



Improved mechanical properties of in-situ Al-Mg₂Si composites via Sr addition

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ABSTRACT

Effects of strontium addition to the Al-Mg₂Si composite on the microstructure, phase formation, and improvement of the mechanical properties in the wrought condition were investigated. Accordingly, 0, 1, 2, 3, and 5 wt.% Sr was added to the Al-15Mg₂Si composite. Results revealed that the optimum Sr addition up to 2 wt.% leads to the modification of Mg₂Si particles. Moreover, the emergence of Al₂Si₂Sr particles resulted in the development of a hybrid composite microstructure. These microstructural modifications, combined with the extrusion process, resulted in the improvement of mechanical properties, in terms of yield stress, ultimate tensile strength, total elongation, and tensile toughness. For instance, tensile toughness, as a measure of the strength-ductility balance, for the Sr addition of 2 wt.% showed 30% improvement compared to the base composite. However, higher Sr additions of 3 and 5 wt.% resulted in the formation of coarse needle-shaped Al₂Si₂Sr and Al₄Sr particles, which resulted in poor ductility and work-hardening behavior. Moreover, the significant reduction in the amount of Mg₂Si phase at high Sr additions was found to be in contrast to the idea of hybrid composite formation. Accordingly, the present work sheds light on the development of hybrid Al-Mg₂Si/Al₂Si₂Sr composites via optimum Sr addition to the Al-Mg₂Si composite for the improvement of mechanical behavior.

Keywords: Hybrid metal-matrix composites; Solidification and casting; Modification; Thermomechanical processing; Tensile properties.

1. Introduction

Al-matrix composites (AMCs) have garnered significant interest from different industries because of their excellent specific strength and stiffness, increased hardness and wear resistance, and favorable mechanical properties [1-4]. Composites made by incorporating particles often experience a weak interface between the matrix and reinforcement, along with an uneven distribution of reinforcement particles. As a result, the in-situ processing has been proposed as a feasible solution to address these issues [5,6].

Among in situ composites, the Al-Mg₂Si system has demonstrated encouraging outcomes, with blocky primary Mg₂Si reinforcements and flake-like eutectic or pseudo-eutectic Mg₂Si developing within the eutectic cell during the solidification of Al alloys containing Si and Mg [7,8]. Unfortunately, the coarse Mg₂Si phase results in the deterioration of mechanical properties [9]. Accordingly, the modification of these particles is required, which can easily be achieved by the addition of certain elements. The influence of various modifiers like rare earth elements [10,11], antimony [12],

phosphorus [13], lithium [14], and strontium [15,16] on the morphology of Mg_2Si in the Al-Mg-Si system has been examined. For example, by incorporating Sr, the shape of primary Mg_2Si particles has been significantly modified toward improved mechanical properties [16].

The processing of hybrid composites is a viable approach for the improvement of various properties of composite materials [17]. This can be achieved via an in-situ method by the addition of other elements into the chemical composition. In this regard, however, the in-situ hybrid Al- Mg_2Si composites with the Sr-containing hybrid phases need to be further investigated. It has been observed that Sr addition leads to the

appearance of Al_2Si_2Sr and Al_4Sr in this system [18]. Accordingly, high Sr additions can be used to process hybrid composites based on the Al- Mg_2Si system. Moreover, for improving the mechanical properties and obtaining good dispersion of the reinforcement phases, thermomechanical processing routes such as hot extrusion are required. In this regard, the impact of hot extrusion [19,20] on the metallurgical characteristics of Al- Mg_2Si composites has previously been examined, confirming the merits of the elevated-temperature deformation in this regard [21]. Nevertheless, the literature lacks reports on the hybrid Sr-containing Al- Mg_2Si composites. Accordingly, the present work aims to deal with this subject.

Table 1- chemical composition (wt.%) and assigned name of the samples.

