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TECHNICAL ARTICLE

Metallothermic Production of Aluminum–Strontium Master Alloy for Modification of Silicon

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Abstract

Strontium master alloy is widely used in today's aluminum foundries for modification of Al–Si alloys. In this study, Al–Sr master alloy was produced in molten Al alloy directly from strontium carbonate instead of using the pure strontium metal which is difficult to store in the air due to its high oxygen affinity. In a laboratory-scale induction furnace, $SrCO_3$ and SrO powders were injected into the liquid metal via argon flow and subjected to reduction reaction. The oxygen-free medium required for the reduction was provided by the gas purged on both the surface and the inside of the liquid metal. Hence, the master alloy was produced directly by an economical process. Moreover, the addition of Mg to the reducing melt decreased the surface tension of the liquid aluminum; as a result, the reduction efficiency was increased. At the best of the results, the master alloy containing 5.15% Sr was obtained by reducing approximately half of the fed SrO by Al6Mg. This master alloy was used to modify A360 alloy, and the same modification effect as the commercial Al5Sr master alloy was observed.

Keywords Al–Sr master alloy · Silicon modification · Metallothermic reduction · Aluminothermy

Introduction

Aluminum–silicon alloys are the most widely used of cast aluminum alloys due to their excellent castability and superior mechanical properties [1, 2]. However, the platelet structure of silicon in the unmodified alloys causes the toughness of the casting alloy to be reduced. As has long been known, this problem can be overcome by modification techniques. Modification improves the mechanical properties of the cast part by transforming large-plate Si phases into fine fibers all of Al–Si alloys [3]. The modification can either be performed by rapid solidification [2] or by the addition of modifiers such as strontium, sodium, antimony, and bismuth [4–8].

Typically, 100–200 ppm Sr in cast alloy is sufficient to transform coarse and sharp-angled Si plates to a fine fibrous form [9]. Formerly, high strontium-containing Al–Sr master alloys (> 15 wt.%) were used for the modification and Sr–Si–Al master alloys as well. However, the use of these alloys has decreased today due to the slow dissolution of intermetallics such as Al_xSr, Al_2Si_2Sr, or $SrSi_2$ at higher melting point (~750 °C) [10, 11]. Nowadays, Al–Sr master alloys containing less than 10% Sr are widely used.

Today's foundry applications involve the use of Al–Sr master alloy which is produced by remelting of pure strontium and aluminum metals. The pure Sr metal is produced by molten salt electrolysis or vacuum metallothermic reduction [12–16]. Pure strontium has a very high O_2 and H_2 affinities. Accordingly, while Sr is added to the molten metal, some of it is lost due to oxidation. Also, the higher the melt temperature causes, the higher the amount of Sr lost [10]. Nevertheless, the alloys containing less than 63% Sr have high corrosion resistance since a protective oxide layer is formed on the surface [11].

Heretofore, different methods have been tried for the production of master alloys in order to eliminate the difficulties in pure strontium production. Al–Sr master alloy was produced by electrolysis of molten salt, NaCl–KCl–$SrCl_2$ [3] or $SrCl_2$–KCl [8], using pure Al or Al alloy as the cathode. Moreover, Mg–Sr master alloy could be produced by reducing Mg and Sr together from $MgCl_2$–$SrCl_2$–KCl salt mixture [17].

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In recent years, some studies have been tried to produce master alloy directly by metallothermic reduction. In the oldest study, Langlais and Harris [18] reduced $SrCO_3$ in liquid aluminum at 1000 °C to produce a master alloy containing approximately 2% Sr. Muñiz et al. [19] were able to increase the Sr content of the alloy up to 5% by injecting SrO into 15 kg of Al alloy containing 1–2.3% Mg at 750–850 °C. With a similar system, Molina et al. [20] modified the Al–Si alloys (A339, A380) containing 0.1–1.2% Mg by SrO injection. Al and Mg reduced SrO, then Sr dissolved in liquid metal modified the silicon platelets. Oxide phases, such as MgO, Al_2O_3, $MgAl_2O_4$, and $SrAl_2O_4$, which are the product of metallothermic reduction reaction, are observed in the structure. Juarez et al. [21] injected SrO into Mg-containing Al scrap at temperatures between 750 and 850 °C to obtain an Al–Mg–Sr alloy with a Sr content of 7.2%. A significant amount of Mg remained in the Al–Sr alloys obtained in these studies.

