Effects of modifying the hypoeutectic AlMg5Si2Mn alloy via addition of Al10Sr and/or Al5TiB

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Abstract

This work demonstrates that the combined addition of Al10Sr and Al5TiB master alloys to the AlMg5Si2Mn effectively refines the grain microstructure and partially modifies the eutectic Mg₂Si phase. Thorough spectroscopic characterization reveals that the grain refinement effect is due to Al₃Ti particles acting as nucleation sites for α -Al grains, and the increased nucleation temperature of α -Al is due to Al10Sr addition. It is also determined that TiB₂ particles can act as nucleation substrates for the primary Mg₂Si phase. The prepared alloy sample with the finest microstructure (treated with both Al10Sr and Al5TiB) exhibits the greatest corrosion resistance among all tested samples.

Keywords AlMg5Si2Mn · Modification · Grain refinement · Al5TiB · Al10Sr · Nucleation

1 Introduction

AlMg5Si2Mn is a non-heat-treatable 5xxx series aluminum foundry alloy that contains Mg and Si as major alloying elements. This alloy exhibits good corrosion resistance, hardness, and thermal conductivity, so it is typically utilized as a lightweight material for structural components in the automotive and aerospace industry [1–3]. The microstructure of Al–Mg–Si cast alloys, including AlMg5Si2Mn, consist of α -Al dendrites, primary Mg₂Si particles, and/or brittle α -Al + Mg₂Si “Chinese script” eutectic structures [4]. The mechanical properties of the AlMg5Si2Mn base alloy can be optimized by modifying the microstructure of these various components. Therefore, an important approach to enhancing the functionality of these alloys involves controlling the

grain size and tuning the morphology of primary and eutectic Mg₂Si phases [5].

Recently, many investigations have been conducted to refine the microstructure and ultimately improve the mechanical properties of Al–Mg–Si alloys or Al–Mg₂Si composites by applying methods such as rapid cooling [6], melt superheating [7], hot extrusion [8], pre-homogenization deformation treatment [9], or chemical modification [10]. Among these strategies, chemical modification is a simple and cost-effective approach to microstructure enhancement and Mg₂Si particle refinement. To date, many reports have described potential methods to obtain effective chemical modification of primary Mg₂Si and/or eutectic Mg₂Si by adding grain modifiers, such as Cr [11], Li [12], Cu [13], Sb [14], Na [15], Sr [16], or TiB [17]. Modification with sodium leads to rapid fading and formation of micro-pores. Although the modification effects of Sb are longer-lasting, modification with this element typically yields inferior results to that of Na or Sr. In fact, Sr and TiB enable very high overall performance and exhibit relatively favorable modification effects. Therefore, they have been widely employed for refining the microstructure because of their modification efficiency and long effective modification period. Qin et al. [18] studied the effect of Sr modification on the size and morphology of Mg₂Si crystals. They found that increasing the Sr content changed the morphology of Mg₂Si phases from dendritic to polygonal, and the particle size decreased from ~200 to ~40 μ m. Additionally, Tebib

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et al. [19] reported that simultaneous refinement of primary and eutectic Mg_2Si crystals could be achieved via modification with strontium. Other researchers [20] reported that the addition of Al10Sr master alloy modifies the eutectic Mg_2Si from a Chinese script shape to a polyhedral or fine fiber shape, and decreases the average particle size of the primary Mg_2Si phase. The Al–5Ti–1B master alloy is a very useful grain refiner for pure commercial aluminum and wrought aluminum alloys [21, 22]. Studies have shown that the TiB_2 and Al_3Ti particles present in Al–Ti–B master alloys can act as nucleation sites for primary aluminum dendrites [23, 24]. It has also been determined that TiB_2 particles may act as nuclei of the primary Mg_2Si phase; therefore, TiB_2 particles can be used to refine and modify the primary Mg_2Si [17]. In combination with TiB, Sr addition can promote the growth of columnar dendrites, thus considerably increasing the amount of dendritic α phase. Therefore, it is expected that refinement and modification employed together will improve the microstructure and mechanical properties even further. However, this may involve some complications because boron can react with strontium, leading to reduced modification effects [25].

To our knowledge, there have not yet been any studies regarding the effects of simultaneous modification by incorporating Sr and TiB in the AlMg5Si2Mn alloy. Taking into account the effectiveness of both additives, the present work describes our investigations into the effect of modification by Al10Sr and/or Al5TiB addition, and our evaluations of the interactions between the Al10Sr/Al5TiB and a commercial hypoeutectic AlMg5Si2Mn alloy.

