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ORIGINAL ARTICLE

Influence of Superheating Melt Treatment on Microstructure of Gd-Modified Al-15wt.% Mg_2Si In-Situ Composite

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ABSTRACT – The influence of melt superheating treatment with different superheating temperatures (750, 800, 850, and 900°C) on the microstructure of Al-15wt. % Mg_2Si composites before and after addition of Gd (1.0 wt.%) were studied. Microstructure characterisation was carried out via optical microscopy (OM) and x-ray diffraction (XRD).The results showed that in the unmodified composite, when the temperature of superheating raised from 750°C to 900°C, the primary Mg_2Si experienced a decrease of the average grain size from 40 to about 32 μm, while, in the Gd-modified composite, the particle size was refined considerably from 27 to 13 μm by increasing the temperature from 750°C to 850°C. Furthermore, with a further increase in temperature to 900°C, the particle size slightly increased to about 15 μm which might be due to the burning loss of Gd in the melt. Nevertheless, for both unmodified and Gd-modified composites, the superheating temperature exhibited minor influence on the Mg_2Si crystals morphologies. It is believed that compiling of Gd addition and superheating melt treatment is efficient to improve the composite properties that could encounter the application requirements.

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KEYWORDS
Aluminum matrix composite (AMC); Mg_2Si, Superheating temperature; Modification

INTRODUCTION

Recently, Al-Mg_2Si in-situ composite received considerable interest in the automotive industry, due to its desirable properties which is due to the existence of the Mg_2Si phase [1-5]. However, during fabrication of the composite under low solidification rate (e.g. gravity casting), the formation of coarse and irregular morphology of primary and eutectic Mg_2Si phases in the Al-Mg-Si melt worsens the mechanical properties of Al-Mg_2Si composite which restricts its development and applications[1,2]. Therefore, it is essential to alter the structure of primary and eutectic Mg_2Si phase with the purpose of the mechanical properties of the subjected composite [1,2].

Besides modification, it has been proposed from different perspectives that for the purpose of refinement, superheating melt treatment is a modest and efficient method [6-13]. Nordin et al. [14] reported that at a superheated temperature of 950 °C and holding time of 15 minutes, the primary Mg_2Si with skeleton structure experienced size decrement of its fine polygonal structure. Furthermore, Saffari et al. [15] claimed that by increasing the superheating melt temperature, the primary Mg_2Si particles size reduced compared to the Mg_2Si particles in samples produced by gravity casting and vibrating cooling slope (VCS). Nevertheless, the exact refinement mechanism of superheating treatment is still blurry; in addition, there are some inconsistencies in its description. According to the first researcher's findings, the particles are not able to act as nuclei at normal pouring temperature as they are not small enough, while they may dissolve at high temperatures and then the fine particles are reformed or precipitated which are appropriate for nucleation of small particles [9,10]. Another explanation attributes to particle refinement is the heredity phenomenon in the materials in which during the process of melting, clusters and solid particles that survived, may have influences on the re-solidified structures [7,8]. For example, Chen et al. [8] claimed that the modification of eutectic Si in Al-7Si-0.55Mg melt by superheating treatment is by decreasing the Si-Si clusters heredity and altering its growth manner.

Furthermore, Li et al. [13] reported that the heredity of Al-16Si alloy decreased at high superheating temperature (1050 °C). Increasing in undercooling by superheating is another explanation responsible for particle refinement. In fact, by increasing the superheating temperature, the thermal diffusion leads to a more uniform distribution of alloying elements, which obviously influence the undercooling and consequent solidification process[12,14]. For instance, Yin et al. [16] found that at elevated superheating temperature (over 1873 K); the M963 alloy was refined due to high undercooling in the melt.

In contrast to the explanations mentioned above, it is believed that at high superheating temperature establishment of new heterogeneous nucleus results in the refinement of the particles [9,17]. In another study [9], it was proposed that in Mg-Al-Mn alloys, superheating melt treatment can generate ε-Al-Mn phase at high temperature, which moderately form into metastable τ-Al-Mn phase, and can turn into an efficient non-homogeneous nucleation substrate.

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Although there are several studies about the role of superheating treatment on the Mg_2Si particles refinement of in Al or Mg composite reinforced with Mg_2Si, limitations exist on the influence of different temperatures of superheating on refinement/modification of Mg_2Si phases in RE-modified Al-Mg_2Si composites. Therefore, in the current work, superheating process on Al-Mg_2Si composite melts without and with Gd addition as well as their effects on the microstructure features are examined experimentally.