In this study, an agile and cost-effective method has been tried without the need of production of pure strontium metal. Modifier Al–Sr master alloy was produced directly from SrO and $SrCO_3$ in aluminum melt. This method will be a practical and economical alternative to laborious and expensive processes such as molten salt electrolysis [3, 8, 17] and vacuum reduction–condensation [12–16]. Instead of using fancy atmospheric controlled systems for metallothermic reaction to lower the partial pressure of oxygen, it was sufficient to purge Ar gas into and onto liquid metal under atmospheric conditions. So, the requirement of extra protection to prevent oxidation was eliminated. Ultimately, the obtained master alloys were tested for modification of a conventional Al–Si alloy and the efficiency of the modifier was compared to the commercial Al–Sr master alloy.

Experimental Study

Materials

Commercially pure aluminum (CP Al) ingot and Al–6Mg alloy were used as a reductant for the metallothermic reaction. Al–6Mg alloy was prepared by adding the calculated amount of commercially pure Mg ingot to CP Al. For the modification experiments, A360 ingot was used. Chemical compositions of the ingots were recorded by optic emission spectroscopy (Table 1). The strontium sources, strontium carbonate ($SrCO_3$) and strontium oxide (SrO) powders, were synthesized in previous studies. $SrCO_3$ was obtained from celestite ore according to the black ash method [22, 23]. SrO powders were obtained in the previous study [24] by calcination of $SrCO_3$ in rotary kiln according to Reaction (1).

$$SrCO_3 = SrO + CO_2. \quad (1)$$

Metallothermic Reduction

All the metallothermic reduction experiments were run in a medium frequency induction furnace. At every experiment, 500 g of the pieces cut from the reductants (CP Al or Al6Mg) was melted in the graphite crucible, and then, the melt temperature was adjusted to 1200 °C (± 50 °C). Subsequently, the mixer (graphite impeller) was placed in the liquid metal. Following the degassing with argon (6N), the slag was removed, and the lid of the pot was closed.

The strontium powders were fed by purging argon flow through the inner way of the mixer (Fig. 1). The amount of powder to be fed was determined by calculating the weight of $SrCO_3$ and SrO required for 500 g of aluminum, including 5 or 10% Sr by weight. During the reduction process, the argon gas was purged toward the melt surface (Table 2).