Alloy Code	Si	Mg	Sr	Al
0Sr	5.5	9.5	0	Balance
1Sr	5.5	9.5	1	Balance
2Sr	5.5	9.5	2	Balance
3Sr	5.5	9.5	3	Balance
5Sr	5.5	9.5	5	Balance

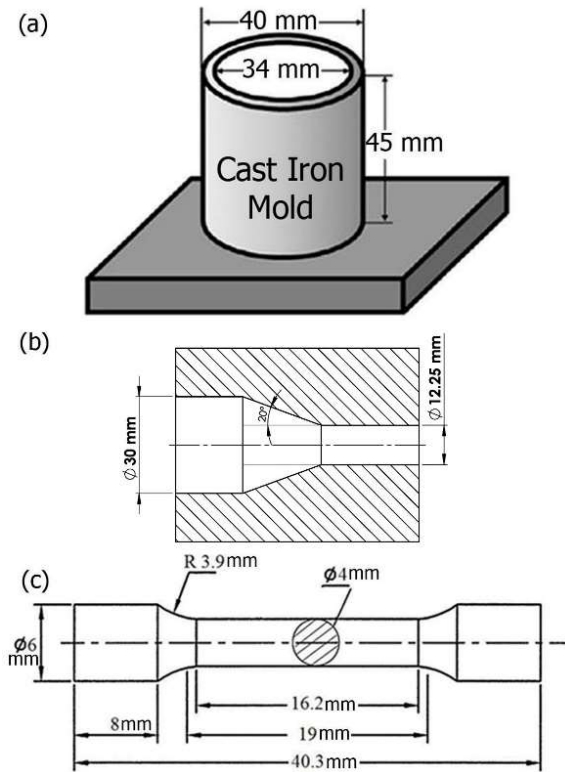


Fig. 1- Schematics of the (a) mold, (b) extrusion process, and (c) tensile testing sample.

2. Experimental details

2.1. Materials and processing

In-situ aluminum matrix composites with the chemical composition listed in Table 1 were produced in an induction furnace utilizing 99.8% Al and 99.9% Mg ingots, as well as Al-30 wt.% Si and Al-10 wt.% Sr master alloys. While the mentioned values in Table 1 are nominal, the chemical compositions of the master alloys have previously been determined by the inductively-coupled plasma spectroscopy (ICP) to make the charge calculation more reliable. When the melt temperature was raised to 770 °C, it was cast into a preheated metallic mold at 200 °C, as illustrated schematically in Figure 1a. After homogenization treatment at 450 °C for 7 h, the extrusion process was performed at 400°C with an extrusion ratio of 6:1 and an extrusion speed of 10 mm/s, as shown in Figure 1b.

2.2. Characterization

After metallographic sample preparation, the samples were etched with Keller's reagent (5 ml HNO₃ – 3 ml HCl – 2 ml HF – 190 ml H₂O). An optical microscope (Olympus Vanox OM) and a scanning electron microscope (MIRA3 TESCAN SEM) were used for microstructural investigations. Moreover, the image analysis technique using the ImageJ software was used for characterization. The SEM was equipped with an energy-dispersive spectroscopy EDS detector for elemental analysis. Phase identification relied on the X-ray diffraction (XRD) technique, utilizing a PHILIPS XRD with Cu-ka radiation. Tensile properties were characterized through room temperature testing at a crosshead speed of 0.1 mm/min using the subsize ASTM E8 samples depicted schematically in Figure 1c. Three samples of each alloy were tested to guarantee the consistency of the results. The strength-ductility balance was assessed by calculating tensile toughness, which is an estimate of the area beneath the engineering stress (S) – engineering strain (e) curve using the trapezoidal method:

$$Area = \sum (e_i - e_{i-1})(S_i + S_{i-1})/2 \quad (1)$$

3. Results and discussion

3.1. Microstructural and phase analyses

Optical micrographs of the as-cast ingots are presented in Figures 2a to 2e. Regarding the base 0Si alloy, according to the microstructure shown in Figure 2a, several phases can be identified: a polygonal dark phase, a eutectic structure, and the lighter phase. Since this sample is the well-known Al-15Mg₂Si composite [22,23] and according to the pseudobinary Al-Mg₂Si phase diagram

