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FADING MECHANISM ON GRAIN REFINEMENT AND MODIFICATION WITH Al-B-Sr MASTER ALLOYS IN AN ALUMINIUM-SILICON CAST ALLOY

A fading mechanism during casting of treated Al-B-Sr master alloys in an aluminium-silicon cast alloy was investigated. Two different master alloys, the Al-3%B-3%Sr and Al-4%B-1%Sr were demonstrated for the efficiency test both grain refinement and modification microstructure. From experimental result, the addition of Al-3%B-3%Sr master alloy led to a refined grain size and fully modified eutectic Si. However, smaller grain sizes were found with Al-4%B-1%Sr master alloy addition while eutectic Si had coarser acicular morphology. The formation of high amounts of SrB_6 compound in the Al-3%B-3%Sr master alloy resulted to increase of grain size and eutectic Si. In fading mechanism test when holding the melt for prolong time, the agglomeration of the SrB_6 and AlSiSr compounds at the bottom of the casting specimen was important factors that decreased both grain refinement and modification efficiency. The stirring of the melt before pouring was found that the grain size and Si morphology were improved.

Keywords: Aluminium; Grain refinement; Modification; Fading mechanism

1. Introduction

Aluminium-silicon (Al-Si) cast alloy is important materials for the manufacture of highly complex parts because of their excellent castability in several casting methods such as sand, gravity or pressure die casting processes. The quality of casting can be improved by several treatment methods. The grain refinement and eutectic Si modification are widely used to improve their microstructure and mechanical properties of Al-Si cast alloys [1-6]. Normally, the addition of conventional master alloy such as the Al-Ti, Al-Ti-B or Al-B and combinations between grain refiner and modifier alloy system such as Al-Ti-B-Sr, Al-Ti-C-Sr and Al-B-Sr [7-16]. These combined master alloys allow for easier control of chemical composition compared to conventional additions. The ratio of grain refiner and modifier elements in liquid metal is an important factor for their efficiency. However, the agglomeration and sedimentation of the $AlTi_3$, TiB_2 [17] and SrB_6 compounds decrease the grain refinement efficiency of those master alloys, especially when holding the melt for prolonged period in the range of 60-120 min [18,19]. The formation of SrB_6 compound is caused the concentration of free strontium (Sr) atom in the melt before the nucleation of α-Al dendrite, which are involved in changing the acicular to a fibrous morphologies of eutectic Si [20]. It had been well known that the SrB_6 compound formed in the liquid during holding time in the furnace as the cubic shape. Liu et al [21] reported that the combined addition between the grain refiner (Al-5%Ti-1%B) and modifier (Al-10%Sr) can improve the mechanical properties of Al-7.5%Si-4%Cu cast alloys. Cui et al [22] found that new Al-3%B-5%Sr can refine the α-Al dendrite and change the acicular Si to a fibrous morphologies in casting of A356 alloy. Limmaneevichitr et al [17] reported that the difference in density of both $TiAl_3$ and TiB_2 compounds are main reduction factor the grain refinement efficiency when using Al-5%Ti-1%B master alloy.

From the previous research, there were no investigation and report about the agglomeration and sedimentation of SrB_6 and other compounds in the Al-B-Sr master alloy system. Therefore, the purpose of present investigation is to study the fading mechanism in the grain refinement and modification of Al-Si cast alloy by addition of Al-3%B-3%Sr and Al-4%B-1%Sr master alloys.

2. Experimental procedure

2.1. Master alloys prepared

In this research work, the amount of alloying elements in the master alloy was calculated by atomic percent (at.%) and the

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addition of master alloy into the melt during a casting was used weight percent (wt.%). In this experiment, the development of Al-3%B-3%Sr and Al-4%B-1%Sr master alloys was prepared by the Al-6%B and Al-10%Sr master alloys. Firstly, the Al-6%B was melted in the alumina crucible by using an electric resistance furnace. Then, the Al-10%Sr was added into the melted and holding for 30 min. Finally, the melt was poured into a stainless steel mould with control a cooling rate for 0.6°C/s. The microstructures were observed by the optical microscope (OM) and scanning electron microscope (SEM). The chemical compositions of their compound in the master alloy were analysed with the energy dispersive spectrometer (EDS) and X-ray diffraction (XRD) techniques.

The study of grain refinement and modification efficiencies was cast in the stainless steel mould with a cooling rate of 0.6°C/s. The melt was treated by addition of 4wt. % master alloys. Before pouring into the mould the melt was stirred by Ar gas injection. The holding times before pouring were 10 and 60 min. The short and prolonged holding times were designed to order to investigate the macrostructure and microstructure.

2.2. Evaluation of the fading mechanism test

A carbon dioxide (CO_2) silicate sand mould was designed inside the carbon steel pipe (4 inch) with cylinder shape. The melt will solidify contact the sand in order to prevent the formation of other intermetallic compounds from C, Fe and other elements form the pipe. The un-modified A356 (Al-7%Si-0.3Mg) alloy was heated and melted in a CO_2 silicate sand mould at 800°C. Ar gas was used to remove inclusions in the casting process for 5 min. The melt was treated with 4wt. % master alloy and stirred with a stainless steel tool. The covering flux was used to prevent gas diffusion into the melt and undesired oxide inclusions. The holding time in the mould was prolonged for 120 min before solidification. The specimen was sectioned from the bottom to top of the casting piece. The microstructure and macrostructure specimens were grinded and polished by Keller's and Tucker's reagents, respectively. The grain size was measured by using the linear intercept method according to ASTM E112. The morphology of the sedimentation compound was deep-etched by the 15% HCl in mixed with distilled water. The chemical compositions of each compound in the master alloys were analysed by using the SEM-EDS.

