the efficiency of grain refinement in high Si aluminium alloys, it is necessary to prevent the formation of Ti-Si phase through reducing the Ti content in the master alloy, which can transfer the constituent of the master alloy from TiB2 with extra Al3Ti to TiB2 with extra AlB2. Consequently, the poisoning effect is expected to be significantly reduced. In particular, the grain refinement can be enhanced under low cooling rate in sand casting process [18-22]. Al3Ti3B is developed with this philosophy and is expected to overcome the grain-refining problem accompanied with Al5Ti1B in high silicon aluminium alloys processed by sand casting process.

Therefore, Al5Ti1B and Al3Ti3B master alloys were investigated side-by-side to understand the grain refinement, microstructure and mechanical properties of a high silicon aluminium alloy using sand casting. The efficiency of grain refinement and the strengthening mechanism were assessed in the sand cast samples. The discussion focuses on the relationship between the microstructure and mechanical properties of cast alloys.

2. Experimental
2.1. Alloy Preparation

The cast Al9Si0.45Mg alloy in the form of ingot was melted in the 12-kg capacity clay-graphite crucible using an electric resistance furnace. During melting, the temperature of the furnace was controlled at 750 °C, and a melt of 8 kg was prepared each time. After one hour of homogenisation, Al-10 wt.% Sr master alloy was added into the melt to make the defined Sr content of 140 to 350 ppm, for modifying the morphology of the eutectic silicon phase during solidification. The melt was subsequently degassed through injecting pure argon into the melt by using a rotary degassing impeller at a speed of 350 rpm for 4 min. After degassing, the top surface of the melt was covered by commercial granular flux, and the melt was held for 10 min for temperature recovery, followed by adding 0.2 wt.% Al5Ti1B or 0.2 wt.% Al3Ti3B master alloys into the melt for grain refinement. After adding the grain refiner, the melt was slightly stirred before casting. The chemical compositions of the investigated alloys were measured by inductively coupled plasma atomic emission spectroscopy (ICP-AES), and the results are listed in Table 1.

| Table 1: Chemical Compositions of Experimental Alloys Analyzed by ICP-AES (wt.%) |
|-----------------|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| Alloy           | Si  | Mg  | Cu  | Fe  | Mn  | Ti  | Sr  | B   | Al  |
| A1(Al9Si0.45Mg+Al5Ti1B) | 8.70| 0.45| 0.00| 0.11| 0.06| 0.141|0.014|0.002|Bal.|
| A2(Al9Si0.45Mg+Al3Ti3B) | 8.73| 0.45| 0.00| 0.11| 0.06| 0.134|0.014|0.006|Bal.|

2.2. Sand Casting and Heat Treatment

With the intention of casting tensile test bars, the prepared melt was poured at 720°C into a British standard sand casting mould preheated at 150°C, as shown in Figures 1(a) and 1(b) shows the sand casting made by the sand mould, and four round tensile test bars with the size of φ20 mm × 170 mm were made from each casting, as indicated by the dashed rectangle box in Figure. 1(b). Four castings were made for each of the Al5Ti1B refined and Al3Ti3B refined condition. After kept at ambient condition for at least 24 h, the cast tensile test bars were subjected to T6 heat treatment in an electrical furnace equipped with forced air circulation, including solution treatment and artificial ageing. Solution treatment was carried out at 540
0°C for 8 h, followed by immediate water quenching to room temperature. Ageing treatment was performed at 170°C for 8 h, followed by air cooling to room temperature. The T6 heat-treated tensile test bars were then machined into the shape shown in Figure. 1(c) for tensile tests, with the gauge dimension of φ10 mm × 50 mm.

Figure 1: (a) British Standard Sand Casting Mould, (b) Sand Casting made by the Mould and (c) Machined Tensile Test Bar from the Casting

2.3. Characterization and Tensile Test

The microstructure was examined using the Zeiss optical microscopy (OM), the Zeiss SUPRA 35VP scanning electron microscope (SEM) equipped with energy dispersive X-ray spectroscopy (EDS), the JEOL-2100 transmission electron microscopy (TEM) and the D8 X-ray diffraction (XRD) instrument. The specimens for OM, SEM and XRD analysis were prepared by the standard technique of grinding. OM observation was conducted after polishing without any etching. Polarized OM observation of grain size was performed after anodised with Barker solution (97 vol.% H2O and 3 vol.% HBF4). Porosity area percentage was counted from the longitudinal section of three as-cast bars. SEM analysis was conducted after etching with 15 vol.% HCl. Post-loading fracture analysis was also performed via SEM. Thin specimens for TEM observation were prepared by standard electropolishing. The electrolytic solution was a mixture of nitric acid and methyl alcohol (2:8), used at -20 to -30°C and 20 V. TEM operating at 200 kV was used for bright field imaging, select area diffraction pattern (SADP) analysis, and high-resolution transmission electron microscopy (HRTEM) imaging. XRD analysis was conducted from 2 Theta degrees 25° to 100° at a scanning speed of 1°/min. tensile tests were conducted following the ASTM B557 standard using an Instron 5500 Universal Electromechanical Testing System. All the tensile tests were performed at room temperature. The gauge length of the extensometer was 50 mm and the ramp rate for extension was 1 mm/min. Each data reported with standard deviation was based on the mechanical properties obtained from 6 to 8 samples.