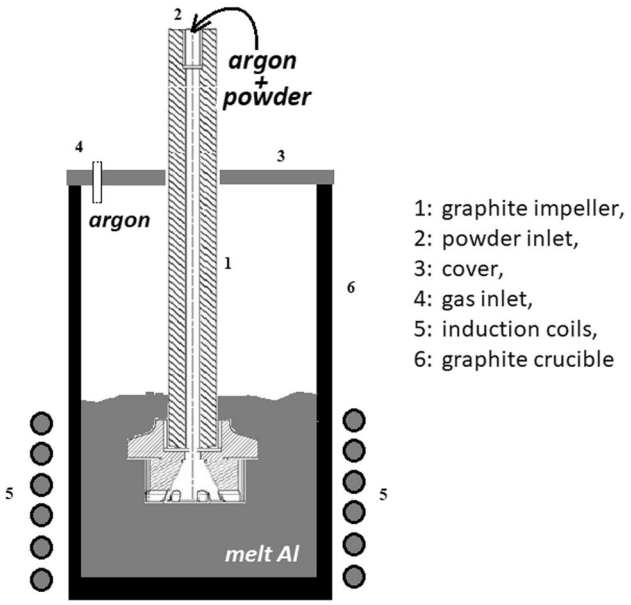


Fig. 1 Experimental setup for metallothermic reduction

Table 1 Chemical composition of the ingots

Ingot	Fe, %	Si, %	Cu, %	Mg, %	Mn, %	Zn, %	Ti, %	Sr, %	Al, %
CP Al	0.225	0.074	0.073	0.033	0.004	0.004	0.018	0.000	99.536
Al6Mg	0.269	0.107	0.029	6.161	0.017	0.015	0.015	0.000	93.375
A360	0.145	8.881	0.005	0.361	0.475	0.007	0.004	0.000	90.151

Table 2 Experiment codes of the studies

Code of Exp.	Powder mass, g	Sr source	Ingot
AC1	43	$SrCO_3$	CP Al
AC2	86	$SrCO_3$	CP Al
AO1	30	SrO	CP Al
AO2	60	SrO	CP Al
MC1	43	$SrCO_3$	Al6Mg
MC2	86	$SrCO_3$	Al6Mg
MO1	30	SrO	Al6Mg
MO2	60	SrO	Al6Mg

In the system, the feed time of the powder at an Ar-gas flow rate of 1.5 L/minute varies between 4 and 10 min depending on the material proportion. After a total of 60 minutes of the experiment, the slag and dross on the surface of the liquid metal were skimmed. It was then poured into a steel mold at room temperature, without waiting for the alloy to cool.

The structural analysis of master alloys was performed with Electron Probe Micro Analyzer (EPMA, CAMECA), Energy-dispersive X-ray spectroscopy (EDS, Bruker), and X-ray diffraction (XRD, Panalytical). Alloys were dissolved, and strontium substances were determined by atomic absorption spectroscopy (AAS, PerkinElmer).

Modification Test

The most current use of Al–Sr master alloys is modifying the Al–Si alloys. The efficiency of the modification performance of the obtained master alloys was evaluated and compared by metallographic examinations with the commercial Al5Sr. Modification experiments were run in an electric resistance furnace. Two grams of the sample from each master alloy was added to 200 g A360 alloy melted in a graphite crucible at 725 °C. After holding of 15 min to completely dissolve the master alloys, the alloys were poured into sand molds. The microstructural investigations were performed with an optical microscope.

Results and Discussion

In Al–Sr binary alloys, the structure is generally composed of α-Al or α-Sr and Al_xSr intermetallics in varying compositions (Fig. 2). The solubility of the strontium in α-Al is less than 54 ppm [25]. Besides the α phase, various intermetallics (Al_4Sr, Al_2Sr, and Al_7Sr_8) are observed in both aluminum-rich and strontium-rich alloys [26]. Al–Sr master alloys commonly used in the industry are limited to a maximum of 20 wt.% of the strontium ratio, so only Al_4Sr

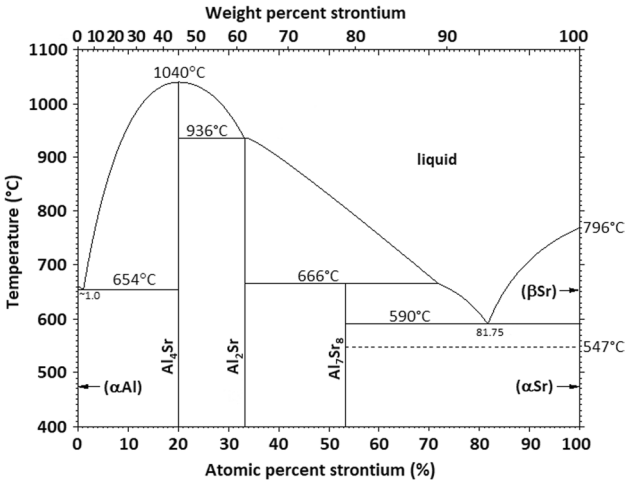


Fig. 2 Binary diagram of Al–Sr system [27]

intermetallic phase is seen in the alloys ordinarily. The Al_4Sr phase containing 55 wt.% of Al and 45 wt.% Sr which of melting temperature is 1040 °C. In the phase diagram, the eutectic (~3.2 wt.% Sr) conversion in the aluminum-rich fraction is seen at a temperature of 654 °C [27]. Therefore, the melting temperature of the alloys is between 654 and 1040 °C depending on the Sr ratio [13].