[24,25], the polygonal dark regions are related to the primary Mg₂Si particles, which form at the onset of freezing up to the eutectic point. These particles are quite coarse, as they form in the melt and can freely grow. The observed eutectic structure in Figure 2a is related to the α-Al/Mg₂Si eutectic structure. Accordingly, two types of Mg₂Si particles are available in this system, namely the primary Mg₂Si particles and the Mg₂Si present in the eutectic structure. The XRD pattern of the 0Sr alloy in Figure 3 also confirms that the only secondary phase in this system is the Mg₂Si phase. On the other hand, the lighter phase observed in Figure 2a is the α-Al, which is a common feature in the microstructures of the Al-Mg₂Si composites [26,27].

As mentioned above, the primary Mg₂Si particles are quite coarse, as they form in the melt and can freely grow, which necessitates the modification of these particles. As can be seen in the microstructure of the 1Sr alloy in Figure 2b, the size of the primary Mg₂Si particles has reduced significantly, confirming the modification effect of Sr, which is consistent with previous reports [15]. On the other hand, as shown in Figure 2b and via its conformation in the XRD pattern of the 1Sr alloy in Figure 3, another phase appeared in the microstructure, which is the Al₂Si₂Sr phase. This phase remains the main secondary phase in addition to the Mg₂Si phase with increasing the Sr content up to 3 wt.%, as shown in Figures 2c and 2d and Figure 3, which is also consistent with the predictions of the JMatPro software [28] for the examined chemical compositions. For the 3Sr alloy, the Al₂Si₂Sr phase has a needle morphology similar to the 2Sr alloy but it has grown and became larger, which might affect the mechanical properties.

On the other hand, at high Sr addition of 5 wt.%, another phase appears in the microstructure, as shown in Figure 2e. The XRD pattern of the 5Sr alloy in Figure 3 reveals that this phase is the Al₄Sr phase, similar to previous works [18], which is also consistent with the predictions of the JMatPro software at high Sr additions. Therefore, it can be seen that Sr addition leads to the formation of the Al₂Si₂Sr phase, followed by the Al₄Sr phase.

A SEM image for the 5Sr alloy is depicted in Figure 2f, where these phases are shown by arrows. The EDS point analysis results from Points A and B are presented in Figure 2g. The exact values are not precise and the elemental analysis is qualitative. If both atomic and weight percents are taken into account, it can be seen that Point A is Sr-rich. This is expected to be the Al₄Sr phase, similar to other work [18]; while Point B is Si-Sr-rich, which is expected to be the Al₂Si₂Sr phase.

