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Melt treatment of Al–Si foundry alloys with B and Sr additions

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ABSTRACT

Individual Sr and B additions to Al–Si alloy melts fail to provide either grain refinement or modification. Sr and B must be used together to achieve both features for high-quality sound castings. Sr and B solutes in addition to insoluble SrB_6 particles are introduced to the melt when Sr and B are used together. SrB_6 particles and B solutes, which form the AlB_2 particles later during solidification, are believed to be responsible for the marked improvement in grain refinement in the co-addition practice. Sr atoms in liquid solution, on the other hand, are adsorbed on the surface of the Si plates leading to twinning once the melt cools to the eutectic solidification temperature. A series of experiments have shown that a very fine grained matrix and a fine fibrous eutectic are readily obtained when the AlSi7Mg0.3 alloy is inoculated with 100 ppm B and 150 ppm Sr.

Introduction

While wrought alloys can be grain refined in the solid state via thermomechanical processing [1–6], foundry alloys must be grain refined during casting. Grain refinement offers uniform structural features, superior mechanical properties, reduced shrinkage as well as better surface quality and is thus a key melt treatment in aluminium foundries [7]. It requires potent substrates in the melt that are readily activated at a low undercooling for sufficiently high nucleation rates. Restriction of the growth process is also needed and is provided by thermal and constitutional undercooling at the interface. The latter increases with increasing solute content as more solute has to be partitioned before the solid–liquid interface can advance.

Titanium nucleates aluminium thanks to a peritectic reaction in the Al–Ti binary system. However, it takes as much as 0.15 wt% Ti to achieve grain refinement via this peritectic reaction. The grain refinement efficiency increases markedly when B is also added to molten aluminium. Al–Ti–B master alloys have thus emerged as potential grain refiners for aluminium alloys. At present, there is a variety of commercial grain refiners of this type, Al–5Ti–1B being the most popular [8–10].

Al–Ti–B alloys offer an outstanding performance in the continuous and semi-continuous casting of wrought alloys but are less effective with aluminium foundry alloys [11]. The latter contain substantial

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Table 1 Chemical composition of the AlSi7Mg0.3 alloy used in the present work (wt%)

Si	Fe	Mn	Mg	Cr	Ti	V	Zr	B	Sr
6.96	0.12	0.02	0.27	0.0003	0.0004	0.0005	0.0003	0.01–0.02	0.01–0.02

levels of Si to improve castability. Si reacts with Ti to form Ti–Si binary phases at >3 wt% and thus reduce the amount and the efficiency of Al_3Ti ve TiB_2 particles, thereby degrading the grain refinement efficiency of the master alloy [12–16]. In contrast to wrought alloys which are grain refined adequately at Ti addition levels of 0.005–0.01 wt%, foundry alloys require at least 10 times more of the same grain refiner. Nevertheless, the commercial practice in shape casting today relies almost entirely on Al–5Ti–1B grain refiners, producing grain structures much coarser than those obtained with wrought alloys.

Al–B alloys have been reported to possess better grain refining efficiency with Al–Si foundry alloys [17–27]. However, measures need to be taken when modification in addition to grain refinement is also to be employed. The former is just as important as the grain refinement and is executed almost invariably through the addition of Al–Sr master alloys. B reacts with Sr to form SrB_6. The interaction of B with Sr is important from both grain refinement as well as modification point of view. Hence, measures are needed to ensure grain refinement and modification when B and Sr are to be employed in the melt treatment of aluminium foundry alloys.

Campbell proposes that the benefits of grain refinement result mainly from the reduction in oxide bifilm content of the melt through sedimentation in the ladle by the action of heavy intermetallics that precipitate on bifilms [28]. This sedimentation is more common in the use of Ti + B grain refinement, where the TiB_2 and $TiAl_3$ particles that form on the bifilms are especially effective in reducing the bifilm content of the melt, resulting in higher properties in the casting. Bifilms in suspension in the melt play a key role also in the modification process [28]. They facilitate the precipitation of Si, and their growth with an extensive planar morphology, leading to the easily nucleated unmodified, faceted form of Si. When Sr is added, it suppresses nucleation of Si on bifilms and hence avoids the formation of large planar Si crystals. A new solidification process then has to take place at a lower temperature, with Si constantly changing its growth direction because of the presence of neighbours leading to a fine, fibrous modified form of Si.

The impact of B and Sr additions on grain refinement and modification has been reported by several investigators [29–34]. The present paper reports the results of additional experiments where B and Sr are added to the popular AlSi7Mg0.3 alloy either individually or together in an effort to identify a sound and cost-effective melt treatment practice for hypoeutectic Al–Si-based foundry alloys.

