RESEARCH PAPER

Assessment of the Microstructure and Solidification Characteristics of Al–20% Mg_2Si Composite under Melt Superheating Treatment Using Thermal Analysis

Saeed G. Shabestari*, Sahar Ashkvary, Farnaz Yavari

* *shabestari@iust.ac.ir*

School of Metallurgy and Materials Engineering, Iran University of Science and Technology (IUST), Narmak, Tehran, Iran

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Abstract: *The influence of melt superheating treatment on the solidification characteristics and microstructure of Al–20%Mg_2Si in-situ composite has been investigated. The results revealed that melt superheating temperature has significant effects on solidification parameters and morphology of primary Mg_2Si particles. Solidification parameters acquired using the cooling curve thermal analysis method, indicate that both nucleation temperature and nucleation undercooling of primary Mg_2Si particles increase by increasing melt superheating temperature, while recalescence undercooling decrease under the same condition. Also, based on the microstructural evaluations, melt superheating treatment can refine primary Mg_2Si particles and alter their morphology from dendritic shape to more spherical shape and the eutectic microstructure of α-Al + Mg_2Si becomes finer and the distance between eutectic layers becomes smaller..*

Keywords: *Al–20%Mg_2Si in-situ composite; melt superheating treatment; cooling curve thermal analysis; microstructure; solidification*

1. INTRODUCTION

Al–Mg_2Si metal matrix composites (MMCs) have the potential to replace the commonly used hyper-eutectic Al-Si alloys due to their improved properties (low density, excellent castability, good wear resistance, and excellent mechanical properties) [1, 2]. The characteristics of Mg_2Si intermetallic compound as reinforcing particles play a major role in superior mechanical properties of this material, which includes low density (1.99 $g.cm^{-3}$), high melting temperature (1085°C), high hardness (4.5×10^9 $N.m^{-2}$), high elastic modulus (120 GPa) and low coefficient of thermal expansion (7.5×10^{-6} K^{-1}) [2-6]. According to Al-Mg_2Si binary phase diagram, Al–Mg_2Si composite containing more than 13.9 wt. % Mg_2Si demonstrates a hypereutectic microstructure [7]. The first phase precipitating from the melt is primary Mg_2Si, and then the rest of the melt solidifies as a binary eutectic structure consisting of Al and Mg_2Si [2, 7]. Mechanical properties of hypereutectic Al-Mg_2Si highly depend on the morphology and size of primary Mg_2Si. In as-cast conditions, primary Mg_2Si has coarse dendritic morphology with sharp edges and needs to be modified to improve mechanical properties [3]. The most popular refinement method is adding a refiner element to the melt. It has been reported that the addition of Bi [8, 9], Sr [3, 9, 10], Sb [9, 11, 12], Mn [13], P [10], and Ce [2] can change coarse dendritic structure of Mg_2Si particles to fine polygonal shapes.

Melt superheating treatment has been reported as an effective technique for Mg_2Si refinement in a few Al and Mg alloys and composites [14-18]. In general, there are two main hypotheses for the grain refining effect of melt superheating treatment. The first theory is called the 'heredity phenomenon'. Based on this theory existing particles in the melt at normal pouring temperatures are too big to serve as nucleation sites. Higher superheating temperatures dissolve these particles and then they precipitate as finer more suitable nuclei [16, 18]. Z. H. Gu et.al [15] reported that reduction of heredity plays an important role in the grain refinement of Mg–1.5Si–1Zn alloy. In the second theory, an increase in undercooling is the reason for the grain refining effect of melt superheating treatment. It means by increasing superheating temperature the distribution of alloying elements will be more homogeneous due to the thermal diffusion which clearly affects the undercooling and the subsequent solidification process [18].