2 Materials and methods

The AlMg5Si2Mn alloy (EN AC 51500) was used as a base material, and its chemical composition was measured using a SPECTROMAXx Arc/Spark OES analyzer (results provided in Table 1). The Al5TiB and Al10Sr master alloys were used for grain refinement and eutectic modification.

The base alloy was melted using an electric induction furnace with a total power of 7.5 kW. The modifying additives were introduced separately or simultaneously, as shown in Table 2, into the liquid at 720 °C overheating temperature. The melt was stirred to achieve a uniform distribution of master alloys, and allowed to rest for 5 min. The molten metal was then poured into a divided mild steel mold (preheated to 200 °C) to produce ingots.

Table 2 Types of compositions investigated

Alloy	Al10Sr, ppm	Al5TiB, ppm
M0—base alloy	0	0
M1	375	125
M2	125	125
M3	125	375
M4	250	250
M5	250	0
M6	0	250

Samples for metallographic examination were all cut from a similar position (at a height of 50 mm from the bottom of the ingots) and then prepared using the standard metallographic technique of grinding with SiC abrasive paper and polishing with diamond paste, followed by final polishing with OP-s colloidal silica suspension. To reveal the grain size, samples were electrolytically etched using Barker's reagent. The optical micrographs were captured using a light microscope (Axio Observer Z1). To characterize the ingots' microstructures, a free image analysis software (ImageJ) was used. The mean Mg_2Si particle area (μm^2) was calculated by analyzing at least 1000 particles in an equiaxed zone using the formula given in Eq. (1),

$$\text{Mean area} = \frac{1}{m} \sum_{j=1}^m \left(\frac{1}{n} \sum_{i=1}^n A_i \right), \quad (1)$$

where A_i is the area of a single silicon particle, n is the number of particles in a single field, and m is the number of the fields. To estimate the average grain size in an equiaxed zone, an intercept technique was employed. Specifically, the size of Al grains was determined based on the polarized light images, and the quantification was performed according to an ASTM E112 standard test procedure using a linear intercept technique. At least two fields were considered for each calculation, with approximately 50 intercepts in each field depending on the grain size. Secondary dendrite arm spacing (SDAS) measurements were performed on the samples etched with Weck's reagent using a linear intercept method. Wherever possible, at least two fields were analyzed for each measurement, with approximately 50 secondary dendrite arms in each field.

Chemical composition microanalysis was performed on the scanning electron microscope (SEM; Zeiss Supra) equipped with an energy-dispersive X-ray spectroscopy

Table 1 Chemical composition of the AlMg5Si2Mn alloy (mass%)

Element	Mg	Fe	Si	Cu	Zn	Ti	Mn	Al
Mass, %	5.17	0.12	1.9	0.005	0.001	0.009	0.58	Balance

(EDS) apparatus. Additionally, X-ray diffraction (XRD) was performed using a PANalytical X'Pert Pro diffraction system equipped with a CoK α radiation source.

The microstructure characterization under higher magnification was performed on the transmission electron microscope JEM 3010UHR from JEOL, at an accelerating voltage of 200 kV.

The samples were subjected to a potentiodynamic test to evaluate their pitting corrosion resistance by recording the anodic polarization curves. The measuring instrumentation consisted of an Atlas 0531 EU potentiostat (supplied by ALTAS-SOLLICH) and an electrochemical cell with a three-electrode system: (1) reference electrode (saturated Ag/AgCl electrode), (2) supporting electrode (platinum wire; PtP-201), and (3) working electrode (tested samples). The associated PC was equipped with AtlasLab software to save and evaluate recorded polarization curves. The potentiodynamic test began by establishing the open circuit potential, E_{ocp} , at equilibrium (electroless) conditions. The polarization curves were recorded starting at the value of the baseline potential, E_{init} , which was determined according to the formula, $E_{init} = E_{ocp} - 100$ mV. The voltage change progressed towards anodic potentials at 2 mV/s. Once the anodic current density, I , reached 1 mA/cm², or the maximum measuring range reached 4000 mV, the polarization direction was switched. Based on the recorded polarization curves, the following characteristic pitting corrosion resistance parameters were determined: potential corrosion (E_{corr}), breakdown potential (E_b), and repassivation potential (E_{cp}). The polarization resistance, R_p , was determined using Tafel's method. The electrochemical tests were carried out in 3% NaCl solution at $T = 24 \pm 1$ °C and pH = 7 ± 0.2 .