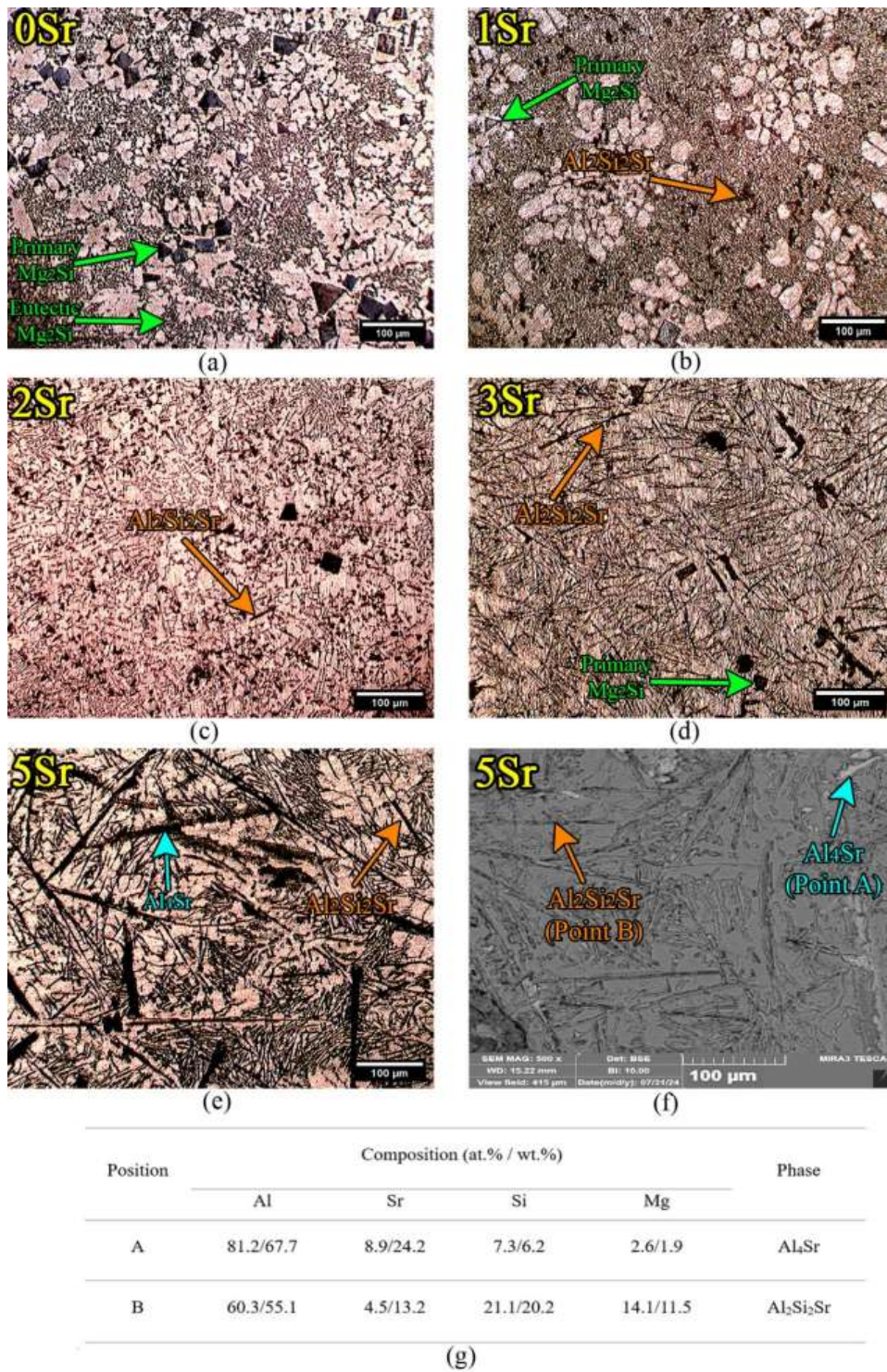


Fig. 2- (a-e) Optical micrographs of as-cast samples, (f) SEM image of 5Sr sample, and (g) EDS point analysis results for the points shown in part (f).

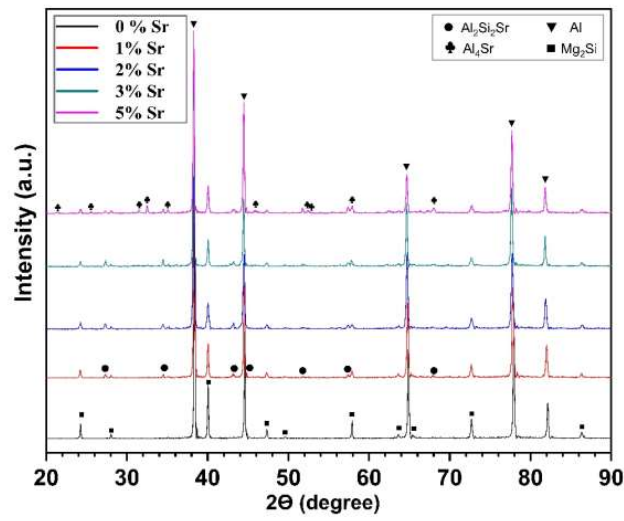


Fig. 3- XRD patterns of different samples.