3. Result and discussion

3.1. Characterization of master alloys

The microstructure of the Al-3%B-3%Sr and Al-4%B-1%Sr master alloys consisted of the AlB_{12}, AlB_2, Al_4Sr and SrB_6 compounds, as shown in Fig. 1(a) and (b). In Fig. 1(c) showed the SEM backscattered electron images of Al-3%B-3%Sr, it was clearly that high amounts of SrB_6, Al_4Sr and AlB_2 distributed in the α-Al matrix, while the AlB_{12} compound is surrounded by the SrB_6 compound. The microstructure of Al-4%B-1%Sr consisted of clusters of AlB_{12} and small amounts of SrB_6 while the AlB_2 and Al_4Sr compounds were present in Fig. 1(d).

Fig. 2 showed compared X-ray diffraction pattern between Al-3%B-3%Sr and Al-4%B-1%Sr master alloys consist of AlB_{12}, AlB_2, Al_4Sr, SrO_2, SrF and SrB_6 compounds. The intensity of the SrB_6 compound of Al-3%B-3%Sr was higher than that of Al-4%B-1%Sr which was consistent with the microstructure in Fig. 1(a-d).

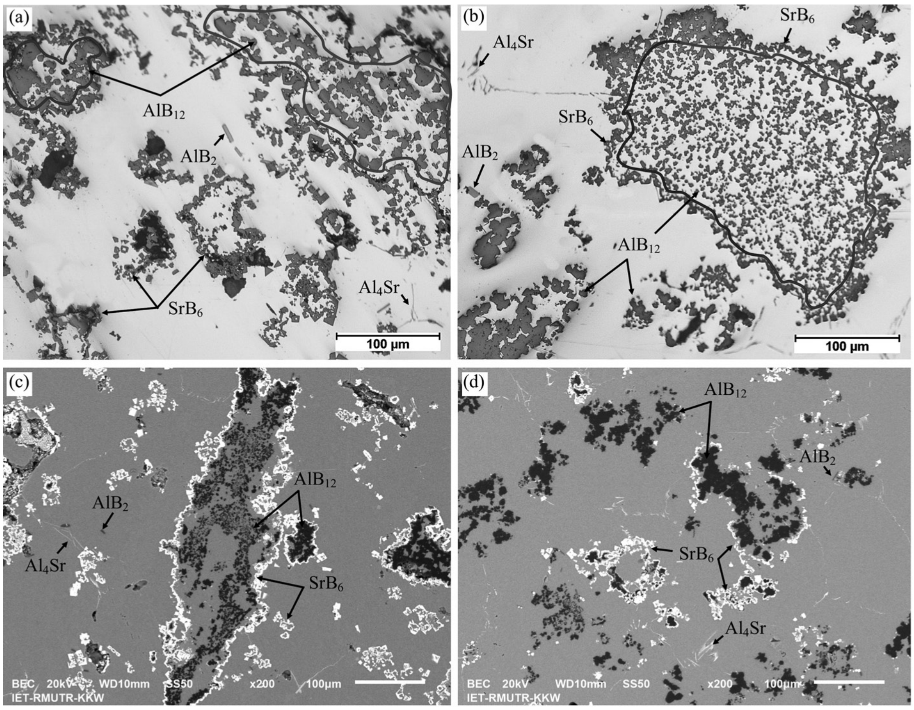


Fig. 1. Optical (a, b) and SEM (c, d) micrographs of the master alloys: (a, c) Al-3%B-3%Sr; (b, d) Al-4%B-1%Sr, respectively

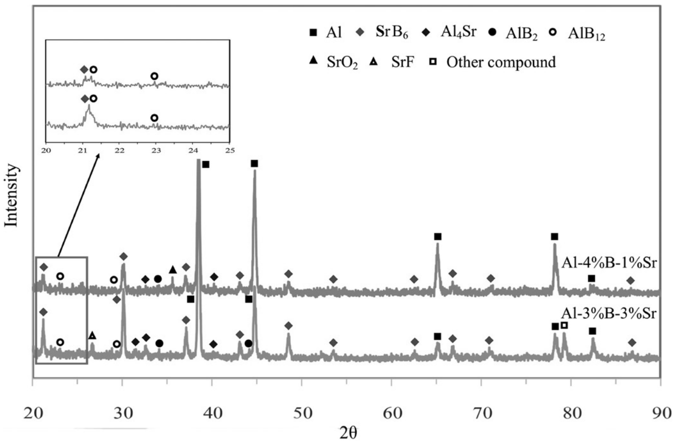


Fig. 2. Compared X-ray diffraction pattern between Al-3%B-3%Sr and Al-4%B-1%Sr master alloys

3.2. Effect of grain refinement and modification efficiencies

In this section the macrostructure uses for measured grain size while microstructure uses for observation the typical characteristic of compounds.