The thermal-derivative analysis (TDA) of melting and solidification series was carried out using a Universal

Metallurgical Simulator and Analyzer (UMSA). Specimens for thermal treatment were all cut from similar positions in the cast ingots and machined into cylinders with 18 mm in diameter and 20 mm in length.

3 Results

3.1 Characterization of the base material

Figure 1a presents the polarized light image of the microstructure of the AlMg5Si2Mn alloy, which is composed of equiaxed grains uniformly distributed throughout the observed area of approx. 220 μ in size. Figure 1b shows the bright field image, thus illustrating the size, morphology, and distribution of secondary phases in the base alloy matrix.

Figure 2 displays an SEM secondary electron image and the corresponding EDS spatial distribution maps of Al, Mg, Si, Fe, and Mn elements in the base alloy. The initial microstructure is composed of α -Al solid solution dendrites, eutectic interconnected Mg₂Si phases with a lamellar or coarse flake-like morphology, and small irregularly-shaped (Fe and Mn) precipitates. The EDS spatial distribution maps also revealed the presence of Mg-enriched clusters located in the inter-dendritic regions. The Al–Mg binary phase diagram represents the β -Al₃Mg₂ phase.

3.2 Characterization of modified materials

3.2.1 Sample quality

Figure 3 provides an overview of the microstructure throughout the entire cross-section of the ingots. The

Fig. 1 Microstructure of the base material, AlMg5Si2Mn alloy. **a** Polarized light image. **b** Bright-field image

Fig. 2 SEM SE image (top, left) and the spatial distribution maps of elements in analyzed microareas of the AlMg5Si2Mn base alloy

microstructure is characterized by primary aluminum dendrites (bright regions) surrounded by eutectic cells of Mg_2Si (dark areas). Close to the surface of the mold (at the skin zone), fine dendrites are observed. The size of the primary aluminum dendrites increases slightly in the band zone and remains nearly constant in an equiaxed zone. Based on the analyzed micrographs, the equiaxed zone contains casting defects in the form of inter-dendritic shrinkage pores, as highlighted in Fig. 3. Potential sources of the cavities in the ingots may be dissolved gas released during solidification, bubbles entrapped by turbulence, or hydrogen absorbed by the alloy due to Al10Sr addition [26]. In the equiaxed zone, the eutectic Mg_2Si particles are clustered into “islands”. The size of the clusters varies slightly depending on the chemical composition of the ingot. Large clusters (about 0.5–1 mm in diameter) are visible in the equiaxed zone of the M1 and M3 samples. In contrast, the distribution of Mg_2Si clusters in other specimens was more uniform, and their size did not exceed 200–300 μm .

3.2.2 Effect of TiB and Sr on the grain refinement and Mg_2Si modification

Figure 4 presents an overview of the grain size in an equiaxed zone of each sample with different amounts of Al5TiB and/or Al10Sr added. The microstructure of a reference sample is shown in Fig. 1a, and the summarized results of the grain size measurements are compiled in Table 3. Regardless of the chemical composition of an

alloy, the microstructure observed in an equiaxed zone is composed of equiaxed grains without any favorable crystallographic orientation. It is also apparent that the grain structure was refined after addition of Al10Sr and Al5TiB master alloys. Based on the obtained results, it was determined that the most effective grain refinement was observed in the M4 sample, which had a measured average intercept length of 144 μm , indicating that the average grain size was reduced by approx. 65%. This result suggests that the simultaneous addition of both Al5TiB and Al10Sr effectively modifies the grain structure of the AlMg5Si2Mn alloy. We also examined whether the microstructure was affected by simultaneous addition of lower quantities of refiners/modifiers. For this purpose, sample M2 was prepared, and this material exhibited decreased grain refinement efficiency. The measured average intercept length is $\sim 202 \mu\text{m}$. To put these observations into context, the effect of a single inoculation on the structure of the AlMg5Si2Mn alloy was also investigated; specifically, Al10Sr and Al5TiB master alloys were separately added into the base alloy in the amount of 250 ppm (samples M5 and M6, respectively). We observed that the grains were refined in both M5 and M6 samples, and the measured average intercept lengths were approximately 160 μm . To determine whether there may be an adverse effect of TiB and/or Sr modification, samples with different TiB/Sr ratios were prepared (M1 and M3). Upon increasing the content of Al5TiB, the refining efficiency was not appreciably enhanced, and the measured average intercept length