Experimental

Aluminium ingot with a purity of 99.85 wt% Al and commercial purity Si were melted in an electric resistance furnace and held at 800°C for 1 h to allow homogenisation. The temperature of the Al–Si melt thus obtained was brought to 720°C. Commercial purity Mg addition was made just before pouring to improve recovery. The molten alloy thus obtained was first treated with B to remove transition elements via gravity settlement. This was done by the addition of Al–4B master alloy into the melt, waiting for 2 h to allow for the formation and settlement of borides of particularly Ti, but also of Zr, Cr, V, etc. and then pouring only the top portion of the crucible in a very stagnant fashion. The master AlSi7Mg0.3 alloy thus obtained was checked for its composition with a commercial optical emission spectrometer (Table 1).

The master AlSi7Mg0.3 alloy was used for different experiments where B and Sr additions to the melt were made either individually or together to identify their effects on grain refinement and modification. In each of these experiments, 1000 g AlSi7 Mg alloy was melted in a resistance furnace and the temperature of

Table 2 Experiments performed for the melt treatment of AlSi7Mg0.3 alloy

Experiment #	Melt treatment practice
1	Addition of 200 ppm Sr
2	Addition of 200 ppm B
3	Addition of 100 ppm B
4	Co-addition of 100 ppm B + 100 ppm Sr
5	Co-addition of 100 ppm B + 150 ppm Sr
6	Co-addition of 100 ppm B + 200 ppm Sr