MATERIALS AND METHOD

To produce Al-15%Mg_2Si composite ingot, pure industrial grade Al, Si and Mg were used. In which, with the aid of a resistance furnace, Al and Si melts were achieved at 800 °C, and once temperature decreased to 750 °C, the melt was inserted with pure Mg. After homogenisation and removing of the dross, the melt was poured into a metal mould. The compositions of the elements exist in Al-15wt.% Mg_2Si composite is demonstrated in Table 1. After cutting off the composite ingot into small pieces, an electric resistance furnace was used to melt 100g of the Al-15wt.% Mg_2Si ingot. After holding the melt for 5 min in the designed temperature (750 °C), Al-10Gd master alloy was added to the melts to achieve the designed Al-15wt. % Mg_2Si-1.0wt. % Gd. After manually stirring the melt with a stainless steel impeller, it was held for 15 min at the pre-set temperature of 750 °C and then poured into a preheated steel mould (100 °C) to fabricate cylindrical samples with 30mm diameter. The same procedure was repeated for temperatures of 800, 850 and 900 °C. The standard metallographic procedure of the samples was initiated with the etching of the samples at room temperature using 0.5% HF (volume fraction) for about 5s. Optical microscopy (OM) (Nikon-MICROPHOT-FXL) and x-ray diffraction (XRD) (Siemens D500 x-ray diffractometer with a Cu Kα line) were used to examine the microstructure characteristics of the specimens. Furthermore, to estimate the particle size, the intercept method was conducted on the microstructure with low magnification, and the calculation was carried out on at least 70 particles.

Table 1. The composition of the elements exists in Al-15%Mg_2Si (wt. %) ingot.

Element	Weight (%)
Al	84.60
Mg	8.50
Si	6.70
Fe	0.12
V	0.02
Cr	0.01
Ni	0.01
Mn	0.01

RESULTS AND DISCUSSION

Analysis of X-ray Diffraction (XRD)

Figure 1 illustrates the XRD pattern of unmodified and 1.0wt. % Gd modified Al-15wt. % Mg_2Si composites processed at 750 °C and 900 °C. As observed, all composites consisted of Al and Mg_2Si phases as expected, in addition to Al_2Si_2Gd and AlSiGd owning to the Gd intermetallic compounds (IMCs) formation in the Gd-treated samples. Therefore, it indicates that the increase in temperature from 750-900°C had no effect on the phase compositions of Al-15wt. % Mg_2Si composites.

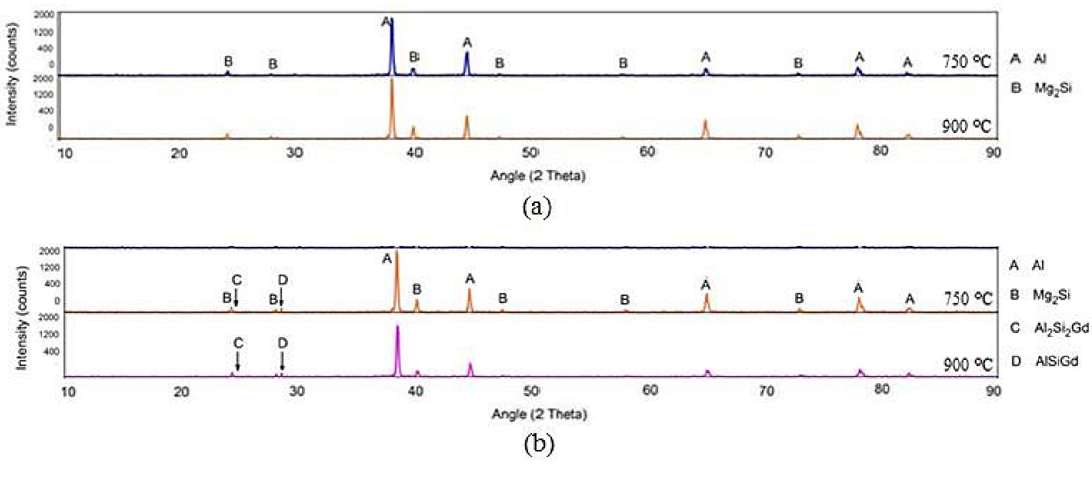


Figure 1. XRD patterns of Al-15wt. % Mg_2Si composites in (a) untreated and (b) Gd-treated conditions at 750 °C and 900 °C temperatures