3. Results

3.1. As-cast Microstructure

Figures. 2(a) and (b) present the polarized optical micrographs showing the grain size of primary α-Al phase in the as-cast sand casting Al9Si0.45Mg alloys refined by Al5Ti1B and Al3Ti3B master alloys, respectively. The primary α-Al phase in the Al5Ti1B refined alloy shows coarse dendritic morphology. The primary α-Al phase in the Al3Ti3B refined alloy is significantly finer than the Al5Ti1B refined condition. The grain size of the primary α-Al phase is 750±220 μm in the Al5Ti1B refined alloy, while that is 443±125 μm in the Al3Ti3B refined alloy. The grain size of primary α-Al phase in the Al3Ti3B refined alloy is reduced by 41 %, when compared with the Al5Ti1B refined alloy. It could be confirmed that Al3Ti3B is more effective grain refiner for the high silicon Al9Si0.45Mg cast alloy than Al5Ti1B under the sand casting condition.

Figure 2: Polarized Optical Micrographs showing the Grain Size of Primary α-Al phase in the as-cast Sand Casting Al9Si0.45Mg Alloys Refined by (a) Al5Ti1B and (b) Al3Ti3B

Figures. 3(a) and (b) present the SEM morphology of the as-cast sand casting Al9Si0.45Mg alloys refined by Al5Ti1B and Al3Ti3B, separately. Primary α-Al phase, Al-Si eutectic phase and β-Mg2Si intermetallic phase were identified coexisting in the as-cast Al9Si0.45Mg alloys both refined by Al5Ti1B and Al3Ti3B. β-Mg2Si phase is located in the eutectic Al-Si eutectic region, and its size is larger than the eutectic Si phase, as indicated by the arrows in Figure 3.Obviously, the primary α-Al phase in the as-cast Al9Si0.45Mg alloy refined by Al3Ti3B is also much finer than that refined by Al5Ti1B under the SEM observation, which is consistent the results shown in Figure 2.
3.2. Microstructure after Heat Treatment

Figures 4(a) and (b) present the SEM morphology of the sand casting Al9Si0.45Mg alloys refined by Al5Ti1B and Al3Ti3B, separately, after solution treatment and ageing treatment. The primary α-Al phase and the spheroidised Si particles were clearly visible, but the β-Mg2Si intermetallic phase was hardly observed in the microstructure both refined by Al5Ti1B and Al3Ti3B, which indicated that the β-Mg2Si intermetallic phase was well dissolved into the α-Al matrix after the solution treatment. The saturated solid solution could ensure the precipitation of nanoscale strengthening precipitates in the α-Al matrix after ageing treatment, which contributes to the strengthening of the alloy after T6 heat treatment. The spheroidised Si particles in the heat-treated alloys refined by Al5Ti1B and Al3Ti3B are much similar to each other. Similar to the as-cast condition, the primary α-Al phase in the T6 heat-treated Al9Si0.45Mg alloy refined by Al3Ti3B is also much finer than that refined by Al5Ti1B, since T6 heat treatment hardly has any effect on the grain size of the primary α-Al phase.

3.3. Mechanical Properties after Heat Treatment

The Al9Si0.45Mg alloy is heat treatment strengthening alloy and mainly used after heat treatment, so its mechanical properties after heat treatment were focused rather than the mechanical properties in the as-cast state. Figure 6 presents the tensile properties of the sand casting Al9Si0.45Mg alloys refined by Al5Ti1B and Al3Ti3B, after T6 heat treatment. The yield strength (YS), ultimate tensile strength (UTS) and elongation (El) of the Al5Ti1B refined alloy are 289±2 MPa, 320±4 MPa and 2.4±0.4 %, respectively. The Al3Ti3B refined alloy provides the YS of 300±1 MPa, the UTS of 335±5 MPa and the El of 4.5±0.6 %. It is clear that the strength could be increased, and the
elongation could be significantly increased by 88% in the Al3Ti3B refined alloy, when compared with the Al5Ti1B refined alloy.