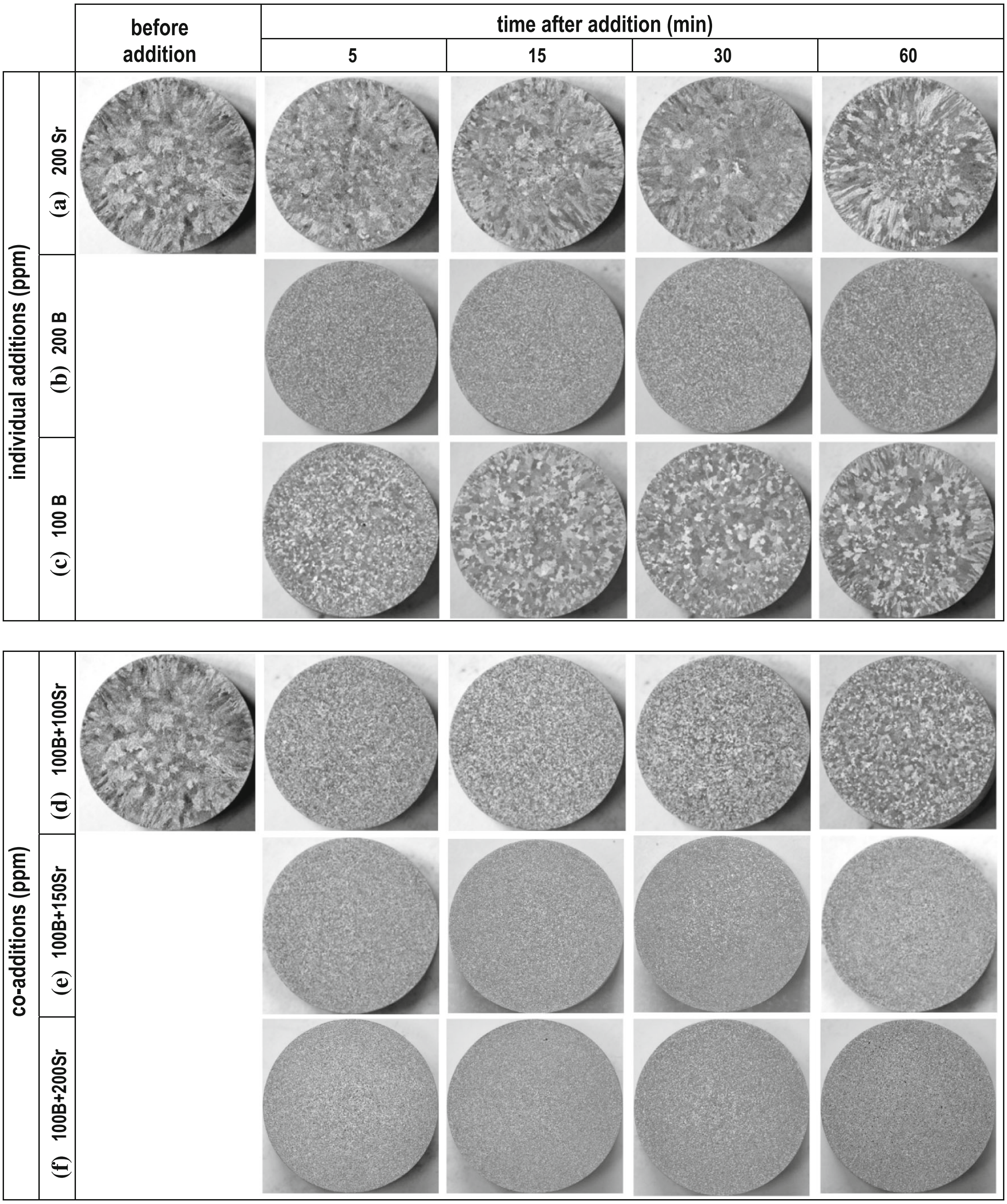

Figure 1 Grain structure before and after individual additions of **a** 200 ppm Sr, **b** 200 ppm B, **c** 100 ppm B and co-addition of **d** 100 ppm B and 100 ppm Sr, **e** 100 ppm B and 150 ppm Sr and **f** 100 ppm B + 200 ppm Sr.

the melt was controlled within 720 ± 10°C. Commercial Al–4B and Al–10Sr master alloys were used to supply 0.01–0.02 wt% (100–200 ppm) B and 0.01–0.02 wt% (100–200 ppm) Sr. A total of six experiments were performed (Table 2). The melt was stirred with a graphite rod for 20 s right after B and Sr additions. The melt was always sampled from the top of the melt until 60 min after B and Sr additions were made. Additional samples were taken from the melt after stirring the melt thoroughly following the 60 min holding step. Samples were solidified in small permanent moulds with a diameter of 25 mm and a height of 50 mm. The samples thus produced were sectioned 20 mm from the bottom surface. Standard