Metallothermic Reduction

Strontium is a metal with high oxygen affinity. In order to reduce the formation of strontium oxide, a condition where the oxygen affinity of aluminum is higher than that of strontium needs to be established. The Gibbs energies of the aluminothermic reaction of SrO reaction (1) calculated with FactSage Thermochemical Software [28] at different pressures and temperatures are given in Fig. 3. In atmospheric conditions (~1 bar), it is compulsory to reach temperatures above 2000 °C in order to be able to reduce SrO with Al. However, the Gibbs energy of reaction (2) can be reduced to negative values at lower temperatures. The reaction Gibbs energy can be reduced to negative values at temperatures of 1530, 1223, and 1012 °C (and above) at pressures of 10^{-1}, 10^{-2}, and 10^{-3} bar, respectively.

$$3SrO + 2Al = 3Sr + Al_2O_3. \quad (2)$$

As the pressure decreases, the boiling temperature of Sr metal is reduced. Thus, under suitable conditions of temperature and pressure where the reduction is possible, the reaction product Sr can be obtained in the gas phase. In order to prevent the strontium evaporation, metallothermic reduction studies were performed under atmospheric pressure. During the experiment, Ar gas was purged to the crucible surface in order

to reduce the oxygen partial pressure. Hence, proper conditions were provided for aluminothermic reduction while preventing the evaporation of pure strontium. The aluminothermic reduction reaction (2) is the sum of the oxidation reaction of aluminum (3) and the dissociation reaction of the strontium oxide (4). In both reactions, because the only gas component is oxygen, the reaction Gibbs energy is only dependent on the oxygen partial (P_{O2}) pressure. So, oxygen partial pressure can be taken into account for the pressure values in the graph in Fig. 3. At the temperature of the experiments (1100 °C), a pressure value needs to be lower than $P_{O2}=10^{-3}$ bar, which was fairly provided by 6N Ar. Therefore, the metallothermic reduction is possible in conditions where P_{O2} was sufficiently low regardless of the ambient pressure.

$$2Al + {}^{3}/_{2}O_2 = Al_2O_3 \quad (3)$$

$$3SrO = 3Sr + {}^{3}/_{2}O_2. \quad (4)$$

The surface tension of pure aluminum is relatively high due to the oxide forming on the surface. However, the addition of various alloying elements, such as magnesium, can reduce surface tension [29, 30]. For this reason, some of the reduction experiments were run with Al6Mg alloy to decrease the effect of surface tension of the liquid metal. Besides reducing the surface tension of the liquid metal, Mg served as a reductant due to the higher oxygen interest of it [18, 31]. Mg in the melt contributed to reducing SrO to provide a metallic Sr reaction (5).

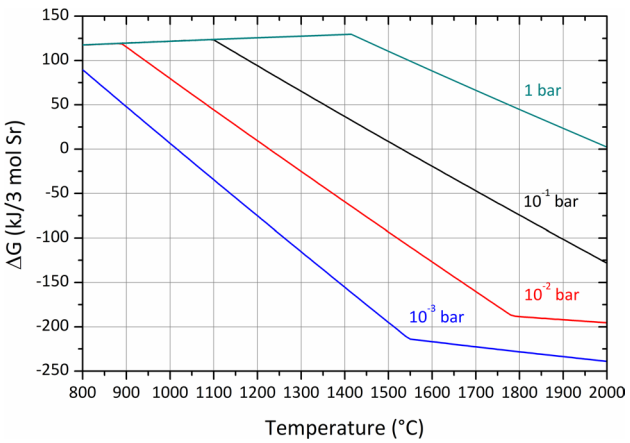


Fig. 3 Gibbs energies of aluminothermic reaction of strontium oxide at different pressures