This paper aims to study the effect of melt superheating treatment on microstructure and solidification parameters of Al-20wt% Mg_2Si

composite. For this purpose, the cooling curve thermal analysis (CCTA) technique has been used along with microstructural evaluations. Backerud et al. [19, 20] and S.G. Shabestari et al. [21-24] have used CCTA as a very useful method to investigate the relationship between solidification characteristics and microstructure of different metals and alloys. To date, only a few numbers of research [14] have reported about the investigation on the effect of melt superheating on nucleation and growth of Mg_2Si reinforcement at in-situ composite through thermal analysis, but there is no report on other solidification aspects of this composite. In the present study characteristic temperatures including nucleation and growth temperatures (T_N, T_G) and nucleation and recalescence undercooling (ΔT_N, ΔT_R) for different phases are determined from cooling curves and their first derivative at different melt superheating temperatures. Also, the relation between solidification parameters and microstructure is studied.

2. EXPERIMENTAL PROCEDURES

The Al–20Mg_2Si composite was prepared by melting commercially A413 alloy and high purity (>99.99 wt %) magnesium in a graphite crucible in an electrical resistance furnace. The melt was protected with Magrex* powder as a coveral flux to prevent melt oxidation and gas absorption. (* It is the trademark of Foseco company.) The melt was held for 10 min at 750±5˚C for homogenization, then it was skimmed and poured into a steel permanent mold coated with graphite and preheated to 250˚C to produce Al–20%Mg_2Si ingot. The chemical composition of the investigated composite is listed in table 1.

Table 1. Chemical composition of prepared Al-Mg_2Si composite

Composite	Elements (mass %)				
	Mg	Si	Cu	Fe	Al
Al – 20%Mg_2Si	13.93	9.63	0.12	0.32	Balance

Thermal analysis samples were taken by remelting 200 g of prepared ingot in a graphite crucible and pouring the molten alloy into a preheated cylindrical stainless steel mold (outer diameter 40 mm, height 70 mm, and wall thickness of 1 mm) which a K-type thermocouple had been placed in the center of it at a position of 20 mm from the bottom. To study the influence of superheating, the melt was held at 750, 800, and 850°C for 25 min. before pouring. The temperature-time data were recorded using an analog to digital converter (A/D converter) linked to a computer. The analog to digital converter ADAM- 4018 used in this study has a sensitive 16-bit microprocessor-controlled sigma-delta A/D converter; the response time of 0.02 seconds and having 8-channel analog input module that provides programmable input ranges on all channels. Each test was repeated at least three times, to check the reproducibility and accuracy. The variations of temperature versus time were recorded with the frequency of 10 readings per second. Solidification parameters have been extracted from cooling curves and their first derivative. The cooling rate (C.R) during thermal analysis tests was 0.50±0.05°Cs^{-1} which was calculated from the slope of the cooling curve, $\Delta T/\Delta t$, between the two first reactions (670°C to 620°C).

For microstructural evaluations, samples were sectioned horizontally from the regions near the thermocouple tip. After grinding and polishing the samples, they were etched using 0.5% HF etchant to reveal as-cast microstructure of samples. The morphology and size of primary Mg_2Si particles were analyzed using HUVITZ – HM25 type optical microscope. For each sample, five pictures were taken from the central zone and Mg_2Si particles size, circularity, and aspect ratio were evaluated for each picture using Image J software. Then, the average particle size, circularity, and aspect ratio of primary Mg_2Si intermetallics were determined in each sample. The phases were also characterized through X-ray diffraction XRD D8 Advance Bruker using Xpert High Score software.

3. RESULTS AND DISCUSSION

3.1. Al-Mg_2Si in situ composite; cooling curve and microstructure

The cooling curve and its first derivative curve for Al-20%Mg_2Si composite with melt superheating temperature of 800˚C are given in Fig 1-a. The derivative curve is projected to reveal the characteristic data, such as nucleation, growth, and minimum temperatures (T_N, T_G, and T_{min}) because these temperatures cannot be identified easily from the cooling curve itself. Three slope

alterations on the cooling curve and three well-known peaks on the first derivative curve are noticeable which correspond to three-phase transformations or phase formation during the solidification. These phases are shown in Fig 1-b.