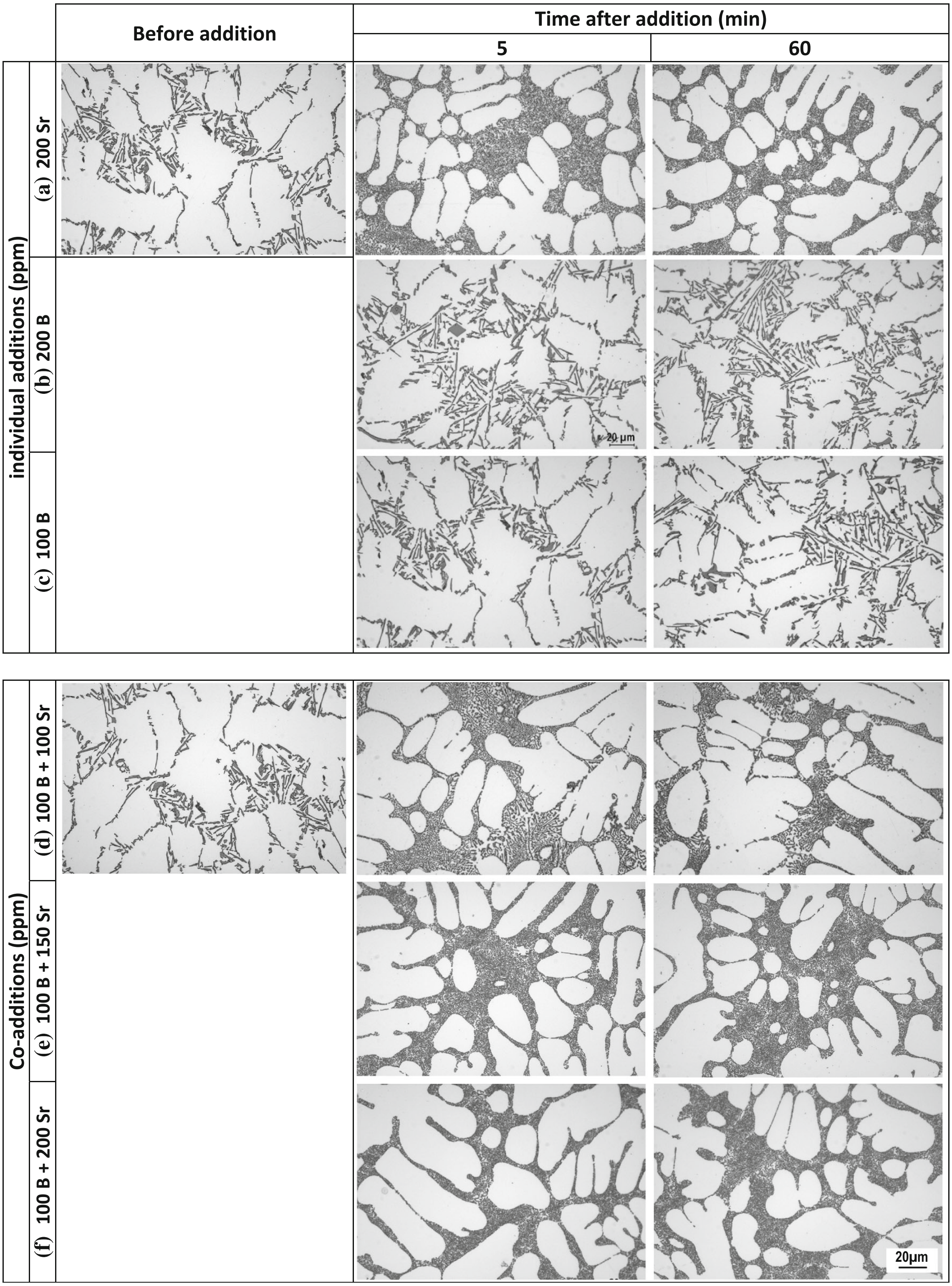

Figure 2 Microstructure before addition and after individual additions of **a** 200 ppm Sr, **b** 200 ppm B, **c** 100 ppm B and co-additions of **d** 100 ppm B and 100 ppm Sr, **e** 100 ppm B and 150 ppm Sr and **f** 100 ppm B + 200 ppm Sr.

metallographic procedures were employed to prepare these sections for grain size measurements. They were etched with Poulton's reagent and were then examined under a light microscope. Some samples were anodised in Barker's solution, 5 ml HBF_4 (%48) in 200 ml water and were examined under polarised light.

Results and discussion

The first set of experiments, Experiments # 1–3, involves the individual additions of Sr and B elements in accordance with the commercial practice. The grain refinement and modification performance test results with the AlSi7Mg0.3 alloy at a Sr addition level of 200 ppm are illustrated in Figs. 1a and 2a, respectively. It is fair to conclude from Figs. 1a and 2a that, while a perfect modification agent, Sr is not a grain refiner for this alloy. The grain refinement performance offered by the addition of 200 ppm B, on the other hand, is remarkable. Grains, as small as 125 µm, are readily achieved at this addition level, much smaller than those achieved with the conventional Al–Ti–B grain refiners (Fig. 1b). The absence of any fading effect with holding time is a further advantage that is typical of grain refinement with B additions [18]. Grain refinement of Al–Si foundry alloys occurs via heterogeneous nucleation of aluminium on AlB_2 particles that form during cooling of the melt once solidification is underway and are thus not susceptible to gravity settlement. While a perfect grain refiner for AlSi7Mg0.3 alloy, 200 ppm B addition fails to modify the eutectic silicon as inferred from the coarse eutectic Si plates and needles (Fig. 2b).

Experiment # 3 of the first set warns, however, that it takes as much as 200 ppm B to offer an outstanding grain refinement effect with the AlSi7Mg0.3 alloy. The grain refinement effect is impaired when the B addition is reduced to 100 ppm (Fig. 1c). This observation is consistent with the results of a recent study where a minimum of 200 ppm B is claimed to be necessary for B additions to introduce grain refinement efficiency much better than the conventional practice [20]. The modification of the eutectic silicon is almost entirely absent also at B addition rates of 100 ppm (Fig. 2c) as one would expect. The key tasks of B and Sr additions can be readily deduced from the foregoing: B is for grain refinement and is not involved in modification while Sr is for the modification of the eutectic Si particles and does not contribute to grain refinement on its own.

However, the modification of the eutectic phase in Al–Si foundry alloys is just as critical as grain refinement and cannot be ignored. Modification treatment transforms the coarse plate-like eutectic Si into fine fibres, producing a very favourable effect on strength and ductility of the casting since coarse Si plates act as stress raisers and lead to premature failure [35]. It is only natural to expect even superior structural features and mechanical properties when grain refinement and modification are introduced together. It is fair to conclude from Figs. 1a–c and 2a–c that individual Sr and B additions fail to provide either grain refinement or modification, and that Sr and B must be offered together to ensure modification of the eutectic phase as well as the refinement of the cast grains.

The second set of experiments (Experiments # 4–6) involves the co-addition of B and Sr to the same Ti-free AlSi7Mg0.3 alloy stock used in Experiments # 1–3. There are marked changes in the grain refinement efficiency and the modification performance of B and Sr when they are added to the AlSi7Mg0.3 alloy melt together. It is clear from Fig. 2d that the coarse Si plates of Experiment # 3 (only 100 ppm B)