Analysis of Microstructure

Figure 2 and 3 depict the effect of superheating temperatures on the microstructure of the unmodified and Gd-modified Al-15wt. %Mg_2Si composites, respectively. As seen in Figure 2(a) to 2(d), with increase of temperature, the grain size of the primary Mg_2Si particles decreased in the unmodified composite. Furthermore, the significant changes in the size of the primary Mg_2Si particles are as depicted in Figure 3(a) to 3(d).

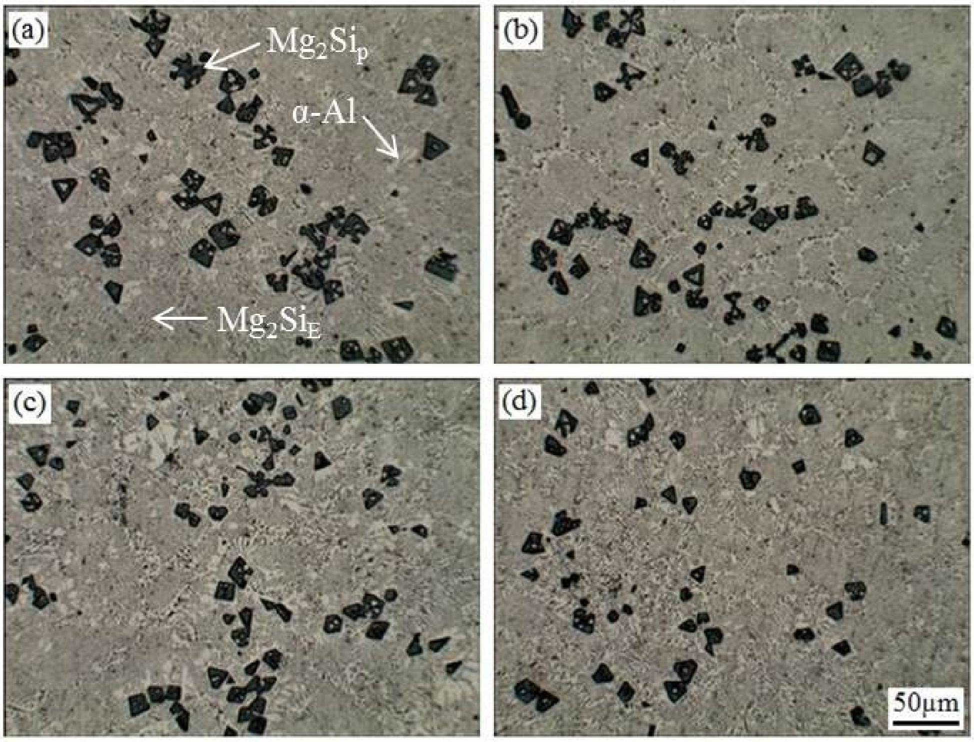


Figure 2. OM micrographs of untreated Al-15wt. % Mg_2Si composites at different temperatures of superheating: (a) 750 °C; (b) 800 °C; (c) 850 °C; (d) 900 °C