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Fig. 4 Microstructures of the M1–M6 (a–f) samples (etched using Barker's reagent) observed under polarized light (equiaxed zone)

Table 3 Quantitative metallography results of the as-cast AlMg-5Si2Mn alloy with different Al10Sr and Al5TiB contents

Alloy	Mg ₂ Si particle area [μm ²]	Inter-fiber spacing [μm]	Grain size (equiaxed zone) [μm]	Secondary dendrite arm spacing [μm]
M0	3.90	–	220	–
M1	1.52	1.04	161	10.4
M2	1.59	1.18	202	11.3
M3	2.96	1.27	185	12.9
M4	1.29	0.98	144	11.4
M5	1.46	1.10	165	11.8
M6	1.76	1.10	160	11.5

was approx. 185 μ. This may indicate an unfavorable interaction between Al5TiB and Al10Sr in this sample. Increasing the Al10Sr content led to grain refinement resulting in particle sizes of about 160 μ. This observation indicates that unfavorable interactions between the modifiers/refiners in the M3 sample depends on the B/Sr ratio.

Figures 1b and 5a–f show the bright-field microstructures of the base and modified alloys, and the results of Mg₂Si particle area measurements are listed in Table 3. It is clear that for the base alloy (Fig. 1b), the eutectic Mg₂Si phase exists primarily as large, interconnected, flake-like particles with an average particle area of 3.9 μm². Compared with Fig. 5, the size of the eutectic Mg₂Si decreased slightly when the modifiers/refiners were added. Moreover, after the addition of the modifiers, the morphology of eutectic Mg₂Si also changes slightly. It is clear that the morphology in M1–M6 samples partially evolved from coarse interconnected and flake-like to lamellar and rod-like, and in some areas, a loss of eutectic Mg₂Si phase interconnectivity was observed. The lowest refining efficiencies (i.e., highest average Mg₂Si particle areas of 2.96 μm² and 1.76 μm²) were measured for samples M3 and M6, respectively, whereas the highest refining efficiency (i.e., lowest average Mg₂Si particle area of 1.29 μm²) was measured for sample M4. Based on these results, we concluded that TiB has a minor effect on the eutectic Mg₂Si size, but the separate addition of Al10Sr (M5

Fig. 5 Bright field images of the M1–M6 (a–f) samples showing the morphology and size of eutectic Mg_2Si precipitates (equiaxed zone)

sample) refines the eutectic Mg_2Si considerably (to an average measured Mg_2Si particle area of $1.46 \mu\text{m}^2$). It should be noted that the greatest Mg_2Si refinement was observed for the M4 sample, thus confirming the beneficial effect of both refining additives on the Mg_2Si size. The quantitative analysis was in agreement with the microstructure shown in Fig. 5. The samples containing the lowest average Mg_2Si sizes have primarily rod-like and lamellar-shaped structures. However, samples with larger average Mg_2Si size contained an increased amount of flake-like and curved flake-like precipitates.

The typical dendrite morphology of $\alpha\text{-Al}$ grains that was used for the secondary dendrite arm spacing measurements is also shown in Fig. 5. The SDAS values reported herein (Table 3) represent the mean values of 10–15 measurements. The data in Table 3 reveal that the applied modifications have also influenced the SDAS. Specifically, the lowest secondary dendrite arm spacing was measured for samples M1 and M4. Since the SDAS was slightly reduced, the eutectic Mg_2Si particles located between $\alpha\text{-Al}$ dendrite arms became smaller and adopted smaller inter-fiber spacings.

3.2.3 XRD investigation

To identify the phases in the modified alloy, XRD analysis was carried out, and Fig. 6 presents the stacked XRD patterns of the prepared experimental ingots. Only the main reflections of the face-centered cubic (FCC) aluminum and Mg_2Si phases were identified. The XRD analysis did not reveal any new phases formed because of the modifications. This may be because their low content and intensity could not be detected by XRD analysis.