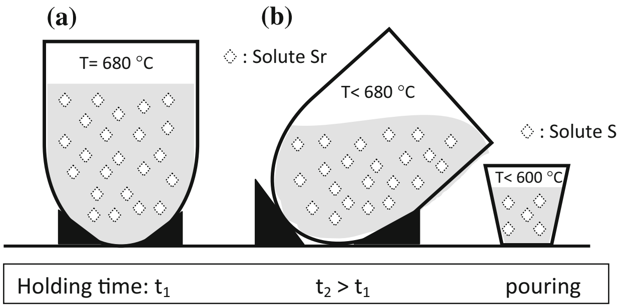

Figure 3 Mechanism involved in the treatment of Al–Si hypoeutectic alloy via the individual addition of Sr, leading only to MODIFICATION. **a** Sr is added individually to the AlSi7Mg0.3 melt. Since Sr is soluble in liquid aluminium at this temperature, entire Sr addition enters liquid solution and is distributed across the melt homogeneously. **b** The melt is poured from the top of the melting pot into the crucible and the temperature of the melt starts to drop. Once the temperature drops to the liquidus line, solute Sr atoms are adsorbed on the faceted surfaces of Si plates and are engaged in the re-entrant twinning mechanism, thereby transforming the coarse Si plates into fine Si fibres. This process offers remarkable modification effect. However, there is no contribution of the Sr in the grain refining process, leading only to modification.

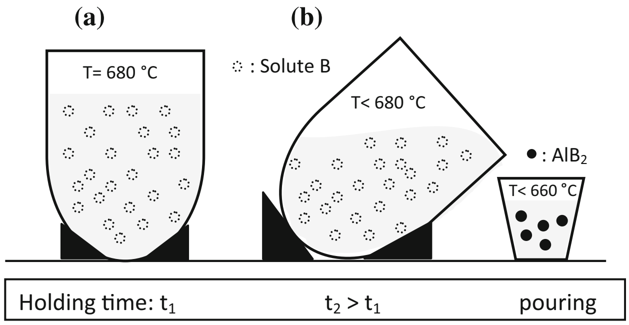

Figure 4 Mechanism involved in the treatment of Al–Si hypoeutectic alloy via the individual addition of B, leading only to GRAIN REFINEMENT. **a** B is added individually to the AlSi7Mg0.3 melt. Since B is soluble in liquid aluminium at this temperature, entire B addition enters liquid solution and is distributed across the melt homogeneously. **b** Since B is in solution, it is not sensitive to gravity settlement and is retained in liquid solution homogeneously throughout the holding time. The melt is poured from the top of the melting pot into the crucible and the temperature of the melt starts to drop. Once the temperature drops to the crystallization temperature of Al boride compound, AlB_2 particles crystallize from the melt. These borides are perfect substrates for the heterogeneous nucleation of primary aluminium, offering remarkable grain refinement, yet no modification.

are entirely replaced by fine Si fibres when 100 ppm Sr was added to the melt together with 100 ppm B. It is thus inferred that there has been adequate modification of the eutectic Silicon phase. However, the underlying issue with Experiment # 4, where B and Sr are co-added to the melt, is the marked improvement in the grain refinement of the matrix phase (Fig. 1d).

The superior grain refinement upon the addition of 100 ppm Sr together with 100 ppm B implies that there ought to be another species other than the soluble AlB_2 particles that facilitate heterogeneous nucleation of the primary aluminium phase. The only borides that can form in aluminium melts upon the addition of Sr and B are SrB_6 and AlB_2 provided that the melt is free of transition metals whose borides are more stable than SrB_6 and AlB_2, such as Ti, V and Zr. SrB_6 forms right after the addition of Sr and B into aluminium melt and is stable. Hence, a portion of the Sr addition reacts with the solute B and forms the SrB_6 compound particles. Since SrB_6 is an insoluble compound, SrB_6 particles are involved in the heterogeneous nucleation process once solidification starts, as long as they are retained in suspension, i.e. the holding time is not too long to allow gravity segregation. The soluble AlB_2 particles that are known to crystallize from the melt only during cooling once the melt temperature drops to around 640°C are also engaged in the heterogeneous nucleation process [36].