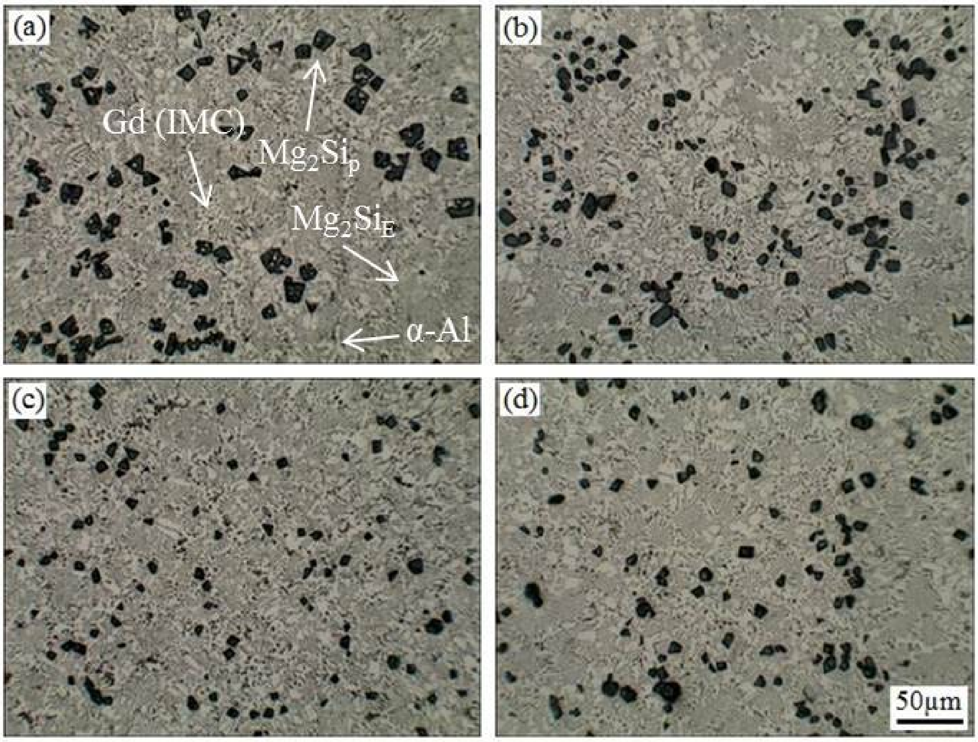
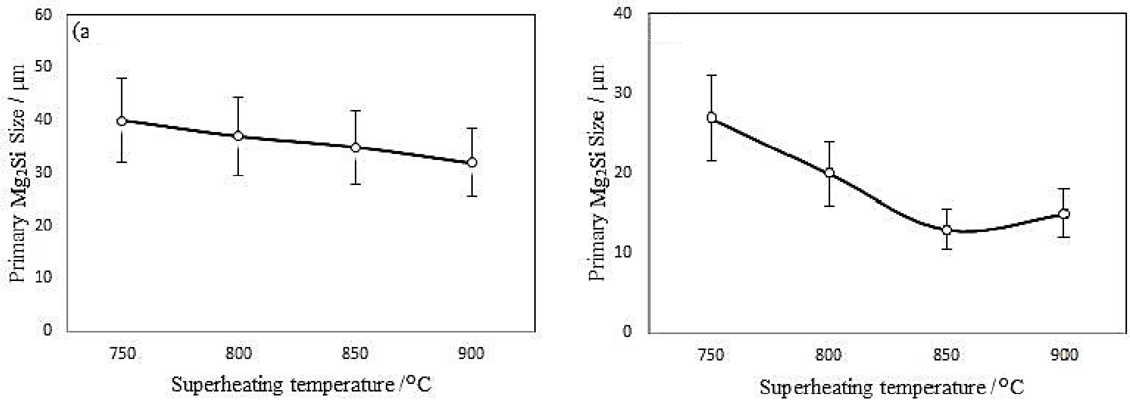


Figure 3. OM micrographs of Gd-treated Al-15wt. % Mg_2Si composites at different temperatures of superheating: (a) 750 °C, (b) 800 °C, (c) 850 °C and (d) 900 °C

Figure 4(a) and 4(b) illustrate the changes in the size of primary Mg_2Si particle in both unmodified and Gd-modified composites as a function of melt superheating temperature, respectively. When the temperature increased from 750°C to

900°C, the coarse dendritic primary Mg_2Si particles was refined considerably in the unmodified composite, from larger than 40 µm to 32 µm or less (as in Figure 4(a)). Simultaneously, the density of Mg_2Si particles increased as a result of size reduction and showed that in the matrix of Al, the particles are distributed uniformly as shown in Figure 2(a) to 2(d). As seen in Figure 4(b), a considerable reduction can be detected in Mg_2Si particles size from about 27 µm to 13 µm in the modified composite with an increase in temperature from 750 °C to 850 °C. With further increase in temperature to 900 °C, the modified Mg_2Si particles showed a reverse trend, and the particles became coarser with the increased size of about 15 µm; refer to Figure 4 (b). Thus, the temperature of 850 °C is considered as the proper superheating temperature for the goal of primary Mg_2Si particles refinement in Al-15wt. % Mg_2Si composites.