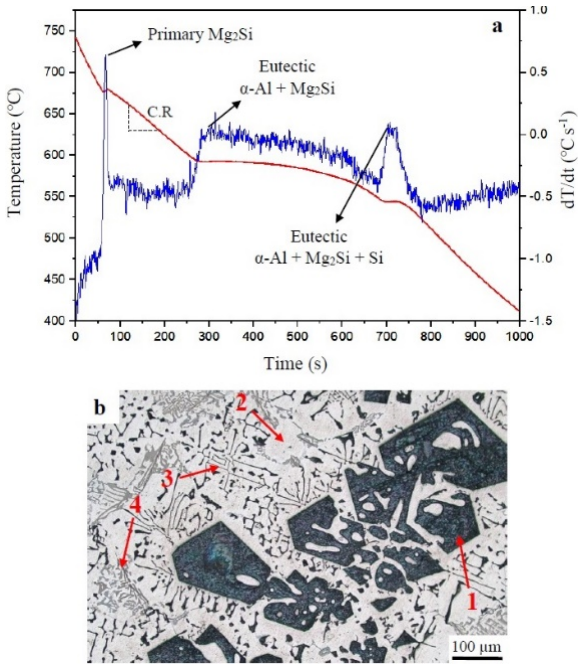

Fig. 1. (a) cooling curve and its first derivative curve (b) microstructure of Al-20% Mg_2Si in situ composite with 800ºC superheating temperature indicated phases: (1) primary Mg_2Si, (2) α-Al, (3) eutectic Mg_2Si, (4) eutectic Si

According to Al-Mg_2Si binary phase diagram [1] (Fig.2), solidification of Al-20%Mg_2Si starts with nucleation of primary Mg_2Si.

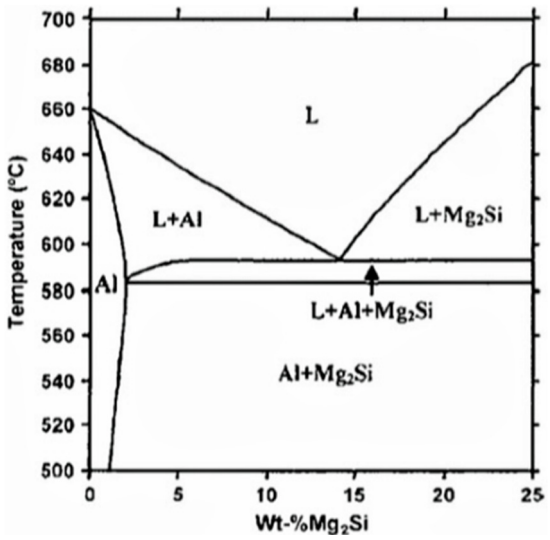

Fig. 2. Al-Mg_2Si binary phase diagram [1]

Therefore, the first peak at 687°C relates to the formation of primary Mg_2Si. The second peak at 600°C and the third peak at 547°C represent binary α-Al + Mg_2Si eutectic and ternary α-Al + Mg_2Si + Si eutectic, respectively. The phases were also analyzed with XRD. They were characterized as α-Al and Mg_2Si phases in the microstructure of the composite (Fig.3). Because of the low concentration of the Si phase, this phase wasn't recognized in the XRD pattern.

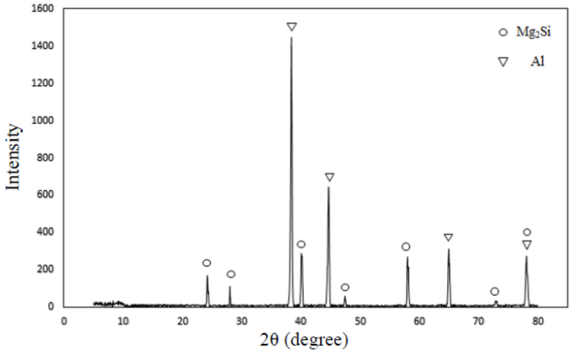

Fig. 3. XRD pattern of as-cast Al-20%Mg_2Si in situ composite

3.2. Effect of melt superheating treatment on cooling curves

The cooling curve of Al-20%Mg_2Si composite with different superheating temperatures is illustrated in Fig 4. Characteristic temperatures for each reaction are extracted from cooling curves and their derivative curves as it is shown in Fig 5 and presented in Table 2. Thermal analysis results in Table 2 reveal that superheating treatment has significant effects on nucleation of primary Mg_2Si particles and binary α-Al + Mg_2Si eutectic.