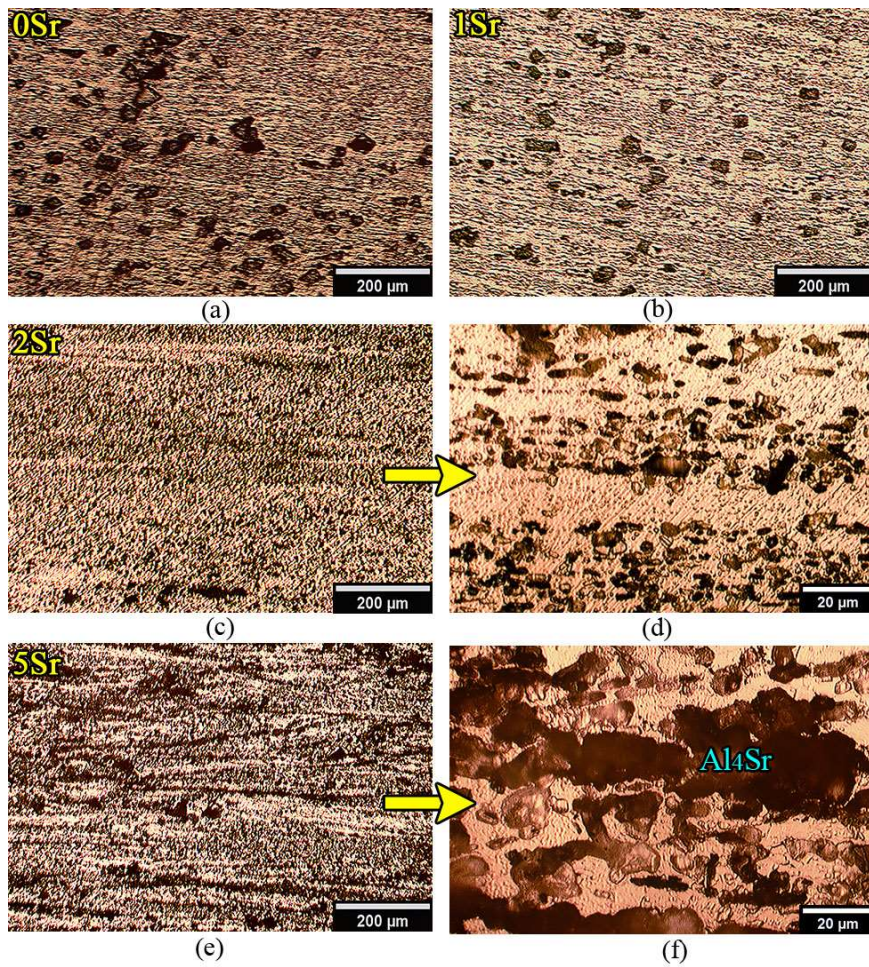


Fig. 4- Optical micrographs of the extruded samples.

Microstructures of the extruded samples are presented in Figures 4a to 2f. It can be seen in Figure 4a that the extrusion process resulted in the fragmentation of the secondary phases, especially the eutectic Mg_2Si phase. However, the primary Mg_2Si particles are less affected and can still be seen. This effect has previously been reported [29,30], relating to the flow of the primary Mg_2Si particles in the matrix.

Similar effects can be seen in the microstructures of the extruded 1Si and 2Sr alloys in Figures 4b to 4d, in addition to the fragmentation and dispersion of the Al_2Si_2Sr particles, which can clearly be seen in Figure 4d. Accordingly, a hybrid Al- Mg_2Si/Al_2Si_2Sr composite is formed, which is expected to exhibit good mechanical properties, as revisited later.

However, at large Sr contents, some larger particles with unfavorable shapes remain in the microstructure. As shown before, for the 3Sr alloy, the Al_2Si_2Sr phase has a needle morphology. This effect becomes more severe in the case of 5Sr alloy

upon the emergence of the needle-shaped large Al_4Sr particles. This effect is shown for the 5Sr alloy in Figures 2e and 2f. It can be seen that the large Al_4Sr particles remain in the microstructure even after the extrusion process, which is expected to adversely affect the mechanical properties, as revisited later.