Figure 6: Tensile Properties of the Sand Casting Al9Si0.45Mg Alloys Refined by Al5Ti1B and Al3Ti3B after T6 Heat Treatment

4. Discussion

4.1. Precipitation Strengthening

Figure 7 presents the TEM micrographs taken along the <001>Al axis showing precipitate in the Al9Si0.45Mg alloy after T6 heat treatment. Figure 7(a) shows the bright field image of precipitate in the α-Al matrix, embedded β" precipitate and lying β" precipitate were found in the α-Al matrix when observed along <001>Al axis, which indicates that the β" precipitate is needle-like, and the embedded β" precipitate and lying β" precipitate were the same β" precipitate in nature. Figure 7(b) shows the SADP of α-Al matrix and β" precipitates in Figure 7(a), the large bright points are diffraction points of the α-Al matrix, while the grey cross lines between the bright points are diffraction patterns of β" precipitates, which indicates that the β" precipitates are in the metastable state and the heat-treated alloy is in the peak strength state. Figure 7(c) shows the HRTEM image of the β" precipitate lying on the (001)Al plane, and Figure 7(d) shows the corresponding FFT patterns of the rectangle area in Figure 7(c), which verifies that the lying precipitate is β", and the β" precipitate is fully coherent with the α-Al matrix. As reported in literatures [24, 25], the needle-like β" precipitate provides peak strengthening of the T6 heat-treated Al-Si-Mg alloy, which contributes to the high yield strength of above 285 MPa of the sand casting Al9Si0.45Mg alloy after T6 heat treatment.

Figure 7: TEM Micrographs taken along the <001>Al Axis showing the β" Precipitate in the Sand Casting Al9Si0.45Mg Alloy after T6 Heat Treatment, (a) Bright Field Image, (b) SADP of (a), (c) HRTEM Image of β" Precipitate and (d) FFT Pattern of (c)

4.2. Effects of Grain Refinement on Microstructure

Figure 8(a) shows the XRD patterns of the Al5Ti1B master alloy used for refinement in the present study, Al5Ti and TiB2 particles were found coexisting in the Al5Ti1B master alloy. It has been well documented that the particles introduced into the melt through the addition of Al5Ti1B grain refiner are the soluble Al3Ti and the insoluble TiB2 particles, which is consistent with the XRD results in Figure 8(a). The TiB2 particles that act as heterogeneous nucleation sites could be poisoned by Si by coating the surfaces with Ti-Si compounds [12-16, 26]. The dissolved Ti from Al3Ti particles, which also contributes to grain refinement under Al5Ti1B by slowing down the growth of the freshly formed α-Al crystals while partitioning between the solid and liquid phases, is also rendered ineffective as it also forms Ti-Si compounds and precipitates out of the melt. It has been reported that solute Ti hardly offers any grain refinement effect in Al-Si alloys with 7 wt.% Si [15]. The Al9Si0.45Mg cast alloy contains a high level of Si at 8.7 wt.%, so the poisoning effect of TiB2 particles is significant, and the solute Ti might hardly provide growth restriction for the primary α-Al phase, due to the formation of Ti-Si compounds, which results in the coarse primary α-Al grains of 750±220 in the alloy refined by Al5Ti1B.

Figure 8(b) shows the XRD patterns of the Al3Ti3B master alloy used for refinement in this article,
TiB₂ and AlB₂ particles were found coexisting in the master alloy, while no Al₃Ti particles were found. AlB₂ particles could also act as heterogeneous nucleation sites for α-Al [19, 27], thus the XRD results indicate that the Al₃Ti₃B grain refiner does not supply soluble Al₃ Ti particles (solute Ti), and relies on the insoluble AlB₂ and TiB₂ borides to promote heterogeneous nucleation. Borides in the Al₃Ti₃B grain refiner are nearly three times as many as that in Al₅Ti₁B, since the B content dictates the population of boride particles. This was also evidenced by the B content listed in Table 1. Furthermore, it has been reported that Si promotes the heterogeneous nucleation of primary α-Al on AlB₂ [27]. Therefore, the performance of the Al₃Ti₃B for the refinement of the high silicon Al₉Si₀.₄₅Mg cast alloy is clearly superior with respect to that of Al₅Ti₁B, under the sand casting condition, with the decrease of size of the primary α-Al grain to 443±125 μm.