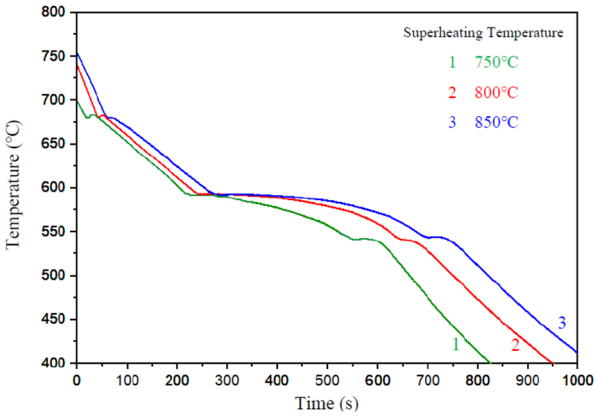

Fig. 4. Effect of superheating temperature on cooling curves

Table 2. Characteristic temperatures determined from cooling curves and their first derivative curves

Superheating Temperature (°C)	Primary Mg_2Si					Eutectic α-Al + Mg_2Si		Eutectic α-Al + Mg_2Si + Si		Solidus temperature (Ts)
	T_N	T_G	T_{Min}	ΔT_N	ΔT_R	T_N	T_G	T_N	T_G	
750	686.0	683.2	678.5	7.5	4.7	599.3	593.7	547.2	540.5	527.2
800	687.5	679.8	676.5	11.0	3.3	601.7	592.6	546.9	540.7	522.5
850	690.3	677.2	676.2	14.1	1.0	602.4	589.5	547.4	541.1	519.9

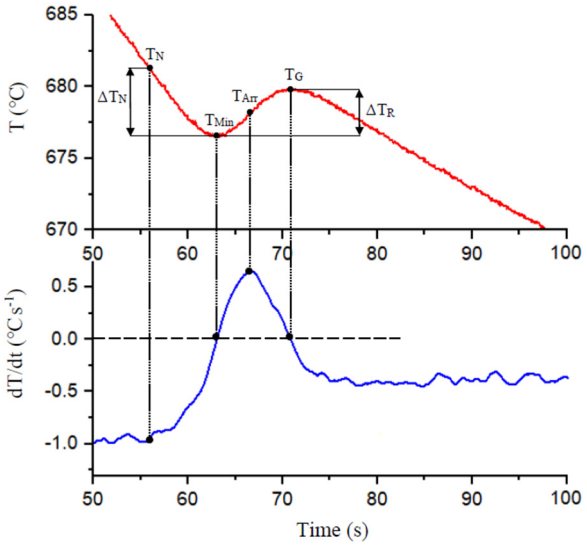

Fig. 5. Characteristic points on cooling curve and its first derivative

3.2.1. Effect of superheating on the nucleation of primary Mg_2Si

Variation of nucleation temperature (T_N), nucleation undercooling ($\Delta T_N = T_N - T_{Min}$), and recalescence undercooling ($\Delta T_R = T_G - T_{Min}$) for primary Mg_2Si particles illustrated in Fig 6. According to this figure, by increasing superheating temperature from 750 °C to 850 °C, T_N and ΔT_N increase from 686.0 °C to 690.3 °C, and 7.5 °C to 14.1 °C, respectively; but ΔT_R decreases from 4.7 °C to 1.0 °C.

Increasing the nucleation temperature of Mg_2Si may be a result of the acceleration of atomic diffusion in the melt by increasing the superheating temperature which provides more potential substrates for Mg_2Si particles to nucleate. Therefore, primary Mg_2Si particles are expected to become finer by melt superheating treatment. In the other hand, with increasing superheating temperature, existing impurities and atomic clusters in the melt decrease. As a result of the reduction in the number of heterogeneous nucleation sites and because the new atomic clusters have a smaller radius than the critical radius of the nuclei, nucleation undercooling of primary Mg_2Si increases.