$$SrO + Mg = Sr + MgO. \quad (5)$$

The obtained master alloys were analyzed by wet chemical technique (AAS), and their Sr contents were calculated (Table 3). When SrO was used as Sr source (experiments coded as *O*), a higher reduction yield was achieved compared to when $SrCO_3$ was used. The carbonate would become the first to be calcined to oxide and then be reduced by Al. As the calcination of $SrCO_3$ in the liquid metal took a long time, the reduction was slower. Furthermore, it was observed that the reduction efficiency increased when more (2 times) powder was fed because the plentiful material in liquid aluminum enlarged the reaction surface.

In the experiments with CP Al (coded as A**), the reduction efficiencies were found to be quite low. Due to the high surface tension of the pure aluminum melt, the powders could not be wetted by Al. Therefore, the metallothermic reduction reaction took place in a scarce area. Even stirring at high speed could hardly help reduce surface tension. The use of Al6Mg as the reducing agent overwhelmed the surface tension and enhanced reaction efficiency. In particular, the master alloy coded as MO2 approached the target Sr ratio (5% by weight).

The obtained master alloys were microstructurally evaluated with EPMA and EDS (Fig. 4). Some shrinkage and cracks have been observed in the microstructure because of the high-temperature casting. The non-reduced oxide phases were clustered in the structure, especially around the cracks. The aluminothermic reaction product Sr was dissolved in the melt, but aluminum oxide reacted with the non-reduced SrO to form a Sr-spinel ($SrAl_2O_4$) (Fig. 4a). Since Mg served as a reducer in Al6Mg melt, the reduction product MgO is also reacted with Al_2O_3 to form Mg-spinels ($MgAl_2O_4$). Mg-spinels and Sr-spinels appeared together in the microstructure as small light-gray phases (Fig. 4b,c); The Mg, Sr, Al, and O patterns were proof of these in the EDS analysis. On the other hand, it could be said that the whole of $SrCO_3$ was calcined until the end of the process through there was no carbonate phase in the system because the temperature was higher than the calcination temperature (Fig. 4b).

It is important to note that in the presence of Sr and/or Mg, the surface oxide of the liquid aluminum alloy begins to form spinel oxides. Thus, two events occur. Firstly, the surface tension of the fluid decreases, and secondly, the fracture is observed as reported by Bainbridge [32]. This is also known as breakaway oxidation. It means that Mg in the melt reduced the surface tension, and allowed SrO and Al to react genially. Then, $SrAl_2O_4$ and $MgAl_2O_4$ formed as a result of the

Table 3 Strontium amounts and the reduction efficiency of the obtained master alloys

Master alloy code	AC1	AC2	AO1	AO2	MC1	MC2	MO1	MO2
Sr in the alloy, %	0.01	0.03	0.49	1.06	0.87	1.45	1.98	5.15
Reduction yield, %	0.2	0.3	9.8	10.6	17.4	14.5	39.6	51.5