Fig. 6 XRD patterns of modified AlMg5Si2Mn alloys

Fig. 7 SEM SE image (top, left) and EDS elemental mapping images of the selected area in an AlMg5Si2Mn alloy (M3 modification)

3.2.4 SEM investigation

Figure 7 presents an SEM SE image and the spatial distribution of Al, Mg, Si, Ti, B, and Sr elements in the M3 sample. The elemental mapping images show large Ti particles ($\sim 15\text{--}20\text{ }\mu\text{m}$ in size) located in the center of $\alpha\text{-Al}$ dendrites. The SEM-EDS maps also reveal an increased concentration of Sr in areas identified as Mg_2Si clusters. However, these findings may suffer from an identification error inherent to the SEM equipment, which results from the fact that silicon ($K\alpha = 1.739$) and strontium ($L\alpha = 1.806$) have overlapping X-ray emission peaks, and insufficient resolution causes these to be identified as the same element.

Figure 8 shows an SEM SE image and the spatial distributions of Si, Mg, Mn, Ti, Sr, Al, and Fe elements in the M4 sample. An increased concentration of Sr in areas identified as Mg_2Si clusters was observed in this sample. The SEM-EDS maps also revealed the presence of phases composed of (Fe/Mn) and (Al/Mg), suggesting the presence of a $\beta\text{-Al}_3\text{Mg}_2$ phase (these regions are indicated with yellow arrows in the top, left image).

Figure 9 displays the SEM SE microstructure and corresponding results from pointwise chemical composition microanalysis of the M4 sample. These results confirm the existence of plate-like TiB agglomerates that are about $1\text{--}2\text{ }\mu\text{m}$ long, located at the center of the eutectic Mg_2Si cells (dark regions).

To characterize the three-dimensional morphology of eutectic Mg_2Si phases in the M4 sample, the Al matrix was deeply etched (removed) using an HCl-water solution (15%).

Fig. 8 SEM SE image (top, left) and EDS elemental mapping images of a selected area in an AlMg5Si2Mn alloy (M4 modification)

Fig. 9 SEM SE image of the M4 sample and the corresponding EDS spectrum

As shown in Fig. 10a, the eutectic Mg_2Si phase has divorced character (i.e., some rod-like and some lamellar morphology) arranged like a “cable harness”. It is also clear that primary Mg_2Si exists in the center of Mg_2Si cells with perfect octahedral morphology (Fig. 10b).

3.2.5 TEM investigation

Figure 11 presents the STEM-HAADF (high-angle annular dark-field) images of the M4 sample, clearly showing the two-dimensional morphology of binary eutectic Mg_2Si . This confirmed that the Mg_2Si eutectic phase has a divorced morphology, with lamellar and rod-like Mg_2Si regions that coexist in the eutectic structure. It was also observed that the Mg_2Si lamellas are “decorated” with other second-phase particles.

According to the STEM-EDS elemental mapping presented in Fig. 12, the precipitates observed at the eutectic

Mg_2Si lamellas are composed of Al, Si, Fe, and Mn elements. However, considering solely the EDS spatial distribution analysis, it is difficult to precisely identify the crystal structure or determine the chemical formulae of the intermetallic phases. Therefore, TEM analysis was employed to support the structure and phase identification. Figure 13a and b presents the bright and dark field TEM images, displaying the 2D morphology of the Mg_2Si phase. The corresponding SAED (selected area diffraction) (Fig. 13c) confirms the presence of the cubic Mg_2Si phase, whose lattice parameter was determined to be ~ 0.638 nm. Similarly, Fig. 14a and b presents the bright and dark field TEM images showing the 2D morphology of the Fe/Mn-rich intermetallic phase. The corresponding SAED (Fig. 14c) indicates a cubic structure with an Im space group, which is consistent with the $\text{Al}_{19}\text{Fe}_4\text{MnSi}_2$ phase. The lattice parameter was determined to be 1.257 nm, which was in good agreement with that reported by

Fig. 10 The 3D morphology of the **a** eutectic and **b** primary Mg_2Si phases in different areas

Fig. 11 STEM-HAADF images of the M4 sample showing the morphology of the Mg_2Si phase

Nuckowski et al. (lattice parameter = 1.256 nm in the $\text{Al}_{19}\text{Fe}_4\text{MnSi}_2$ phase) [27].

Figure 15 shows the high-resolution transmission electron micrographs (HRTEM) and corresponding SAED of the M4 sample. As observed in Fig. 15a–c, the M4 sample contains two types of precipitates. One type of precipitate, identified as Mg_2Si with a diameter of 100 nm (Fig. 15a), has a polycrystalline structure, which was confirmed by the observed moiré fringes. The second type of precipitate was identified as a Mg_9Si_5 phase, which has a very fine nanostructure; these precipitates are coherent with the Al matrix, as shown in Fig. 15c.