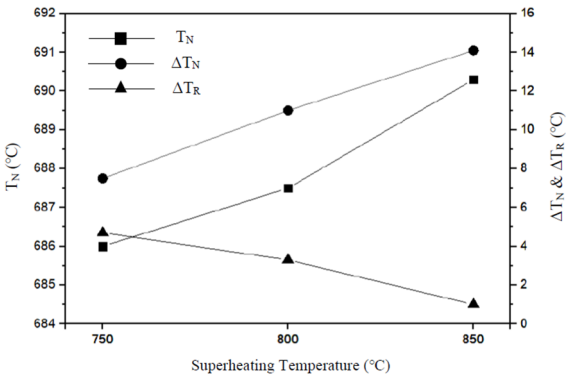

Fig. 6. Effect of superheating temperatures on nucleation temperature (TN), nucleation undercooling (ΔTN) and recalescence undercooling (ΔTR) for primary Mg_2Si particles

Backerud et al. [19] and Shabestari et al. [24] reported that grain refinement in different Al alloys causes to decline of recalescence undercooling until there would be no recalescence undercooling ($\Delta T_R = 0$) when the optimum amount of refiner is added. In the present work, the reduction of ΔT_R can be another sign for the refinement of primary Mg_2Si particles, too. In Table 2 and Fig. 6, the effect of melt superheating on the recalescence undercooling has been presented. As can be seen, the recalescence undercooling has been decreased by increasing superheating temperature. The reason for this phenomenon is that with increasing superheating temperature, existing nuclei in the melt can become more active because of the higher diffusion rate that happens at higher superheating temperatures and can establish the condition of the growth. In this situation, it can achieve the growth temperature with lower recalescence undercooling. Therefore, a decrease in recalescence undercooling can cause the

refinement of primary Mg_2Si.

In addition, solid fraction curves were plotted based on the Newtonian model adopted by Stefanescu et al. and Tamminen [25] and shown in Fig. 7. According to this figure, when superheating temperature increase from 750°C to 850°C, the solid fraction between the two first reactions (nucleation of primary Mg_2Si and binary α-Al + Mg_2Si eutectic) increases significantly.

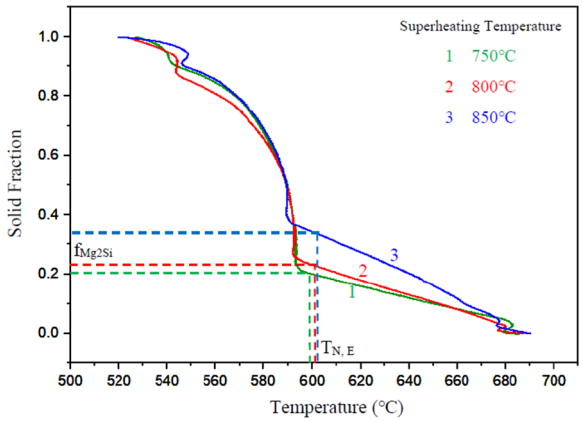

Fig. 7. Effect of superheating temperature on solid fraction curves

Solid fraction of primary Mg_2Si (solid fraction at nucleation temperature of binary eutectic) was extracted from Fig.7. By increasing superheating temperature, the solid fraction of primary Mg_2Si increases from 0.20 to 0.33. It means the fraction of eutectic Mg_2Si has decreased since the amount of Mg and Si in the composition of samples are constant.

3.2.2. *Effect of superheating on the final solidification temperature, solidification range, and total solidification time*

Fig. 8 shows the effect of superheating on the final solidification temperature, solidification range, and total solidification time. As seen from this figure, with increasing superheating temperature, the final solidification temperature decreases, while the solidification range, and total solidification time increase.

In section 3.2.1, it has been reported that the nucleation temperature of primary Mg_2Si increases with superheating. In Fig. 8, it is shown that the final solidification temperature decreases with superheating. Therefore, the solidification range which is the difference between these two parameters will increase with superheating temperature.