(a) (b)

Figure 4. Influence of temperature of superheating on the size of primary Mg_2Si particles in Al-15wt. % Mg_2Si composites in (a) untreated; (b) Gd-treated.

From the comparison between Figure 2 and Figure 3, it can be taken into account that the primary Mg_2Si crystal altered from a coarse dendritic structure to a fine truncated octahedral shape. Moreover, the eutectic Mg_2Si crystal transformed from Chinese script shape to fibre-like structure. The results show that the Gd addition can efficiently modify the Mg_2Si particles, in which the corresponding modification mechanism is discussed later. The magnified images of Mg_2Si particles in unmodified and Gd-modified Al-15wt. % Mg_2Si composites at different superheating temperatures of 750°C and 900°C are illustrated in Figure 5 and 6, respectively.

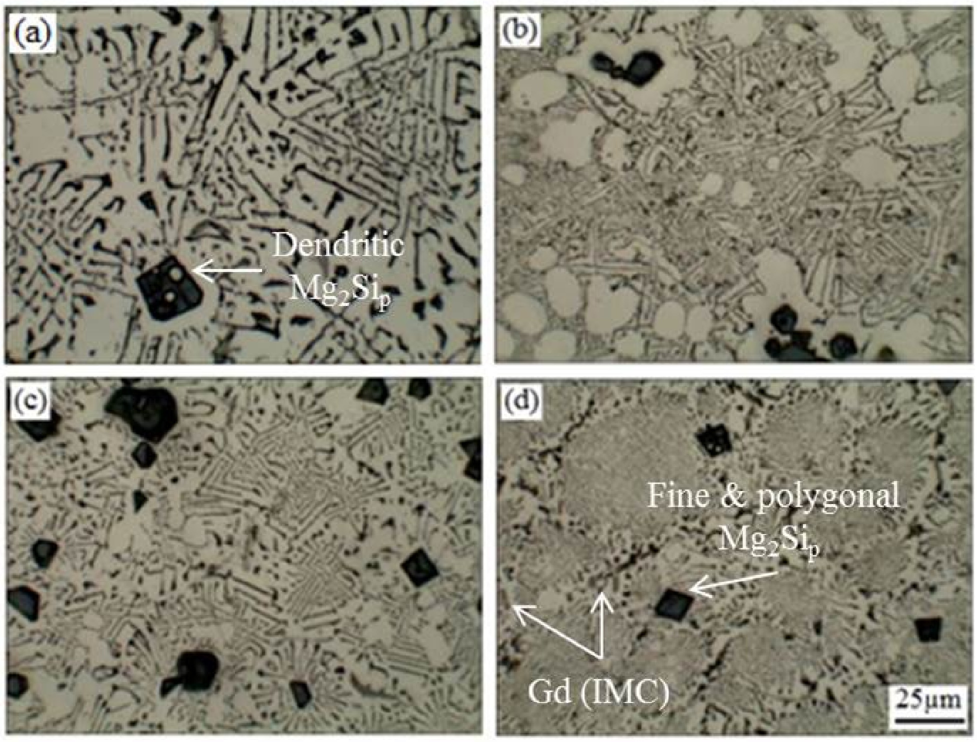


Figure 5. Magnified structures of primary Mg_2Si in Al-15wt. % Mg_2Si composites in untreated and Gd-treated conditions in at various temperatures of superheating: (a) 750 °C (untreated); (b) 900 °C (untreated); (c) 750 °C (treated); (d) 900 °C (treated)