Macrostructure of as-cast A356 in Fig. 3(a) was showed the coarse dendrite with grain size of 3120 µm. and microstructure in Fig. 3(b) consisted of the acicular eutectic Si. These typical characteristic had low quality and mechanical properties in Al casting process.

Fig. 3(c-f) and Fig. 3(g-j) showed the macrostructure and microstructure of A356 treated with the Al-3%B-3%Sr and Al-4%B-1%Sr master alloys with different holding times in range of 10-60 min. respectively. Fig. 3(k) showed the measured grain sizes of each casting condition. It was clearly that by treated with of Al-3%B-3%Sr master alloy, the grain size decreased from 3210 µm (as-cast) to fine grain size of 620 µm (Fig. 3(c). Then, when holding time was longer the grain become coarser again to 1020 µm (Fig. 3(e). The eutectic Si was fully modified with 10 min Fig. 3(d) compared to the prolonged holding time (Fig. 3(f), simultaneously.

This is because the concept development of Al-3%B-3%Sr master alloy, the ratio of B and Sr was 3:3, which enhances a formation of the SrB_6 compound. Therefore, free B atom in the melt is low, leading to medium refinement in order to reduce the grain size. The Al_4Sr and SrB_6 compounds in this master alloy are very effective to change the eutectic Si after 10 min of holding time.

In the Al-4%B-1%Sr master alloy, we increased B content to higher and reduced Sr to lower, as the ratio of 4:1. The grain size was fully refined into relatively smallest grains of 309 µm and 315 µm, as shown in Fig. 3(g) and Fig. 3(i). The eutectic Si was partially modified form the acicular and fibrous morphologies, as shown in Fig. 3(h) and Fig. 3(j).

From the efficiency test, it was indicated that the grain refining and modifying compounds in master alloys affect to the grain refinement and eutectic modification of α-Al and eutectic Si. The Al-3%B-3%Sr master alloy can decrease the grain size to a small grain within 10 min. However, the grain size was increased when prolonger holding time. This fading phenomenon can be explained by the high amount and high density of

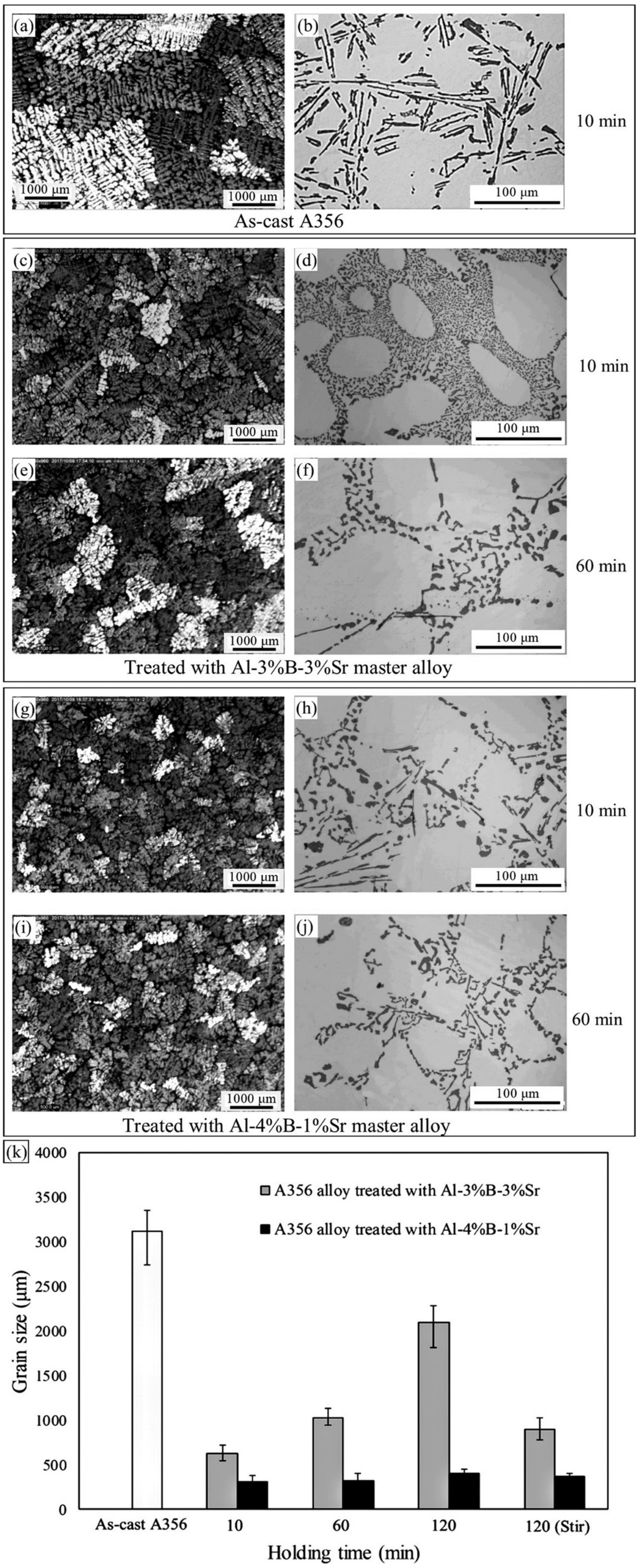


Fig. 3. Macrostructure and microstructure: (a-b) As-cast A356; (c-f) Treated with Al-3%B-3%Sr; (g-j) Treated with Al-4%B-1%Sr; (k) Measured grain sizes in range of 10-120 min of holding times

the SrB_6 compound, it was not appropriate for prolong holding time. High grain refining efficiency was found the Al-4%B-1%Sr master alloy, while it had lower eutectic modification efficiency compared to Al-3%B-3%Sr master alloy.