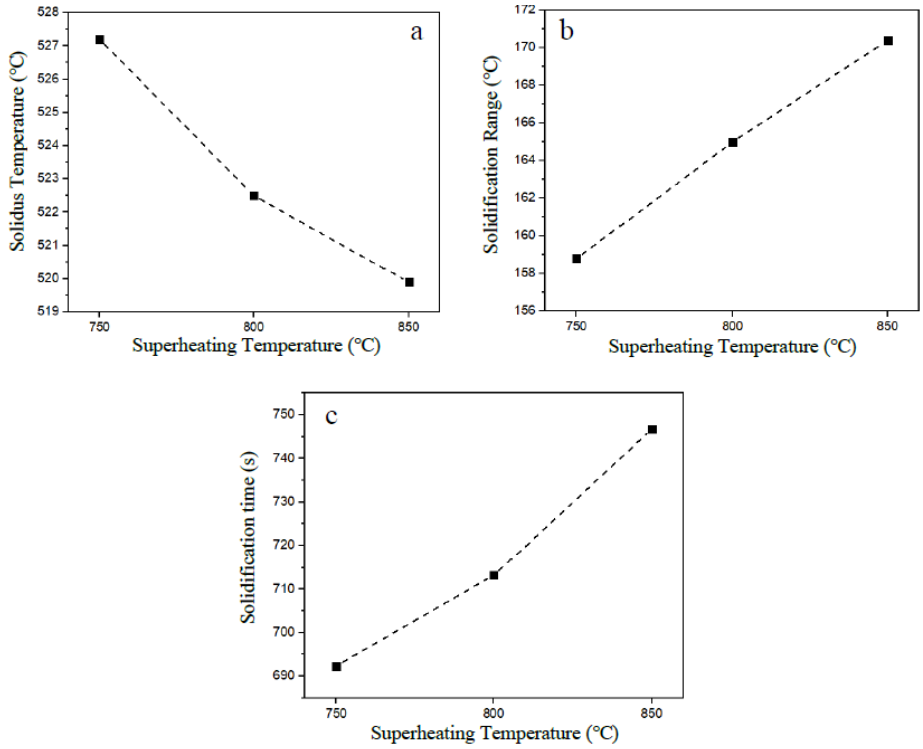

Fig. 8. Effect of superheating on: (a) the final solidification temperature, (b) solidification range, (c) total solidification time.

3.2.3. *Effect of superheating on the eutectic reactions*

Fig. 9 indicates the effect of superheating on the binary eutectic reaction of α-Al + Mg_2Si. It can be observed that with increasing superheating temperature, nucleation temperature of the binary eutectic reaction increases and the growth temperature decreases. It has been proved that primary Mg_2Si particles act as heterogeneous sites for the nucleation of eutectic α-Al [26, 27].

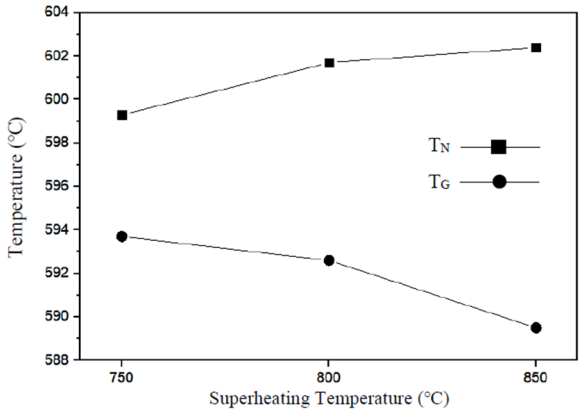

Fig. 9. Effect of superheating on nucleation and growth temperatures of α-Al + Mg_2Si eutectic reaction

In section 3.2.1, it has been explained that primary Mg_2Si particles will be finer and their volume fraction increase by increasing superheating temperature. Therefore, there are more suitable sites for nucleation of eutectic α-Al and it can be the reason for the increase in nucleation temperature of the binary eutectic reaction. On the other hand, because of the higher volume fraction of primary Mg_2Si, the concentration of Si and Mg in the melt would be lower and it probably reduces the growth temperature of this eutectic reaction.

Results of cooling curves obtained from applying melt superheating temperatures of 750ºC, 800ºC, 850ºC indicate that they do not have any effect on ternary eutectics.

3.3. Effect of melt superheating treatment on microstructure

3.3.1. *Effect of superheating on primary Mg_2Si*

Fig. 10 shows the changes of morphology and size of primary Mg_2Si particulate in Al-20% Mg_2Si in situ composite as a result of various superheating temperatures.