3.3 Effect of modifications on the corrosion resistance

The polarization curves obtained for all tested samples are shown in Fig. 16, and the pitting corrosion resistance parameters are presented in Table 4. There is no substantial difference among the polarization curve shapes of

the tested samples, although they have different breakthrough potentials. The highest breakthrough potential values were recorded for the M1 and M4 samples, which had mean values of approximately $E_b = -633$ mV and $E_b = -640$ mV, respectively. The other tested samples had values in the range between -670 and -720 mV. These breakthrough potentials were closer to the values for the material in its initial state ($E_b = -760$ to -690 mV), as reported by Hu et al. [28]. Observation of the breakdown potential, E_b , in all samples indicated the initiation and development of pitting corrosion, regardless of the type of modification in the samples. In contrast, a repassivation potential, E_{cp} , was only recorded for the M4 and M6 samples. This repassivation potential represents the reconstruction of the passive layer, which was damaged by corrosion processes. However, the curves corresponding to all of the samples have a wide passive region, which reflects the protection of the oxide film on the surface. For all tested samples, regardless of the type of modification, the values of corrosion resistance, E_{corr} ,

Fig. 12 STEM-EDS elemental mapping images of the selected area (M4 sample)

Fig. 13 Bright field (a), and dark field (b) TEM micrographs, and the corresponding SAED (c) of the M4 sample. Images show the eutectic Mg_2Si phase

Fig. 14 Bright field (a), dark field (b) TEM micrographs, and the corresponding SAED (c) of the M4 sample. Images show the $\text{Al}_{19}\text{Fe}_4\text{MnSi}_2$ phase

Fig. 15 TEM images showing the nanoscale precipitates present in the M4 sample (a–c) and corresponding SAED (d–f)

Fig. 16 Potentiodynamic polarization curves of modified alloy samples

were similar (in the range from -855 to -725 mV) and were almost 1.5 times higher than the values presented by Hu et al. [28] for the baseline AlMg5Si2Mn alloy. These results can be attributed to the decreased influence of the Al+Mg₂Si eutectic region on the corrosion behavior of AlMg5Si2Mn.

Table 4 Results of potentiodynamic tests

No	E_{corr} [mV]	E_b [mV]	E_{cp} [mV]	R_p [k Ω cm ²]
M1	-774	-633	–	31
M2	-818	-690	–	30
M3	-725	-670	–	16
M4	-776	-640	-775	46
M5	-770	-720	–	4
M6	-855	-670	-947	30

3.4 Discussion

The influence of the TiB refiner and Sr modifier on alloy morphology and properties have been studied by many scientists in recent years, because of their broad use in combination with Al–Si alloys [29, 30]. Liao et al. [31] reported that, in the Al–Si hypoeutectic alloys, a mutual poisoning effect occurs when the content of Sr and B exceed a certain limit. This mutual poisoning effect in near-eutectic Al–Si alloys was also recently confirmed by Farahany et al. [32] and Goldbahar et al. [33]. Some additional research has also probed the refinement of Mg₂Si by Sr [19, 34] and/or TiB [17, 35] additions. Many theories have been proposed to elucidate the refinement mechanism, and this subject has been somewhat controversial

over the years. According to the nucleation theory, Al_3Ti core-shell particles dispersed in the Al matrix can promote the nucleation of α -Al grains. In the TiB-modified alloys, a thin layer of Al_3Ti typically covers the TiB_2 particles [36], thus reducing the lattice mismatch. The effectiveness of the nucleating particle depends on the similarity between the crystal structure type and lattice mismatch of the nucleating particle and that of the α -Al matrix. Therefore, Al_3Ti phases with lattice structures similar to that of Al and a low lattice mismatch relative to the aluminum matrix of $\sim 2.08\%$ ($3.967 \text{ Al}_3\text{Ti}$) [37] and (4.049750 Al) [38] can act as a potential substrate for nucleation of the α phase, thus enhancing its grain refinement efficiency. Several researchers [33, 35, 39] also indicated that TiB_2 particles act as the heterogeneous nuclei of the primary Mg_2Si phase. An investigation conducted by Li et al. [17] confirmed that TiB_2 has good lattice matching coherence with Mg_2Si (lattice mismatch of only 4.64%), so these particles could act as nuclei for the primary Mg_2Si phase. It is also widely accepted that the modification of a primary Mg_2Si phase depends on the heterogeneous nucleation mechanism induced by Sr-rich particles. Tang et al. [20] determined that the modification of a primary Mg_2Si phase mainly depends on the dissolved Sr, such that the preferred growth manner of the Mg_2Si phase is depressed, while the isotropic growth is enhanced in the modified alloys. It has also been proposed that the absorption of Sr on $\{100\}$ facets of Mg_2Si crystals can be responsible for the refinement and morphological changes of the Mg_2Si phase [40]. In general, changes in the morphology, size, and density of Mg_2Si particles are related to nucleation and growth phenomena occurring during solidification.