3.3. Fading phenomena test by the cylinder-CO_2 sand mould

The macrostructure of specimens were held time for 120 min as shown in Fig. 4. The as-cast A356 alloy was very coarse grain as shown in Fig. 4(a). In Fig. 4(b), the addition of Al-3%B-3%Sr master alloy had slightly effect on grain size compared to conventional as-cast A356 alloy. It was clearly that the A356 alloy treated with Al-3%B-3%Sr presented low grain refinement because of high SrB_6 compound content leads to a decrease in the grain refinement efficiency [18,19]. The relatively smallest grain size was achieved by addition of Al-4%B-1%Sr master alloy, as clearly shown in Fig. 4(c). The grain refining efficiency depend on the concentration of the AlB_{12} compound, which has high potential for the heterogeneous nucleation site in the melt during casting [23].

Fig. 5 showed the acicular eutectic Si morphology in the as-cast A356. From the bottom to 30-60 mm. of specimen of both

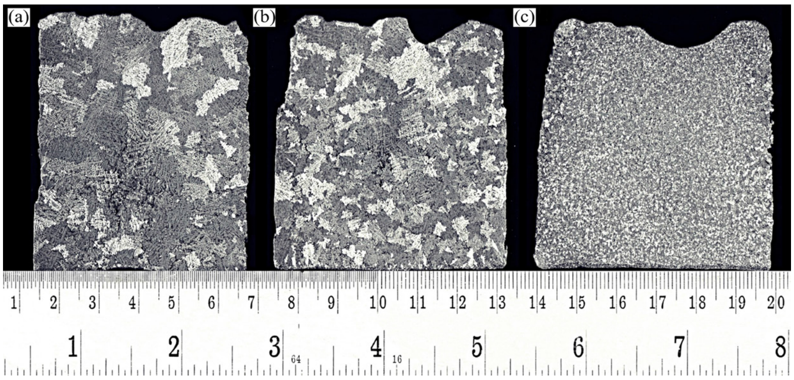


Fig. 4. Macrostructure of specimens were held time for 120 min. (a) As-cast A356; (b) Treated with Al-3%B-3%Sr; (c) Treated with Al-4%B-1%Sr