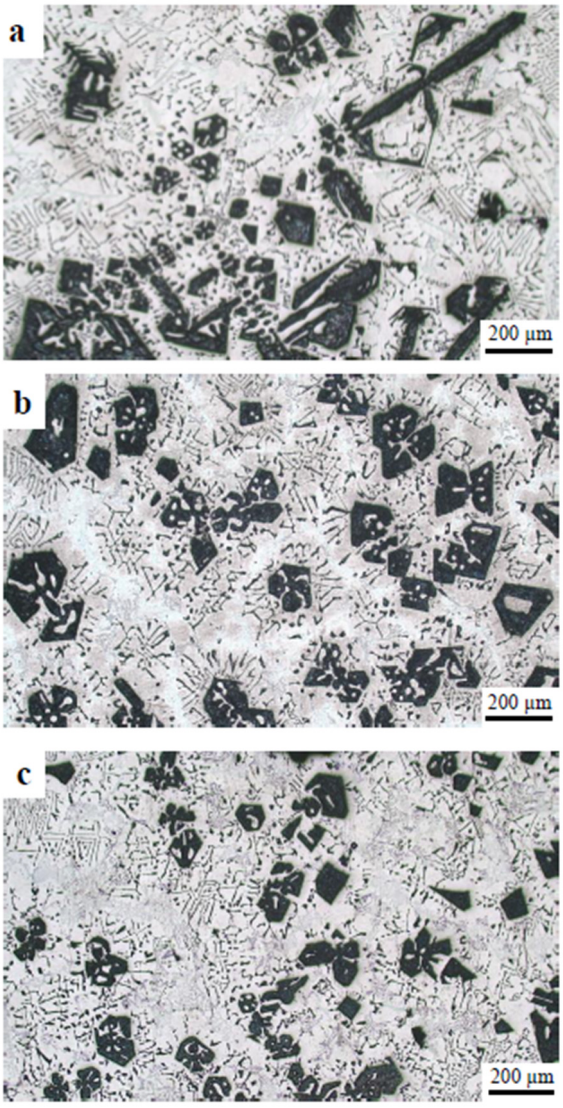

Fig. 10. Primary Mg_2Si microstructure of Al-20% Mg_2Si composite by applying melts superheating, (a) 750ºC, (b) 800ºC, (c) 850ºC

Obviously, higher melt superheating temperature leads to better refinement of primary Mg_2Si particles. Microstructural analysis indicates that by increasing superheating temperature from 750ºC to 850ºC, Mg_2Si particles size decrease from 134.5 µm to 113.3 µm (Fig. 11). This is in good agreement with thermal analysis results. Reduction of heredity of the molten composite by melt superheating treatment can explain this phenomenon [14-16]. It means that all solid particles and impurities would dissolve completely in higher superheat temperatures and re-nucleate as finer particles. Since these particles might be responsible for primary Mg_2Si precipitation, finer particles lead to finer Mg2Si particles. Also, an increase in nucleation

undercooling and a decrease in recalescence undercooling, which has been discussed in section 3.2.1, is another explanation for the refinement of primary Mg_2Si particles.

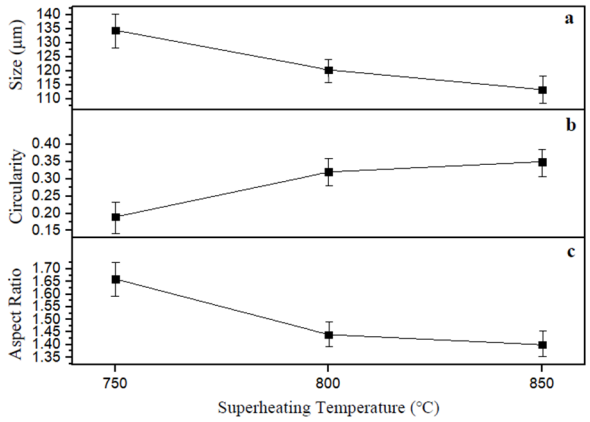

Fig. 11. Variation of (a) size, (b) circularity and (c) aspect ratio of primary Mg_2Si particles versus melt superheating temperature

In addition to the reduction of Mg_2Si particles size, the dendritic shape of the particles has changed to a more spherical shape. The influence of superheating temperature on aspect ratio and circularity of primary Mg_2Si particles are shown in Fig. 11. The aspect ratio defines as the ratio of the maximum length to the minimum length of the primary Mg_2Si particle.