In this work, the microstructural evolutions were studied in terms of intermetallic compound fragmentation. However, during hot extrusion, dynamic recovery (DRV) and dynamic recrystallization (DRX) may occur within the aluminum matrix, especially when the secondary particles are available. These aspects are quite important in determining the final microstructures and mechanical properties. Accordingly, in future works, the recrystallization phenomena, especially particle stimulated nucleation (PSN) can be investigated by electron backscattered diffraction (EBSD) and transmission electron microscopy (TEM) [31-34].

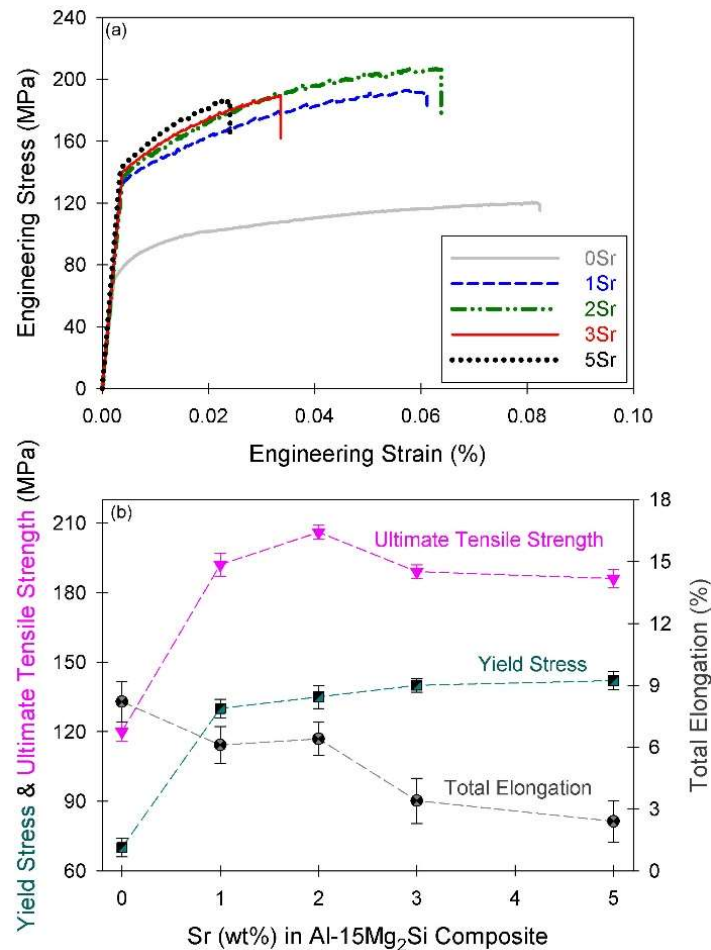


Fig. 5- (a) Tensile stress-strain curves of the extruded samples and (b) summary of mechanical properties.

3.2. Mechanical properties

Figure 5a illustrates the tensile curves of the extruded alloys, and the tensile properties are summarized in Figure 5b. It can be seen that the base 0Sr sample shows low strength but higher ductility compared to the Sr-added samples. Upon Sr addition up to 2 wt.% and formation of a hybrid composite, the yield stress (YS) and ultimate tensile strength (UTS) increase sharply, as depicted in Figure 5b. These remarkable increases come at the expense of a negligible decrease in the total elongation. The increase in YS and UTS is related to the modification of the Mg_2Si phase, as confirmed before, as well as the introduction of the Al_2Si_2Sr phase with appropriate morphology (Figure 4), which further strengthened the alloy. The retention of ductility up to 2 wt.% Sr is also related to the appropriate morphology of the Al_2Si_2Sr phase and the modification of the Mg_2Si phase, and the decreased size of these particles.