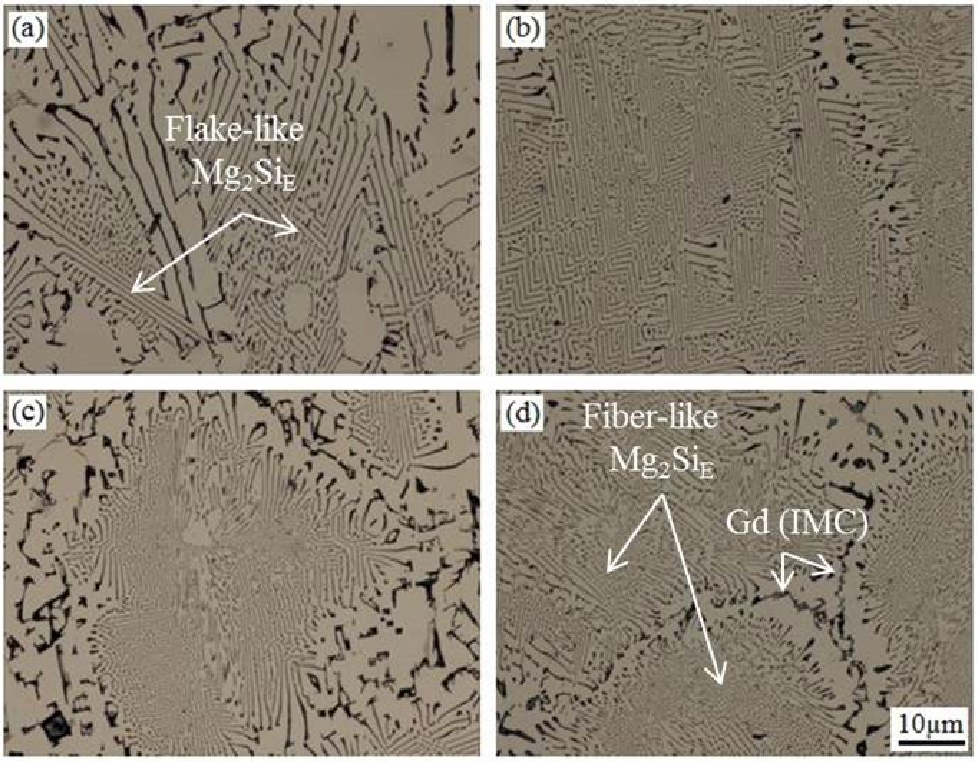


Figure 6. Magnified structures of eutectic Mg_2Si in Al-15wt. % Mg_2Si composites in untreated and Gd-treated conditions in at various temperatures of superheating: (a) 750 °C (untreated); (b) 900 °C (untreated); (c) 750 °C (treated); (d) 900 °C (treated)

Mechanism of Particle Refinement/Modification

The reduction of heredity is one of the explanations for particle refinement. In Al-Si-Mg-Cu, morphology transformations of the primary Mg_2Si particles after superheating melt treatment due to alteration of the initial structure and melt heredity, as proposed by QIN et al. [12]. Furthermore, the decrease of heredity has a significant influence in Mg_2Si particles refinement in Mg-1.5Si-1Zn alloy during superheating melt treatment, as proposed in another study [2]. In the present study, it is proposed that in the unmodified Al-15wt. % Mg_2Si composite, refinement of Mg_2Si phases (both primary and eutectic) after superheating treatment is because of the reduction in heredity. It is believed that in low superheating temperature (e.g. 750ºC), there are a number of solid spots and somewhat atom clusters with large movements which have similar features to the ingot in the melt because of heredity. The nucleation occurs due to the existence of solid spots and large clusters; thus, during subsequent solidification procedure, the particles grow coarser easily. When the superheating temperature is increased, the atom clusters with large moving feature and solid particles dissolve gradually and form into fine clusters which are suitable for small particles nucleation. For Gd-modified Al-15 wt. % Mg_2Si composite, the mechanism of refinement of Mg_2Si particles is due to the probable descriptions as follows:

i. For the refinement of Mg_2Si particles in Gd-modified composite, the decrease of heredity plays a significant role.
ii. The existence of Gd addition in melt leads to refinement and modification of the Mg_2Si particles due to the presence of Al_3Gd phase as non-homogeneous nucleation substrates for primary Mg_2Si particles and poisoning effect of Gd element, as well as restricted growth mechanism as a result of presence of Gd rich intermetallics (AlSiGd, Al_2Si_2Gd) in the composite structure (of Figure 1), which promotes particle size reduction [18]. Nonetheless, the burning loss of the Gd in the melt occurred at elevated superheating temperature (i.e. 900 ºC), which led to a minor increase in the particle size; refer to Figure 3(d) and 4(b).