It can be seen that the aspect ratio decreases from 1.66 to 1.40 when superheating temperature changes from 750°C to 850°C; while circularity increase from 0.19 to 0.35 at the same condition. In another word, superheating treatment is caused primary Mg_2Si particles to become more spherical and finer.

3.3.2. *Effect of superheating on eutectic microstructure*

Fig. 12 indicates the effect of superheating on the eutectic microstructure of Al-20% Mg_2Si composite. The morphology of the α-Al + Mg_2Si eutectic does not change; but the eutectic microstructure becomes finer and the distance between eutectic layers becomes smaller.

4. CONCLUSION

Effect of various melt superheating temperatures (750, 800, and 850°C) on Al-20%Mg_2Si in-situ composite was investigated through thermal analysis technique. Microstructure and

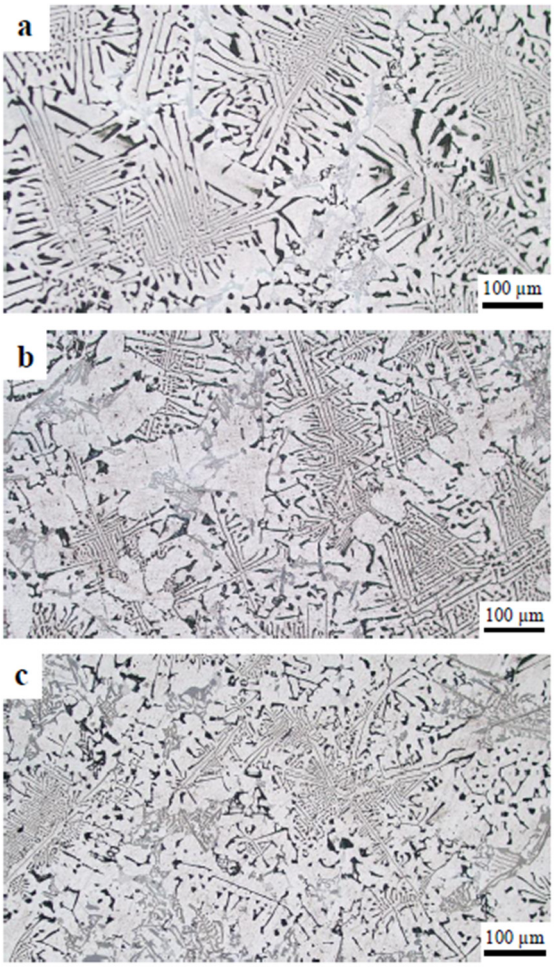

Fig. 12. Eutectic microstructure of Al-20%Mg_2Si composite by applying melts superheating, (a) 750°C, (b) 800°C, (c) 850°C

solidification parameters of the composite were studied using cooling curves. Nucleation and growth of Mg_2Si intermetallic as reinforcement phase at in-situ composite was studied.

Microstructural analysis indicated that by increasing superheating temperature from 750°C to 850°C, Mg_2Si particles size decrease from 134.5 μm to 113.3 μm, and the dendritic shape of the particles has changed to a more spherical shape. Also, the eutectic microstructure of α-Al + Mg_2Si becomes finer and the distance between eutectic layers becomes smaller.

When melt superheating temperature was 850°C, primary Mg_2Si particles had the highest nucleation temperature (TN) and the lowest recalescence undercooling (ΔTR). These two parameters are known as the main sign of grain refinement in various alloys. Since microstructural evaluations revealed that increasing superheating temperature leads to finer

and more spherical primary Mg_2Si particles, TN and ΔTR can be used as the sign of Mg_2Si particles refinement in Al-Mg_2Si composites. Therefore, the cooling curve thermal analysis technique can be used as an online method to control the refinement of Mg_2Si reinforcement particles during the casting process of Al-Mg_2Si composites.

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