The sizes of the primary Mg_2Si particles in the extruded alloys are summarized in Figure 6a. It can be seen that the Sr addition resulted in a significant refinement of these particles. Moreover, according to the JMatPro predictions, Figure 6b reveals that the amount of the Mg_2Si phase remains at high values, while the hybrid Al_2Si_2Sr particles also appear. These findings are in line with the good mechanical properties up to 2 wt.% Sr addition. These findings can better be evaluated when compared to other competitive composites. For instance, the extruded Al-25%Mg2Si composite showed a UTS of 134 MPa and total elongation of 2.1%, which were improved by 1 wt% phosphorous addition to UTS of 210 MPa and total elongation of 4.1% [35]. In both condition, the UTS and total elongation values are inferior compared to the 2Sr alloy in the present work, which showed a UTS of 206 MPa and total elongation of 6.4%.

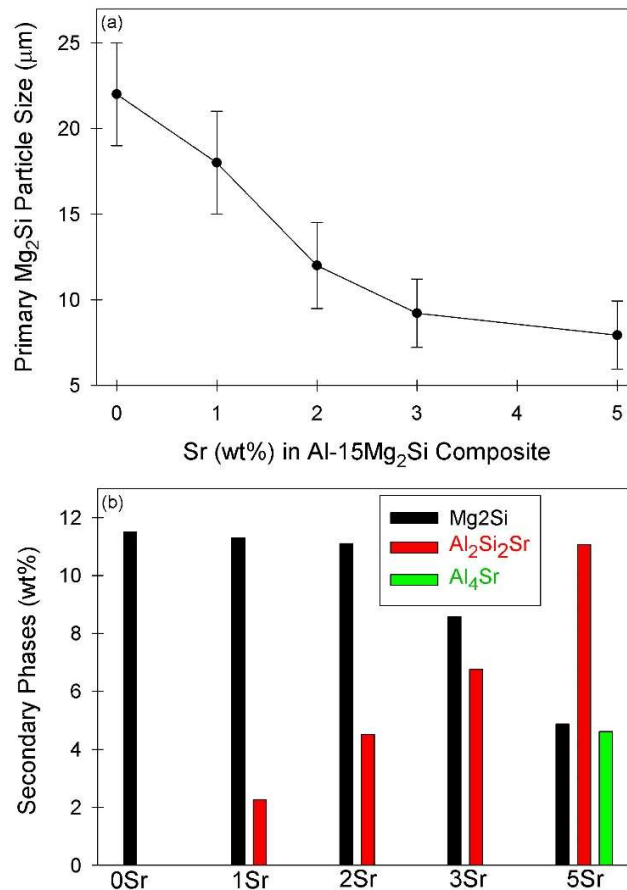


Fig. 6- (a) Size of Mg_2Si particles in extruded samples and (b) JMatpro phase predictions.

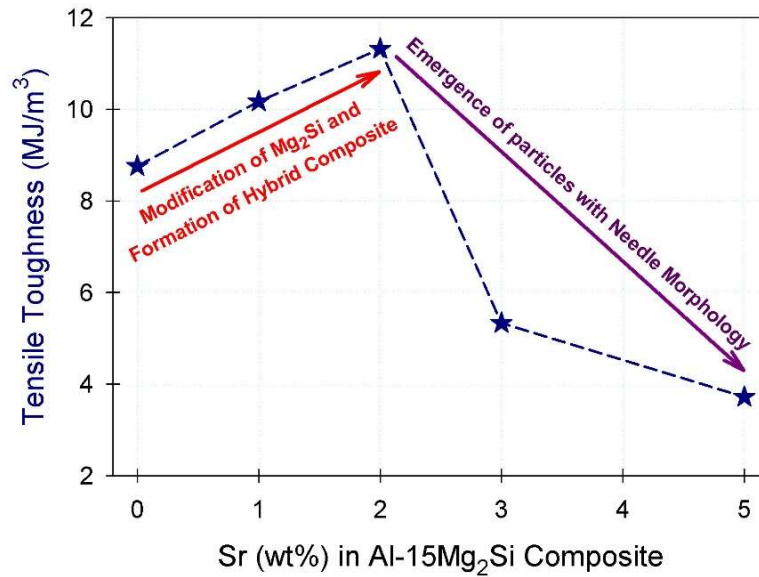


Fig. 7- Calculated values of tensile toughness for different samples.