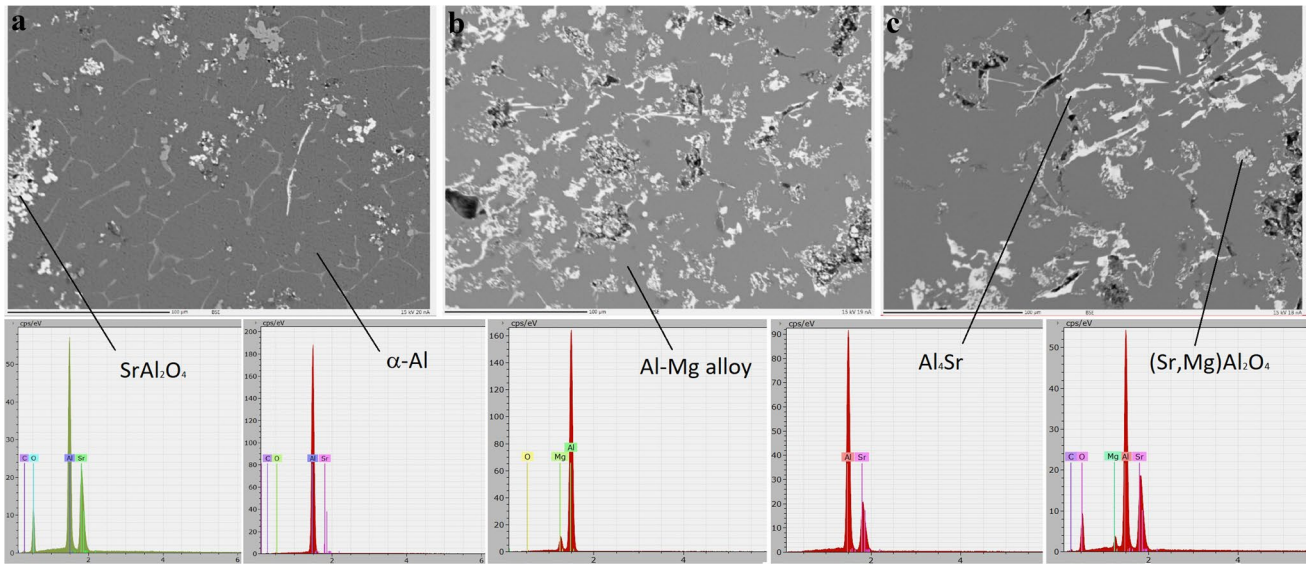


Fig. 4 Backscattered images of (**a**) AO1, (**b**) MC2, (**c**) MO2, and EDS analyses of selected points

combination of the reaction products. The spinels decreased the surface tension and also broke the oxide layer of aluminum. So, the reaction was accelerated, and the efficiency increased exponentially.

Depending on the reduction efficiency, Al_4Sr intermetallic phases are observed with the characteristic shapes of rectangular stripe and plates. Because of a low amount of Sr, e.g., AO1 coded alloy, a minimal amount of slender Al_4Sr phases were identified (Fig. 4a). As the reduction efficiency raised, the size and amount of intermetallics in the structure heightened. In particular, in MO2, the intermetallics are heavily dispersed in the structure (Fig. 4c).

As the master alloys poured directly from 1200 °C after the metallothermic reduction process, the cooling rate was slightly higher. The rapid cooling made it difficult for the reduced Sr to precipitate in the form of intermetallics. Thus, a metastable α-Al solid solution contained more Sr than its solubility [33], and the Al_4Sr intermetallics were fairly thin [34]. Since the Al_4Sr intermetallics are quite sparse and thin (Fig. 4) and the XRD patterns appear weak (Fig. 5). Besides, the peaks of oxides (mostly SrO and rarely spinels) remaining in the master alloys were marked with ϕ symbol. On the other hand, in alloys prepared with Al6Mg (MO2 and MC2), Mg dissolved in the primary phase slightly shifted the peaks of the α-Al phase to the left.

Modification Performance

To measure the modification performance of the obtained master alloys, they were added to A360 alloy in a fixed amount and compared to the commercial Al5Sr master alloy. After the modification experiments, the amount of