4.3. Effects of grain refinement on strength and ductility

Generally, the strength of cast aluminium alloys is decided by grain size strengthening, secondary phase strengthening, solution strengthening, precipitate strengthening and strain strengthening. So the strength of the sand casting Al₉Si₀.₄₅Mg cast alloys after T₆ heat treatment is dependent on the grain size of primary α-Al phase, the spheroidised secondary Si phase and the β'' precipitation phase in the α-Al matrix. From Figure 4, the spheroidised secondary Si particles in the Al₅Ti₁B and Al₃Ti₃B refined alloys are much similar to each other both in size and shape. Moreover, for structural materials, the increase in strength is inevitably sacrificed by the reduction in ductility, which is known as the strength-ductility trade-off dilemma [28]. As shown in Figure 6, the strength of the Al₅Ti₁B refined alloy is higher than that of the Al₅Ti₁B refined alloy, so the increase of strength in Al₃Ti₃B refined alloy should contribute to the decrease of ductility, from the view point of strength and strength-ductility trade-off dilemma. However, the ductility of the Al₃Ti₃B refined alloy is significantly higher than that of the Al₅Ti₁B refined alloy. Thus the ductility of the T₆ heat-treated Al₉Si₀.₄₅Mg cast alloys is mainly controlled by defects and the primary α-Al phase.

According to Campbell’s theory, the bi-film oxides defect controlled the failure properties of cast aluminium alloys, the precipitation of inoculation particles from the addition of Al-Ti-B grain refiners can sediment oxides in the melt before pouring into the mould, which will reduce the content of oxides in the casting, and that is beneficial to the ductility of cast aluminium alloys, since crack is easy to start from the oxides defect [23]. As discussed in section 4.2, the inoculated particles under the addition of Al₃Ti₃B grain refiner were nearly three times as many.

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$$\sigma_y = \sigma_0 + \frac{k}{\sqrt{d}}$$

Where $\sigma_y$ (MPa) is the yield strength, $\sigma_0$ is a material constant for the starting stress for dislocation movement, $k$ is the strengthening coefficient, and $d$ (μm) is the grain diameter.
as that of the Al5Ti1B condition, so the sedimentation of oxides under the refinement of Al3Ti3B was significantly superior to that under the refinement of Al5Ti1B. Thus the content of oxides in the Al3Ti3B refined alloy is lower than that of the Al5Ti1B refined alloy, which contributes to the improvement of ductility in the Al3Ti3B refined alloy, when compared with the Al5Ti1B refined alloy. As shown in Figure 2, the grain size of the primary α-Al phase in the Al3Ti3B refined alloy is reduced by 41%, when compared with the Al5Ti1B refined alloy, so the number and area of grain boundary in the Al3Ti3B refined alloy are larger in the Al3Ti3B refined alloy comparing with the Al5Ti1B refined alloy. After the formation of crack source from the oxides, the crack will propagate from the crack source to the whole fracture section. The increased number and area of grain boundary in the Al3Ti3B refined alloy will be more efficient in impeding the propagation of crack, which contributes to the increase of ductility as well, when compared with the Al5Ti1B refined alloy. Thus the increase of ductility in the Al3Ti3B refined alloy resulted from the reduced oxides as well as the decreased grain size of the primary α-Al phase, when compared with the Al5Ti1B refined alloy.

5. Conclusions

1. Al3Ti3B is more effective than Al3Ti3B for the grain refinement of high silicon cast aluminium alloys processed by sand casting. The as-cast grain size of primary α-Al phase in a sand cast Al9Si0.45Mg alloy refined by Al5Ti1B is 750±220 μm, and that is significantly decreased to 443±125 μm in the Al3Ti3B refined alloy.

2. Both the strength and ductility are improved in the sand cast alloy refined by Al3Ti3B, after T6 heat treatment, when compared with the alloy refined by Al5Ti1B. With the change of grain refiner from Al5Ti1B to Al3Ti3B, the yield strength is increased from 289±2 MPa to 300±1 MPa, and the ultimate tensile strength is increased from 320±4 MPa to 335±5 MPa, while the elongation is significantly increased by 88% from 2.4±0.4% to 4.5±0.6.

3. TiB2 and Al3Ti coexist in the Al5Ti1B master alloy, while TiB2 and AlB2 coexist in the Al3Ti3B master alloy. It is expected that the co-existence of AlB2 and TiB2 in Al3Ti3B results in the significant grain refinement of the high silicon aluminium alloy under sand casting condition. The increase of strength in the sand cast alloy refined by Al3Ti3B resulted from the refinement of the primary α-Al grains, while the increase of ductility in the sand casted alloy refined by Al3Ti3B is attributed to the reduce defect levels as well as the decreased grain sizes.

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References