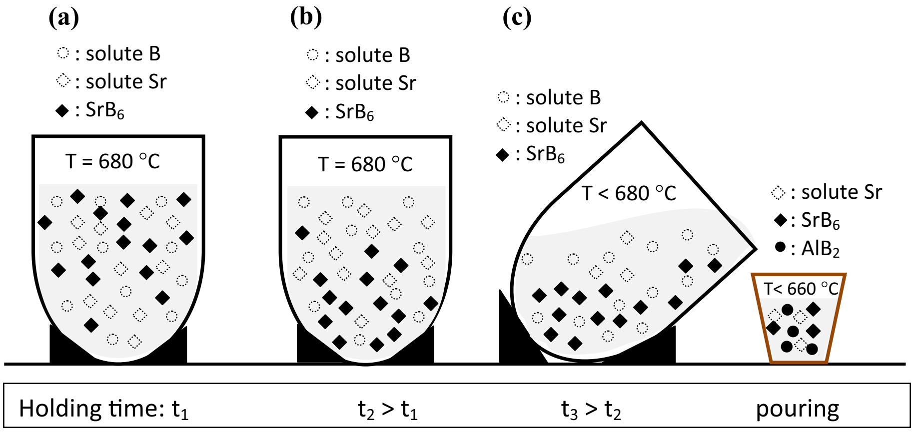

Figure 5 Mechanism involved in the treatment of Al–Si hypoeutectic alloy via the co-addition of B and Sr leading to both MODIFICATION and GRAIN REFINEMENT. **a** Sr and B are co-added to the AlSi7Mg0.3 melt. A fraction of Sr immediately reacts with a fraction of B addition to form SrB_6 particles. Rest of the B addition dissolves in liquid aluminium. Likewise, the unreacted Sr is also in liquid solution. **b** There are three species in the alloy melt at the holding temperature: solute Sr, solute B, insoluble SrB_6. While solute Sr and B are homogeneously dispersed throughout the melt, SrB_6 particles are prone to gravity settlement and are enriched at the bottom of the crucible with time. **c** SrB_6 particles (relatively smaller one that are more resistant to settlement) and solute Sr and solute B atoms are transferred to the crucible. SrB_6 particles are ready to nucleate primary aluminium as soon as the liquidus temperature is reached during the cool down. The solute B, on the other hand, waits until the crystallization temperature of AlB_2 (∼ 640°C) to form in the melt. They also start to nucleate primary aluminium, leading to an enhanced nucleation activity. Solute Sr is engaged in the modification process once the temperature of the melt drops to the liquidus line.

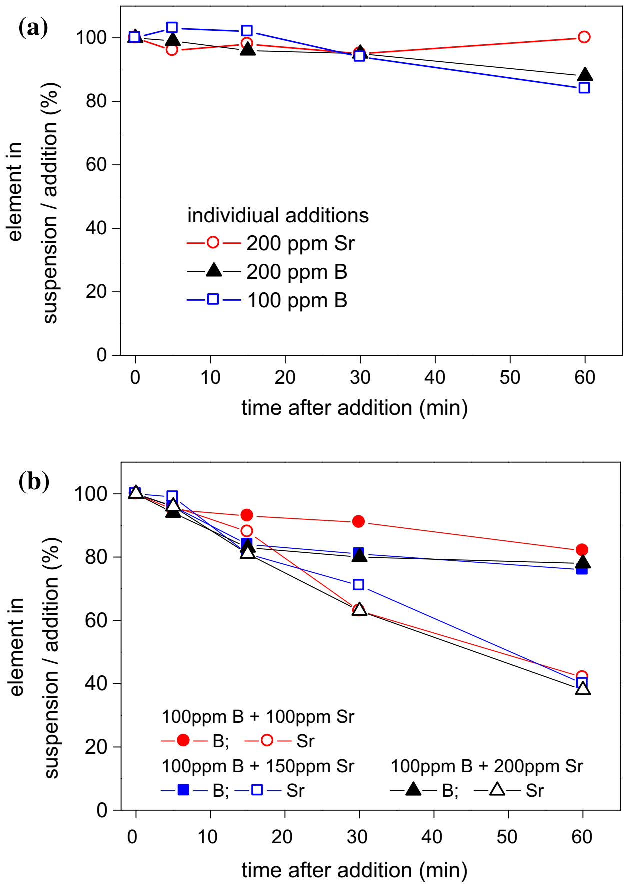

Figure 6 Change in the concentrations of elements in suspension with respect to the addition levels with time after **a** individual and **b** co-additions of Sr and B.