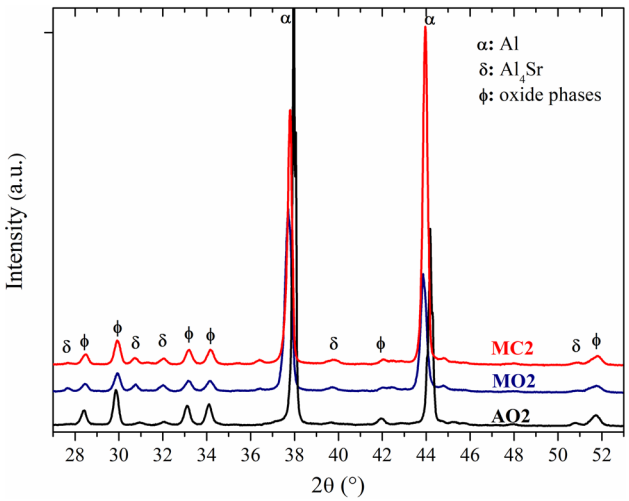


Fig. 5 XRD patterns of master alloys

Sr in A360 was determined by optical emission spectrometry (Fig. 6). Furthermore, the effect of master alloys on the modification of A360 alloy was examined microstructurally by EPMA (Fig. 7).

In the structure of the unmodified A360 alloy, coarse silicon platelets were duly observed because there was no Sr content to cause any modification (Fig. 7a). In the alloy modified with the commercial Al5Sr alloy, 498 ppm Sr was determined after the casting. The eutectic and homogeneous dendritic structure was formed with the distributed fibrous silicon (Fig. 7b). Master alloy AC1 showed no modification effect on A360 alloys based on the fact that Sr content was 4ppm. Si platelets on the alloys are still coarse. The

microstructures are similar to the original (unmodified) of A360 (Fig. 7c). Master alloy AO2 provided partial modification ability. The amount of Sr in the alloy (51 ppm) did not show a homogenous modification effect. There are still coarse Si grains between the arms of the secondary dendrites (Fig. 7d). The modification work was initiated with master alloy MC2 by providing 150 ppm Sr in the cast. However, in the unmodified areas in the arms of the dendrites, there are Si platelets. Modification had started in the structure, but it was not homogenous. The size of Si in the structure is different (Fig. 7e). The master alloy coded as MO2 yielded approximately the same value of Sr ratio with a similar effect to the commercial Al5Sr. In the metallographic examinations, it is seen that Si in the alloy was in a thinner fiber structure than the former cases. Thin and fibrous Si was eutectic phase. It was seen that the eutectic structure is distributed homogeneously with a continuous network between the dendrites (Fig. 7f).

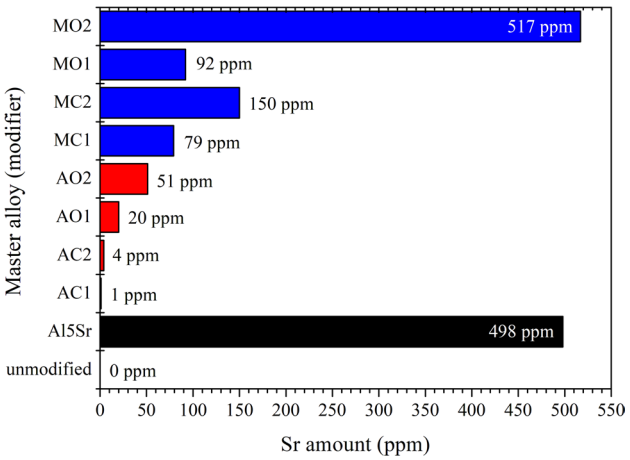


Fig. 6 Sr contents of the cast A360 after modification with master alloys

As a result of the modifications experiments, when the amount of Sr in A360 alloys was above 50 ppm, the modification effects were observed in varying proportions. Zarif [35] had claimed that 50 ppm was fairly enough to modify Al–Si alloys Nevertheless, the results of this work showed that 50 ppm Sr is only enough to start the modification. Thereof, in cast houses, a minimum of 200 ppm is selected for optimum modification [36].