Hardness Properties

The influence of the superheating process on the hardness property of Al-15%Mg_2Si composites with and without Gd addition is illustrated in Figure 7. It can be observed that the hardness of both unmodified and Gd-modified composites increased when the temperature of superheating is increased. However, in the Gd-modified composites, further increase of the temperature to 900 °C resulted in the decrease of the hardness value, which is due to the reduction of the modification effect of Gd on the Mg_2Si particles, owing to burning of the Gd element at high temperature (900 °C). In fact, the formation and Mg_2Si particles distribution in the matrix of the composites affect the hardness of the fabricated materials [19]. The hardness of the composites was enhanced when the particles are distributed homogeneously in the matrix, as harder ceramic particles can resist the stress [20,21]. As depicted in Figure 7, higher superheat temperature contributed to better particles distribution, which affected the hardness of the composites. When the superheat temperature increased from 750 °C to 900 °C, the hardness values of the unmodified composites increased from 54.98 HV to 65.30HV.

In addition, the hardness value of Gd-modified composites increased from 67.36 HV to 73.17 HV with increasing the temperature of superheating from 750 °C to 850 °C.

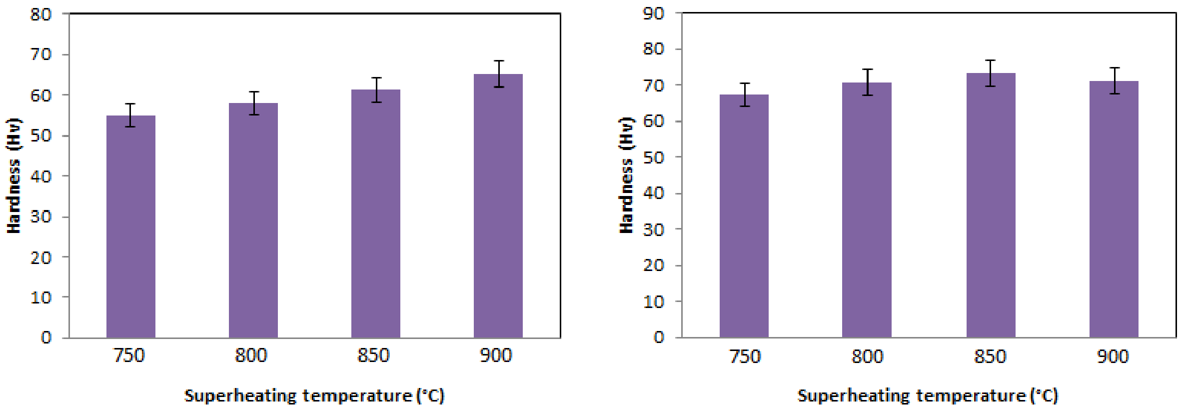


Figure 7. Hardness values as a result of different temperatures of superheating in (a) untreated and (b) Gd-treated Al-15%Mg_2Si composites.

CONCLUSION

The influence of the superheating melt treatment on microstructure and hardness property of Al-15%Mg_2Si in-situ composites in unmodified and 1.0 wt.% Gd-modified conditions was investigated. The concluding observations are as follow:

i. To obtain a refined Mg_2Si particles in Al-Mg_2Si composites, superheating melt treatment can be a good option, in which in the unmodified Al-15wt. % Mg_2Si composite, the coarse Mg_2Si particles was refined progressively by increasing the temperature of superheating from 750 °C to 900 °C. Likewise, in the Gd-modified composites, the primary Mg_2Si particles refined considerably when the temperature increased from 750 °C to 850 °C.
ii. Furthermore, with increasing the temperature to 900 °C, the particle size increased slightly, which can be as a result of a loss of Gd in the melt due to burning. In higher superheat temperature, the heredity of the composite melt decreased, which led to the re-melting of all solid particles, impurities, and finer particles to be re-nucleated. These fine particles served as non-homogeneous nucleation substrate for precipitation of Mg_2Si particles; therefore, little undercooling was required, and as a result, the fine Mg_2Si particles were formed.
iii. The primary and eutectic Mg_2Si morphologies were marginally affected by superheating temperature in unmodified and Gd-modified composites. However, refinement/modification of Mg_2Si particles depicted the enhancement in hardness value of the composites.

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REFERENCES

[1] Gu Z, Wang H, Zheng N, Zha M, Jiang L, Wang W, Jiang QC. Effect of melt superheating treatment on the cast microstructure of Mg–1.5 Si–1Zn alloy. Journal of Materials Science. 2008;43(3):980-984.

[2] Wang H, Jiang Q, Ma B, Wang Y, Wang J.G, Li J.B. Modification of Mg_2Si in Mg–Si alloys with K_2TiF_6, KBF_4 and KBF_4+ K_2TiF_6. Journal of Alloys and Compounds. 2005;387(1-2):105-108.