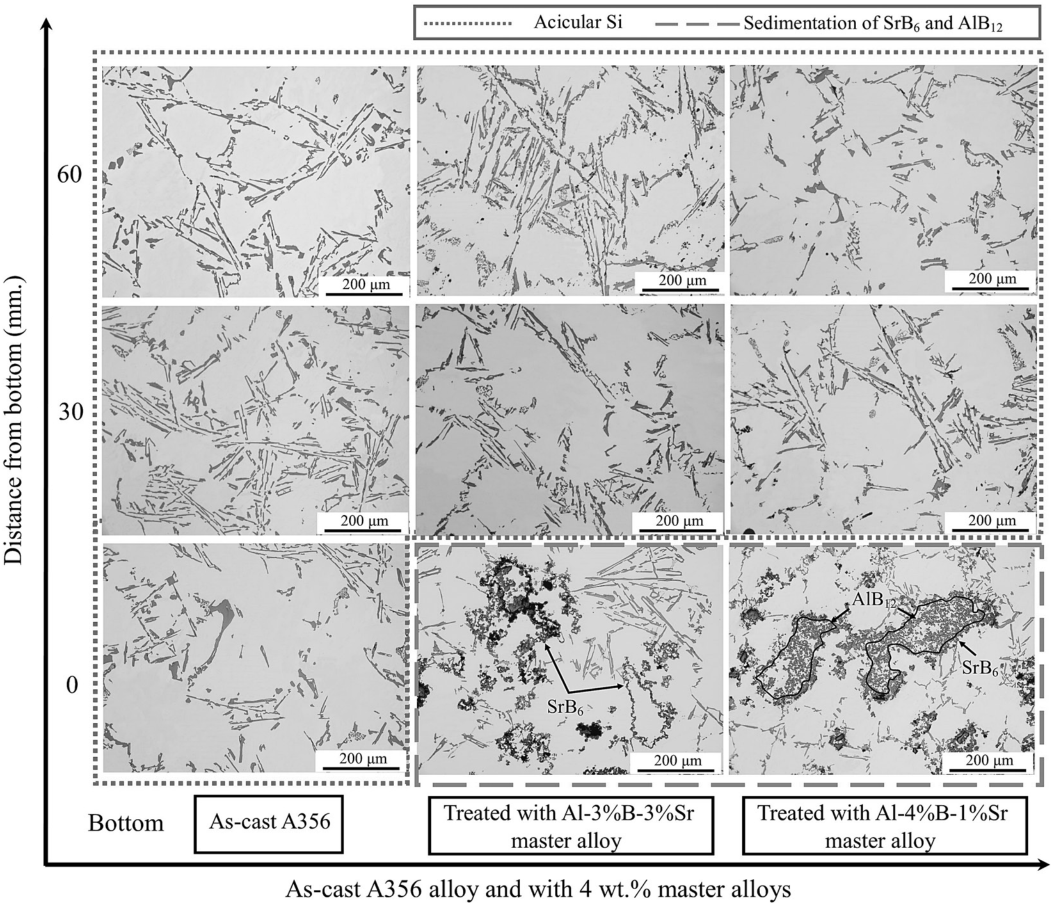


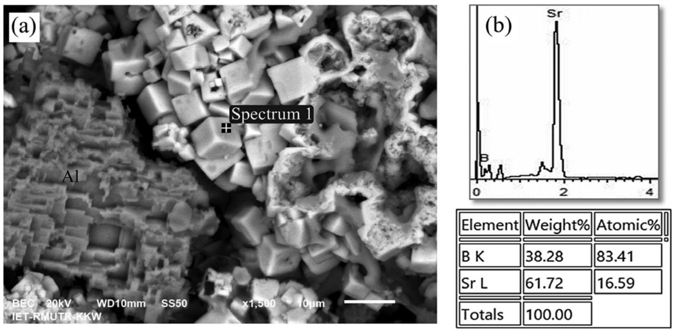
Fig. 5. Sectioned microstructures from bottom to the top of as-cast A356 (Left column), treated with Al-3%B-3%Sr (Middle column) and treated with Al-4%B-1%Sr (Right column), there was took from 120 min holding time

treated master alloys. It was indicated that the SrB_6 and Al_4Sr compounds does not modify the acicular Si into a fibrous morphology. This is because prolong holding time for 120 min. At the bottom of treated with Al-3%B-3%Sr specimen, it was found the SrB_6 compound (a black color phase). While at the bottom of treated with Al-4%B-1%Sr specimen, it was found both SrB_6 and AlB_{12} compounds. The formation of those compounds at bottom of treated specimens was caused by the sedimentation of higher density compounds. This phenomenon had been well known as, the fading of grain refinement and eutectic modification [17].

Fig. 6 showed the deep-etched SEM micrograph and point EDS spectrum of sectioned bottom specimen of A356 alloy 4wt. % treated of Al-3%B-3%Sr master alloy. It was clearly seen that the SrB_6 compound was cubic-like morphology in the α-Al matrix. The EDS was used to identify the chemical compositions of the compound, as shown in Fig. 6(b). Spectrum 1 consisted of B and Sr based compositions. This was resulted from a SrB_6 compound formation.

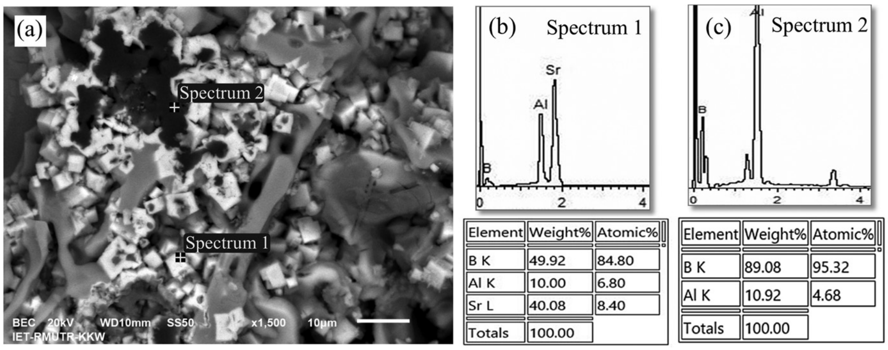
Fig. 7 showed the deep-etched SEM micrograph and point EDS spectrum of sectioned bottom specimen of A356 alloy 4wt. % treated of Al-4%B-1%Sr master alloy. It was found that the AlB_{12} compound was surrounded by the SrB_6 compound with a similar microstructure of it master alloy. The spectrums 1 and 2 present the chemical-based composition in this compound, which consists of Al, B and Sr as shown in Fig. 7(b, c).

Many studies have reported the Si modification efficiency in a hypoeutectic Al-Si alloy, which indicated that Sr addition could modify the eutectic Si phase over a prolonged time (2 hours) [24]. We also try to find an evidence of solidified cast specimens. Fig. 8 showed the deep-etched SEM micrograph