The experimental results presented herein indicate that the addition of Al10Sr and/or Al5TiB master alloys represents a promising method for grain refinement and Mg_2Si phase refinement/modification of the AlMg5Si2Mn casting alloy. Our results clearly indicate that even minimal amounts of Al10Sr and/or Al5TiB master alloys added to the base AlMg5Si2Mn alloy decrease the grain size and alter the size and morphology of the eutectic Mg_2Si phase. The results of EDS elemental mapping in combination with a thorough literature review allowed the conclusion that the Ti particles, covered by a thin layer of Al_3Ti (Fig. 17) due to low lattice mismatch, can act as nucleating sites for the α -Al phase, thus enhancing the grain refining efficiency in AlMg5Si2Mn alloy (see Table 3). However, the obtained experimental data also suggest that the grain refinement efficiencies corresponding to samples M5 and M6 modified separately with 250 ppm Sr and 250 ppm TiB, respectively, are similar. Consequently, it can be assumed that another factor must be responsible for the grain refinement, because the M5 sample does not contain Al5TiB, therefore, it cannot be influenced by Al_3Ti nucleating particles.

Fig. 17 EDS elemental map showing a Ti particle with an Al_3Ti layer. The EDS signal from Al and Ti are overlapped (M3 sample)

Table 5 Solidification parameters calculated from cooling and first derivative curves

Sample	$T_{\alpha\text{DEN}}^{\text{NUC}}$ ($^{\circ}\text{C}$)	$T_{\alpha+\text{Mg}_2\text{Si}}^{\text{NUC}}$ ($^{\circ}\text{C}$)	$T_{\text{Al}_3\text{Mg}_2}^{\text{NUC}}$ ($^{\circ}\text{C}$)	T_{SOL} ($^{\circ}\text{C}$)
M0	616.10	587.46	434.5	414.1
M4	619.79	586.17	432.1	416.1
M5	620.33	586.60	436.6	421.4
M6	617.11	586.98	432.5	416.1

To better understand the impact of individual additives on the crystallization process, differential thermal analysis (DTA) was performed. The full DTA curves are included as supplementary data for this article, while the main solidification parameters calculated from the cooling and first derivative curves are presented in Table 5.

From the obtained cooling curves, it was possible to extract the characteristic temperatures for the formation of Al dendrites, and these temperatures are listed in Table 5. It is evident that the nucleation event for the α -Al dendrite (616.1°C) in the M0 sample occurs at a higher temperature (617.11°C) than for the sample with 250 ppm Al5TiB added (M6 sample). This characteristic temperature is even higher (619.79°C) for the sample with 250 ppm Al10Sr added (M5 sample), and further increases (620.33°C) following mutual addition of 250 ppm Al10Sr and 250 ppm Al5TiB (M4 sample). Based on the obtained data it can be concluded that Sr has a significantly higher impact than TiB on the nucleation event for α -Al dendrites. The addition of Al10Sr accelerated the α -Al nucleation, with more nucleation sites in the melt. Since the grain size in casting is associated with the number of nucleation sites available in the melt at the liquidus or nucleation temperature of the α -Al phase [41], a higher nucleation temperature of α -Al allowed new crystal growth ahead of the solidification front, and thus, enhanced

the grain refinement. This phenomenon explains the pronounced microstructure refinement effect observed in the M5 sample, without Al5TiB addition.