[3] Ghandvar H, Idris MH, Ahmad N. Effect of hot extrusion on microstructural evolution and tensile properties of Al-15% Mg_2Si-xGd in-situ composites. Journal of Alloys and Compounds. 2018;751:370-390.

[4] Czerwinski F. The generation of Mg–Al–Zn alloys by semisolid state mixing of particulate precursors. Acta Materialia. 2004;52(17):5057-5069.

[5] Aaron H, Idris M, Ang B, Nanda IP, Arafat A. Effect of casting and mould cooling temperatures on the warping phenomenon of aluminium at high pressure die-casting. Prosiding CELSciTech. 2018; 3:61-61.

[6] Haque M, Ismail AF. Effect of superheating temperatures on microstructure and properties of strontium modified aluminium–silicon eutectic alloy. Journal of Materials Processing Technology. 2005;162:312-316.

[7] Bian XF, Wang WM, Qin JY. Structures of liquid Al-Si alloy modified by Sr. Materials Science Forum 2000; 331-337: 349-354.

[8] Zhong-wei C, Wan-qi J, Rui-jie Z. Superheat treatment of Al–7Si–0.55 Mg alloy melt. Materials Letters 2005;59(17):2183-2185.

[9] Qiu D, Zhang M-X, Taylor J, Fu HM, Kelly PM. A novel approach to the mechanism for the grain refining effect of melt superheating of Mg–Al alloys. Acta Materialia 2007;55(6):1863-1871.

[10] Lee Y, Dahle A, StJohn D. The role of solute in grain refinement of magnesium. Metallurgical and Materials Transactions A 2000;31(11):2895-2906.

[11] Cao P, Qian M, StJohn DH. Mechanism for grain refinement of magnesium alloys by superheating. Scripta Materialia 2007;56(7):633-636.

[12] Qin Q, Zhao Y, Liang Y, Zhou W. Effects of melt superheating treatment on microstructure of Mg_2Si/Al–Si–Cu composite. Journal of Alloys and Compounds 2005;399(1-2):106-109.

[13] Li P, Nikitin V, Kandalova E, Nikitin KV. Effect of melt overheating, cooling and solidification rates on Al–16wt.% Si alloy structure. Materials Science and Engineering: A 2002;332(1-2):371-374.

[14] Nordin NA, Abubakar T, Hamzah E, Farahany S, Ourdjini A. Effect of superheating melt treatment on Mg2Si particulate reinforced in Al-Mg_2Si-Cu in situ composite. Procedia Engineering 2017;184:595-603.

[15] Saffari S, Akhlaghi F. Influence of melt superheating on microstructure evolution in Al− Mg_2Si composites fabricated by gravity casting and vibrating cooling slope methods. In: Proceeding of Iran International Aluminum Conference (IIAC2014). Tehran, Iran; 2014.

[16] Yin F, Sun X, Li J, Guan HR, Hu ZQ. Effects of melt treatment on the cast structure of M963 superalloy. Scripta Materialia. 2003;48(4):425-429.

[17] Cao P, Qian M, StJohn DH. Effect of iron on grain refinement of high-purity Mg–Al alloys. Scripta Materialia. 2004;51(2):125-129.

[18] Ghandvar H, Idris MH, Ahmad N, Emamy M. Effect of gadolinium addition on microstructural evolution and solidification characteristics of Al-15% Mg_2Si in-situ composite. Materials Characterisation. 2018;135:57-70.

[19] Shankar G, Kini A, Shettar M, Hiremath P. Aging kinetics and microstructural features of Al6061-SiC+ B4C stir cast hybrid composites. International Journal of Automotive and Mechanical Engineering. 2019;16(4):7211-7224.

[20] Latif NA, Sajuri Z, Syarif J. Effect of tensile strain rates on flow stress for extruded AZ31 and AZ61 magnesium alloys. International Journal of Automotive and Mechanical Engineering. 2017;14:3812-3823.

[21] Rajesh R, Sharma S, Gowrishankar M. Influence of solutionising and aging treatments on mechanical behavior of stir-cast eutectoid steel powder reinforced Al 7075 metal matrix composites. International Journal of Automotive and Mechanical Engineering. 2018;15(3):5583-5591.