Element	Weight%	Atomic%
B K	38.28	83.41
Sr L	61.72	16.59
Totals	100.00	

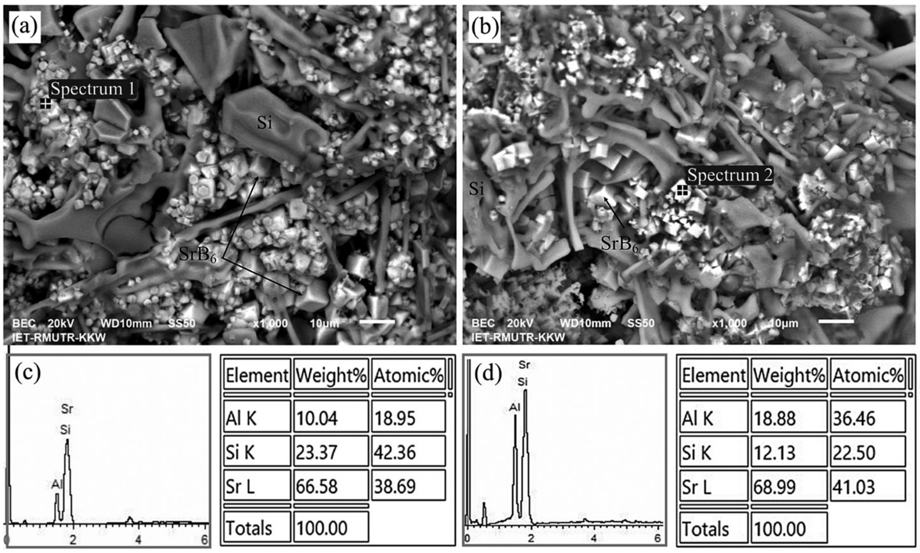
Fig. 6. (a) Deep-etched SEM micrograph and (b) Point EDS spectrum of sectioned bottom specimen of A356 alloy treated with Al-3%B-3%Sr, it was took from 120 min holding time



Element	Weight%	Atomic%
B K	49.92	84.80
Al K	10.00	6.80
Sr L	40.08	8.40
Totals	100.00	

Element	Weight%	Atomic%
B K	89.08	95.32
Al K	10.92	4.68
Totals	100.00	

Fig. 7. (a) Deep-etched SEM micrograph and (b, c) Point EDS spectrums of sectioned bottom specimen of A356 alloy treated with Al-4%B-1%Sr, it was took from 120 min holding time



Element	Weight%	Atomic%
Al K	10.04	18.95
Si K	23.37	42.36
Sr L	66.58	38.69
Totals	100.00	

Element	Weight%	Atomic%
Al K	18.88	36.46
Si K	12.13	22.50
Sr L	68.99	41.03
Totals	100.00	

Fig. 8. Compared SEM micrographs and EDS spectrums at the bottom specimens of A356 alloy: (a, c) treated with Al-3%B-3%Sr; (b, d) treated with Al-4%B-1%Sr, there was took from 120 min holding time

of treated Al-3%B-3%Sr and Al-4%B-1%Sr. It is found that a cubic-like particle is SrB_6 compound. The AlSiSr compound is formed adjacent to the eutectic Si phase, this phase is formed during solidification process. The EDS spectrums clearly presented of chemical compositions of the compound as shown in Fig. 8(c) and (d).

3.4. Effect of stirring before pouring on grain refinement and modification efficiencies

In the previous sections described the fade phenomenon on both grain refinement and modification efficiency. Limmaneevichitr et al [17] reported that stirring the melt in A356 alloy with Al-5%Ti-1%B and prolonged time can improve the refinement of α-Al. Thus, this experimental study also considers this influence. The macrostructure of the as-cast A356 alloy shows that the grain size is large, as shown in Fig. 9(a). It was clearly that stirring the melt before pouring influenced grain size. A reduction in grain size was found with Al-3%B-3%Sr addition as shown in Fig. 9(b) which can compares to the stirred melt to the non-stirred melt in Fig. 4(b). The smallest grain size was achieved by the addition of Al-4%B-1%Sr with stirring the melt, as shown in Fig. 9(c). The grain refinement efficiency will depend on the amount of AlB_{12} compound in the melts before pouring. Moreover, a black colour phase at the bottom of the specimen was disappeared by stirred before pouring. The recovery of grain refinement could be from the agitated SrB_6 compound diffusing into the melt. Thus, the final casting can be achieved with a relatively small grain size.

The eutectic Si morphology of stirring the melt before pouring was shown in Fig. 10. The fibrous and small acicular eutectic Si were formed in the A356 treated with both master alloys. The agglomerated and sedimented of compounds were not found at the bottom region of specimen. Moreover, the distributed AlB_2 compound was found in the treated with Al-4%B-1%Sr specimen. These results confirmed that stirring the melt before pouring and solidification can improve the eutectic Si efficiency.

Fig. 11 showed measured grain size of un-treated Al-Si-Mg cast alloy showed an average grain size of 2,700 mm. In comparison, the addition of Al-3%B-3%Sr master alloy decreased the average grain size to 1,800mm at the bottom of specimen. The addition of Al-4%B-1%Sr master alloy had the smallest average grain size of approximately 300-400 mm both non-stirred and stirred conditions. The stirring the melt before pouring by a stainless steel tool and Ar gas injection for 3 min reduced the grain size of treated with Al-3%B-3%Sr specimen in the range of 870-1250 mm. The grain size increased further from the bottom to 1400-1600 mm. Therefore, stirring had less influence on the treated Al-4%B-1%Sr specimen compared to the Al-3%B-3%Sr specimen.