Figure 18 shows the Mg₂Si eutectic region of the cooling curves constructed for selected alloys treated with various amounts of Al10Sr and/or Al5TiB. It is clear that the addition of 250 ppm Sr, 250 ppm TiB, and 250 ppm Sr + 250 ppm TiB to the melt, slightly decreased the eutectic Mg₂Si nucleation temperature from 587.46 °C (M0 sample) to 586.60 °C (M5 sample), 586.98 °C (M6 sample), and 586.17 °C (M4 sample), respectively. The lowest eutectic Mg₂Si nucleation temperature (M4 sample) corresponds to the finest eutectic structure, as shown in Fig. 5d and Table 4. The presented results are in good agreement with an investigation conducted by Farahany et al. [42], who reported that minor addition of Sr (0.01%) to an Al–Mg₂Si–Cu in-situ composite caused a depression in the eutectic Mg₂Si nucleation temperature, leading to refinement of the eutectic Mg₂Si phase.

In this study, it was also determined that the TiB₂ particles can act as heterogeneous nuclei (Fig. 9) of a primary Mg₂Si phase because they have a low lattice mismatch with Mg₂Si. Similar findings involving an Al–12.67Mg–10.33Si alloy treated with 0.2% Al5TiB were reported by Li et al. [17]. From the experimental results discussed herein, it can be concluded that many factors are responsible for the beneficial effect of the applied microstructure modifications. The microstructure enhancement (grain refinement) is caused by Al₃Ti particles from the Al5TiB refiner, and the increased nucleation temperature of the α-Al phase is a result of the addition of the Al10Sr modifier. Moreover, the addition of Sr reduces the Mg₂Si nucleation and growth temperature, which contributes to the refinement and partial modification of the Mg₂Si eutectic phase [43]. In general,

Fig. 18 Cooling curves focusing on the Mg₂Si eutectic reaction in the M0, M4, M5, and M6 samples

the multi-modification effect observed when treating a sample with both Al10Sr and Al5TiB causes the greatest microstructural enhancement (M4 sample).

The potentiodynamic test revealed that the addition of Al10Sr and Al5TiB in equal proportions had a positive effect on the corrosion characteristics of the tested AlMg-5Si2Mn alloy. Generally, aluminum alloy materials have a high affinity for oxygen, which spontaneously generates a passive Al₂O₃ film. The M4 sample in this study had the lowest grain and eutectic Mg₂Si particle size, as well as a repassivation potential, which means that the aggressive ions were removed from the pit via diffusion to the electrolyte, allowing the corrosion pits to be replaced by an passive oxide layer. As a result, the pitting could not be initiated. According to research conducted by Zeng et al. [44], the corrosion properties of aluminum alloys depend primarily on the alloy's chemical composition and the size of the precipitates. Therefore, the higher pitting corrosion resistance exhibited by the M4 sample may be associated with the fact that this modified alloy had the smallest size of Mg₂Si eutectic, as reported by Li et al. [45].

4 Conclusions

In this article, we investigated the influence of the combined addition of Al10Sr and Al5TiB on the microstructure of AlMg5Si2Mn alloy. The major conclusions from this work are summarized below:

- Addition of Al5TiB and/or Al10Sr causes a partial morphological change and refinement of the eutectic Mg₂Si phase;
- Simultaneous addition of Al10Sr and Al5TiB enhances the grain microstructure of the AlMg5Si2Mn alloy;
- SEM results show that TiB₂ particles can act as the heterogeneous nuclei of the primary Mg₂Si phase, while the Al₃Ti particles heterogeneously nucleate the primary aluminum grains;
- Addition of Al10Sr to the melt increases the α-Al nucleation temperature, which decreases the grain size of the AlMg5Si2Mn alloy;
- Potentiodynamic tests revealed that the finest microstructure enhanced the corrosion resistance of tested samples. The optimum corrosion resistance was recorded for the M4 sample, which contained the smallest measured average grain and eutectic Mg₂Si particle size.

Further investigations are necessary to comprehensively investigate the interactions between Sr and TiB in an AlMg-5Si2Mn alloy to clarify their mutual influence on the crystallization process and microstructural enhancements. In addition, future research will focus on interesting aspects of

tuning the microstructure of the AlMg₅Si₂Mn alloy due to grain boundary wetting by the secondary phase (as discussed in [46] and [47], using heat treatment processes, or applying the strategies presented in [48] and [49].

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Data availability The data that support the findings of this study are available from the corresponding author, [Przemysław Snopiński], upon reasonable request.

Code availability All data generated or analysed during this study are included in this published article.

Compliance with ethical standards

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

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