Conclusion

Al–Sr master alloy was successfully produced by metallothermic reactions using liquid aluminum. The low partial pressure of oxygen, which required for aluminothermic reduction of SrO, was provided by purging of Argon onto and into the liquid metal. $SrCO_3$ was chosen as the starting material; however, it was found that the efficiency was quite low compare to the use of SrO. Accordingly, it is crucial to calcinate $SrCO_3$ initially. The decrease in surface tension in the presence of Mg was found to be the critical factor for the production of Al–Sr master alloy. Initially, Mg decreased the

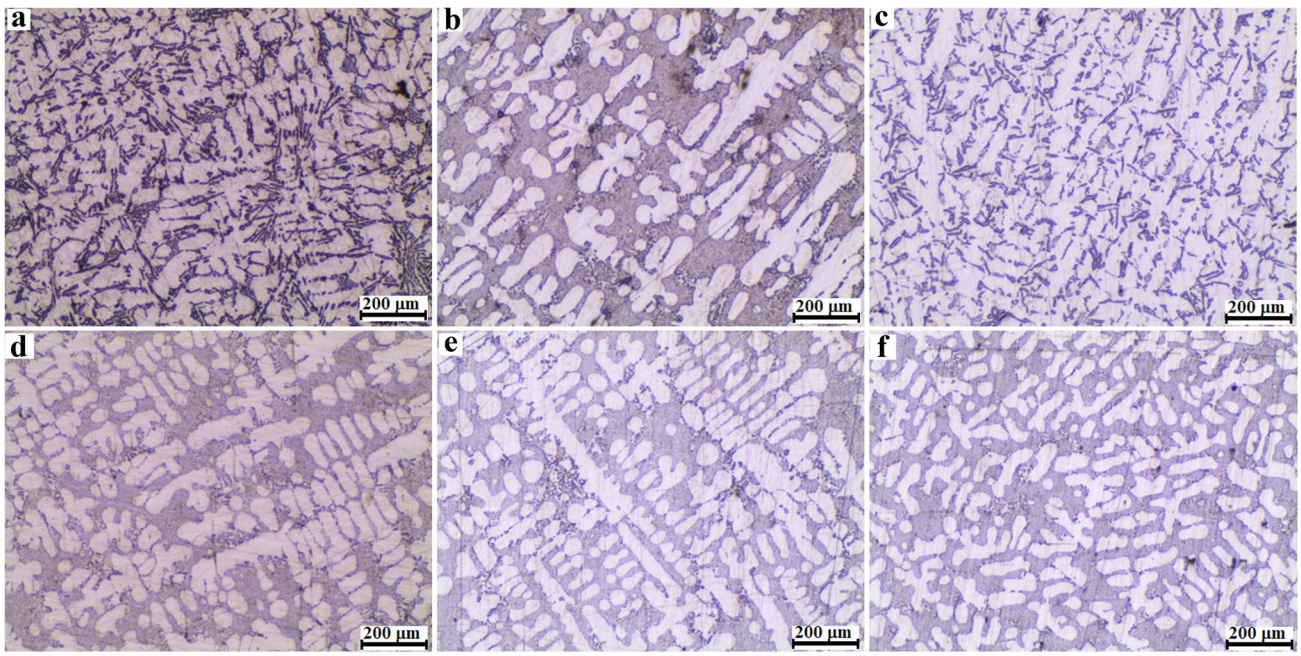


Fig. 7 Microstructural images of A360 alloy; (**a**) unmodified, (**b**) modified with commercial Al5Sr and obtained master alloys such as (**c**) AC2, (**d**) AO2, (**e**) MC2, and (**f**) MO2

surface tension of the melt and facilitated the reaction of Al and SrO, and then $MgAl_2O_4$ spinels formed that afforded the breaking of the oxide film on the liquid metal surface. The low reduction efficiency at the start of the reaction was increased exponentially. The maximum yield was achieved when Mg-containing alloy was employed with a value of 51%. Furthermore, the modification exercising this alloy (MO2) performed equally well with the commercial Al5Sr. On the other hand, Sr content over 50 ppm in Al–Si alloy permits the modification to start, but more Sr is obliged for the full modification.

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Availability of Data and Materials Data will be made available on request.

Compliance with Ethical Standards

Conflict of interest The authors declare that they have no conflict of interests.

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