3.5. The fading mechanism

The fading mechanism of both master alloys was discussed in this section. From the development of the Al-3%B-3%Sr master alloy contained the AlB_2, Al_4Sr and AlB_{12} compounds, which were surrounded by the SrB_6 compound. When addition it into the melt these compound were dissolved. During solidification, the AlB_2 formation can promote the α-Al dendrite formation. The α-Al will reject Si, Sr and other elements into the melt because they have low solubility. From the Al-Si-B-Sr phase diagram shows that the SrB_6 formation temperature is in the range of 650-900°C, which depends on the B and Sr content [25]. The sedimentation of SrB_6 decreased the coherence fraction of the solid, and the solute concentration of the grain refiner in the melt as shown in (Fig. 6). The large grain size of A356 treated with Al-3%B-3%Sr master alloy caused sedimentation of SrB_6, which has a high density (3.422 g/cm^3) compared to Al (2.7 g/cm^3) [19].

For development of the Al-4%B-1%Sr master alloy contained SrB_6, Al_4Sr and AlB_{12} compounds as shown in Fig. 1(b). When addition in to the melt these compounds were dissolved. The smallest grain size of A356 treated with the Al-4%B-1%Sr master alloy was caused by the high solute concentration of the grain refiner [26]. The fading after a prolonged time of 2h did

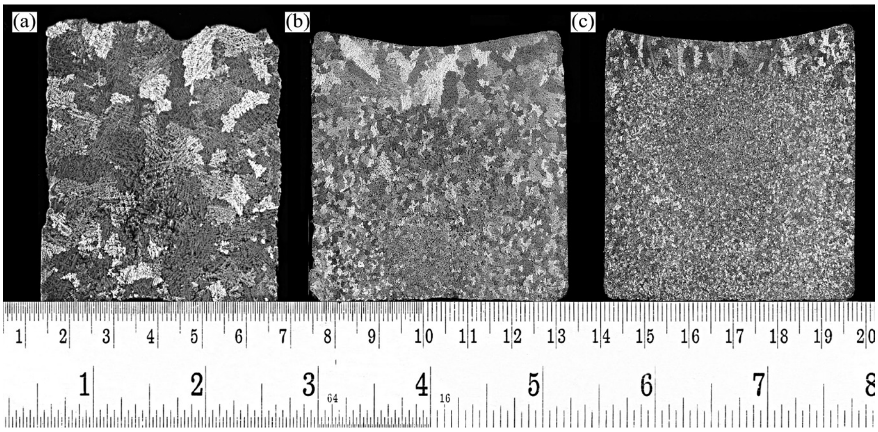


Fig. 9. Compared macrostructures after stirred the melt before pouring: (a) as-cast A356; (b) treated with Al-3%B-3%Sr; (c) treated with Al-4% B-1%Sr, it was took from 120 min holding time