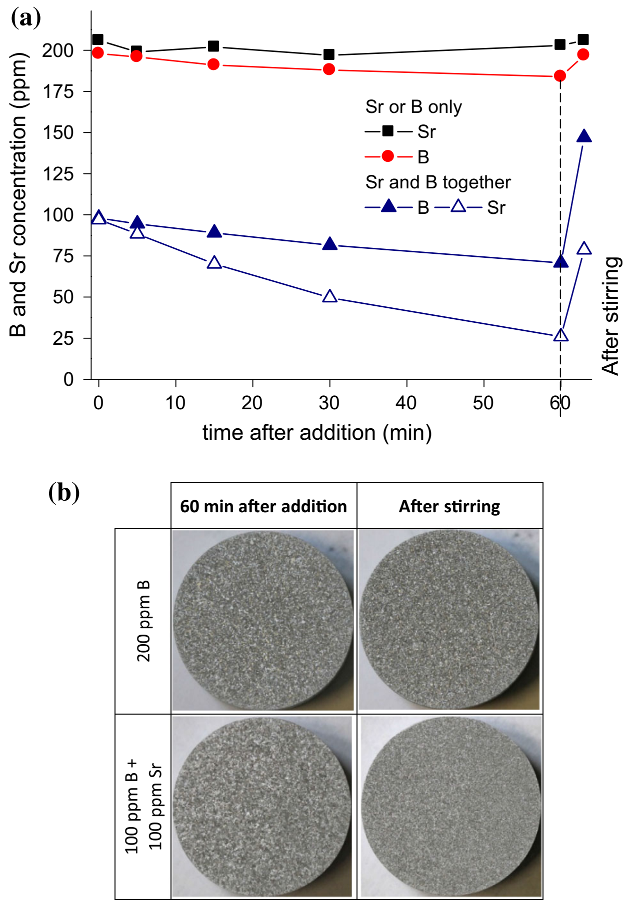

Figure 7 **a** The change in B and Sr concentrations in suspension before and after stirring at the end of 60 minutes **b** grain structures before and after individual additions of 200 ppm and co-addition of 100 ppm B and 100 ppm Sr.

SrB_6 particles are thus believed to be responsible for the marked improvement in grain refinement when Sr is introduced to the melt together with 100 ppm B. With a face-centred cubic structure with a lattice constant of 4.9 Angstrom, SrB_6 is undoubtfully a potent substrate for primary aluminium. Without any Sr, this treatment is a poor grain refinement practice with merely a modest population of AlB_2 particles dictated by 100 ppm B (Experiment # 3). Modification of the eutectic phase in Experiment # 4, in addition to grain refinement, evidences that a portion of the Sr addition was retained in solution. These Sr atoms were then adsorbed on the surface of the Si plates leading to twinning once the temperature dropped to the eutectic solidification line [37].

Both the modification and the grain refinement improve slightly when the Sr addition is increased to 150 ppm (Figs. 1e, 2e). However, increasing the Sr addition further has not produced any change (Figs. 1f, 2f). This is believed to be due to the fact that increasing Sr alone has no effect since the modification was already remarkable at 150 ppm Sr. Any potential improvement in the grain refinement, on the other hand, requires an increase not only in Sr but also in B concentration. It is thus fair to conclude that very small grains and a remarkable modification are readily obtained when the AlSi7Mg0.3 alloy is inoculated with 100 ppm B and 150 ppm Sr. The mechanisms involved in the individual and co-additions of Sr and B are schematically illustrated and are detailed in Figs. 3, 4, 5.

The slight fading of the grain refinement performance after a holding time of 60 min is claimed to be associated with the gravity settlement of SrB_6 particles that exhibit slightly higher densities than liquid aluminium. This fading effect is not as pronounced as that encountered with Al–Ti–B ternary grain refiners since grain refinement in the present case is partially