At high Sr additions of 3 and 5 wt.%, the values of YS do not change considerably but are slightly higher compared to the 2Sr alloy. This is related to the presence of higher amounts of secondary phases. However, these samples show lower UTS values and poor total elongations. This is directly related to the presence of the phases with unfavorable needle morphology, which are the easy crack growth paths. This prevents the alloy from showing good work-hardening upon yielding, which prevents reaching high UTS values and severely limits ductility during tensile deformation [36]. On the other hand, most of Si has been used to form Al_2Si_2Sr phase at high Sr addition, resulting in a sharp decrease in the amount of Mg_2Si , as shown in Figure 6b, which is in contrast to the idea of hybrid composite formation.

These results can better be evaluated based on the tensile toughness, which was calculated according to Equation 1, and the results are presented in Figure 7. It can be seen that Sr addition up to 2 wt.% resulted in an increase in tensile toughness, revealing the improvement of the strength-ductility balance. However, further Sr additions of 3 and 5 wt.% led to the sharp decrease in the tensile toughness, which reveals that the Sr content needs to be carefully controlled. Therefore, optimum Sr addition for a hybrid Al- Mg_2Si/Al_2Si_2Sr composite with modified Mg_2Si phase and Al_2Si_2Sr phase with appropriate morphology is a viable approach to improve the mechanical properties of the in-situ composites based on this system.

Regarding the influential parameters on the mechanical behavior, it should be mentioned that factors such as the relative strengthening effects of different secondary phases should also be considered. However, by the addition of Sr, while the amount of Mg_2Si phase decreases, but the total amount of secondary phases increases, as depicted in Figure 6b, which makes it hard to evaluate each secondary phase. On the other hand, texture changes via Sr addition and hot extrusion process should also be taken into account, which are suggested to be performed in future works.

Since the Mg_2Si is the most common in situ formed phase in Al composites and the optimum amount of Sr for the improvement of mechanical properties is as low as 2 wt.%, the hybrid Al- Mg_2Si/Al_2Si_2Sr composites via optimum Sr addition to the Al- Mg_2Si composite for the improvement of mechanical behavior is cost effective. Accordingly, these hybrid composites might be compared to other common systems such as in-situ Al/Al-Fe one in terms of performance and cost effectiveness.

4. Summary

The impact of strontium addition into the Al- Mg_2Si composite on the microstructure, phase development, and enhancement of mechanical properties in the wrought state was examined. The following conclusions can be drawn:

(1) Strontium addition of up to 2 wt.% results in modification to Mg_2Si particles. Image analysis

revealed that the primary Mg₂Si particles refined from 22 μm in the base composite to 12 μm in the composite containing 2 wt.% Sr, revealing ~50% refinement. In addition, the formation of Al₂Si₂Sr particles led to the creation of a hybrid composite microstructure.

(2) Strontium addition of up to 3 wt.% resulted in the appearance of needle-like Al₂Si₂Sr particles; while higher Sr addition up to 5 wt.% resulted in the formation of coarse needle-like Al₄Sr particles, at the expense of Mg₂Si particles.

(3) For the optimum Sr addition of 2 wt.%, the combination of microstructural modifications led to enhancements in mechanical properties, specifically YS, UTS, total elongation, and tensile toughness. For example, tensile toughness, indicating the strength-ductility balance, for Sr addition of 2 wt.% exhibited a 30% enhancement relative to the base composite.

(4) Increased Sr additions of 3 and 5 wt.% deteriorated the tensile ductility and work-hardening characteristics. Accordingly, while high YS values were retained, the total elongation decreased to low values. Accordingly, Sr addition of 5 wt.% exhibited a 67% reduction in the tensile toughness relative to the optimum hybrid composite with 2 wt.% Sr.

(5) This study highlights the opportunity for the processing of hybrid Al-Mg₂Si/Al₂Si₂Sr composites through optimal Sr addition into the Al-Mg₂Si composite to enhance mechanical properties.

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