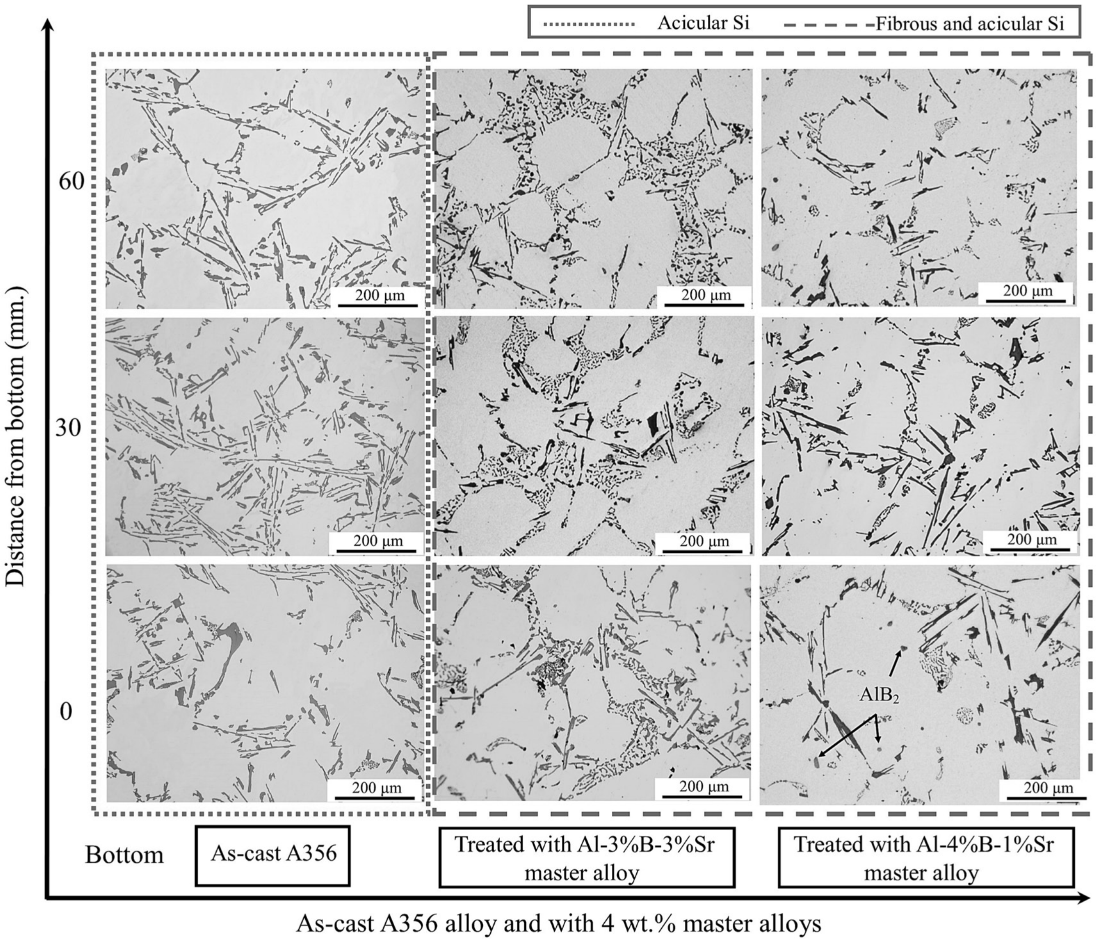


Fig. 10. Sectioned microstructures (after stirred the melt before pouring) from bottom to the top of as-cast A356 (Left column), treated with Al-3%B-3%Sr (Middle column) and treated with Al-4%B-1%Sr (Right column), there was took from 120 min holding time

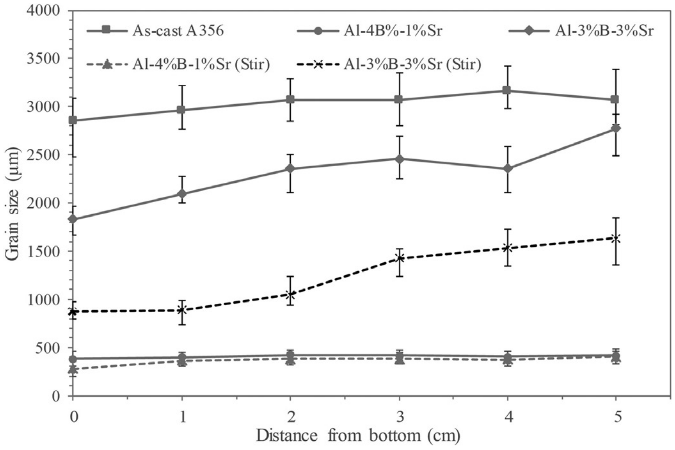


Fig. 11. Comparison measured grain size of various casting conditions of treated with Al-3%B-3%Sr and Al-4%B-1%Sr master alloys, there was took from 120 min holding time

not affect grain refinement efficiency. Moreover, high B content in Al-4%B-1%Sr master alloy led to increase grain refinement efficiency compared with the Al-3%B-3%Sr master alloy. It was also found that the AlSiSr compound was agglomerated near the eutectic Si at the bottom specimens both Al-3%B-1%Sr and Al-4%B-1%Sr master alloys as shown in Fig. 8(a) and (b), leading to a decreased eutectic Si modification efficiency. Normally, AlSiSr compounds will form in a eutectic Si region at a high Sr content [24]. The quantity of AlSiSr depends on proportion to Sr content in the master alloy [27]. However, this experiment controlled the fading mechanism of the high density of various compounds in master alloys with prolonged time. Thus, the AlSiSr compound at the bottom of the cast specimen did not contribute to change the Si morphology.

Stirring method before pouring the melt can recover the gain refinement and modification efficiencies. The grain sizes of A356 treated with Al-3%B-3%Sr master alloy were decreased to 52% compared to un-treated specimen. While, the addition of treated Al-4%B-1%Sr master alloy have full grain refinement similar to the non-stirred specimen. The mechanical stirring method recovered B and Sr concentration from sedimentation SrB_6 compound into the melt, especially due to the high SrB_6 compound content. The eutectic modification efficiency is improved by stirring before solidification and the Si morphology was modified from the acicular to fibrous.

Form all experiments in this research work, the fading phenomenon of both master alloys causes the agglomeration and sedimentation of the SrB_6 and AlSiSr at the bottom of casting, which are important factors for decreasing both grain refinement and modification.

4. Conclusion

1. The microstructure of developed master alloys contained AlB_{12}, AlB_2, Al_4Sr and SrB_6 compounds. The α-Al grain refinement efficiency depended on the amount of AlB_{12}, AlB_2 and SrB_6 compounds.
2. The addition of 4 wt.% Al-3%-3%Sr master alloy with 10 min of holding resulted in full grain refinement and eutectic Si modification. The grain size increased when the holding time was increased to 60 min. The Al-4%B-1%Sr master alloy successfully refined α-Al into relatively small grain sizes at every holding time, while eutectic Si was modified and exhibited acicular and fibrous morphologies.
3. When holding time for a 120 min, the Al-3%B-3%Sr master alloy had poor refinement of α-Al, while eutectic Si was not modified. The Al-4%B-1%Sr master alloy successfully refined α-Al into relatively small grain sizes, while eutectic Si was not modified.
4. The fading mechanism of grain refinement and modification resulted in agglomeration and sedimentation of SrB_6 and AlSiSr compounds in the bottom of the cast specimen. Stirring method before pouring can improve the grain size and Si morphology.

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