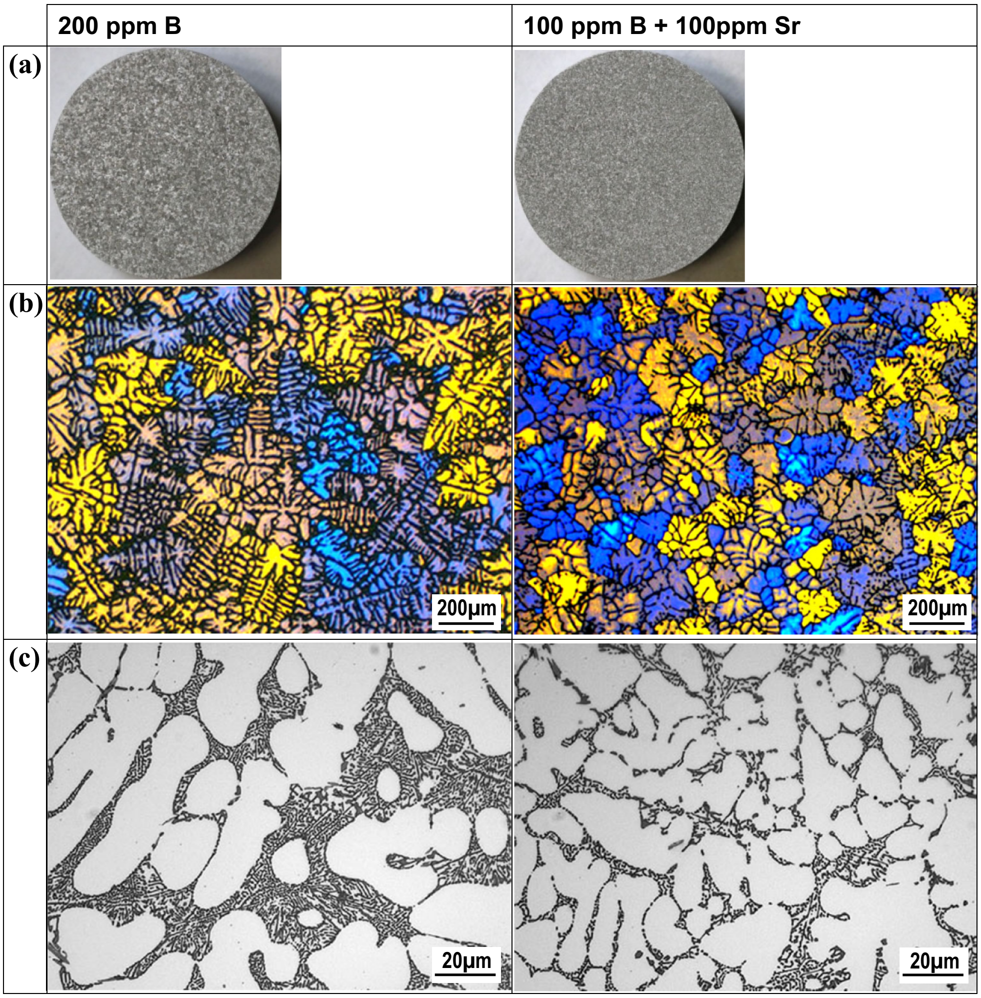

Figure 8 **a** Macro and **b** Barker grain structure and **c** microstructure 60 min after co-addition of 100 ppm B and 150 ppm Sr and after thorough stirring of the melt at the end of 60 min holding time.

provided by AlB_2 particles that are not susceptible to gravity settlement. This account is further supported by the change in B and Sr concentrations in suspension with time (Fig. 6). In the case of individual additions, one can see that there is hardly any reduction in the B and Sr concentrations at the top of the melt, implying that the B and Sr additions are largely retained in suspension without any gravity settlement (Fig. 6a). The slight reduction of B with time is claimed to be due to the transition element impurities which are inevitably present in aluminium melts and hence tie up some B and settle at the bottom of the melt in the form of transition metal borides. The situation is entirely different when B and Sr are added together, instead of individually (Fig. 6b). Both B and Sr concentrations decrease with time evidencing a gravity settlement phenomenon. Several features are worth mentioning. The decrease in Sr in suspension is greater than that of B implying a greater degree of Sr depletion of the top of the crucible with time. While SrB_6 compound contains more Sr than B, the reduction in Sr concentration is even higher than the SrB_6 stoichiometry. Oxidation of Sr may be responsible for at least a portion of the Sr depletion with time. Increasing the Sr addition level does not make a big difference in these concentration profiles. It is thus fair to conclude that the settlement process is controlled by the B concentration.

Stirring of the melt vigorously before casting leads to marked increases in B and Sr concentrations, suggesting that the stirring action puts the settled SrB_6 particles back in suspension (Fig. 7a). In the meantime, the slight loss of grain refinement after a holding time of 60 min is restored (Fig. 7b). This confirms the key role of the SrB_6 particles in the grain refinement of the cast structure. In spite of a very favourable effect on the grain size, this increase in Sr and B concentrations upon stirring of the melt fails to improve the modification of the eutectic silicon. It is the solute Sr, not the SrB_6 particles, that is required for the modification of the eutectic phase. On the other hand, Sr that is put back into suspension in the melt upon stirring is entirely in the form of SrB_6 particles and thus is of no use when it comes to the modification process. This is elegantly demonstrated with a series of grain structure macrographs and optical micrographs taken from AlSi7Mg0.3 samples cast into permanent moulds after a holding time of 60 min before and after stirring of the melt (Fig. 8). The stirring action has produced a remarkable improvement in grain refinement (Fig. 8 a, b) yet failed to increase the modification rating (Fig. 8c). It is thus fair to conclude that the particles that are rejuvenated with this stirring action are all of the SrB_6 variety. Hence, SrB_6 particles are credited for the improvement in grain refinement efficiency.

Conclusions

Individual Sr additions introduce to the melt solute Sr that offers only modification. When B is added in the same manner, there is only solute B in the melt, which forms AlB_2 particles once solidification starts and offers only grain refinement. Sr and B solutes, in addition to SrB_6 particles, are introduced when B and Sr are added to the melt together. B in solution once again forms AlB_2 particles once solidification is underway. Hence, AlB_2 particles are activated later while SrB_6 particles are engaged in the heterogeneous nucleation process from the onset of solidification, leading to an enhanced nucleation activity. Solute Sr, on the other hand, is involved in the modification of eutectic phase. In summary, either modification of the eutectic phase or refinement of the cast grains is achieved in the individual additions of Sr and B, respectively, while both modification and grain refinement are encountered when they are added together. A series of experiments have shown that a very fine grained matrix and a fine fibrous eutectic are readily obtained when the AlSi7Mg0.3 alloy is inoculated with 100 ppm B and 150 ppm Sr.

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Compliance with ethical standards

Conflict of interest The author declares that he has no conflict of interest.

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