International Scholarly Research Network
ISRN Metallurgy
Volume 2012, Article ID 631096, 7 pages
doi:10.5402/2012/631096

Research Article

Effect of Isothermal Holding on Semisolid Microstructure of Al–Mg_2Si Composites

A. Malekan,[1] M. Emamy,[1] J. Rassizadehghani,[1] and M. Malekan[2]

[1] *School of Metallurgy and Materials Engineering, University of Tehran, P.O. Box 14395-731, Tehran, Iran*
[2] *Center of Excellence for Advanced Materials and Processing (CEAMP), School of Metallurgy and Materials Engineering, Iran University of Science and Technology (IUST), Narmak, Tehran 16846-13114, Iran*

Correspondence should be addressed to M. Malekan, malekan@iust.ac.ir

Received 5 February 2012; Accepted 3 April 2012

Academic Editors: A. Chrysanthou, C.-G. Kang, D. V. Louzguine-Luzgin, G. Purcek, J. M. Rodriguez-Ibabe, and A. Squillace

Effect of heat treatment and isothermal holding has been investigated on the microstructure and degree of globularity of Al–Mg_2Si composites. Different contents of reinforcement, 15, 20, and 25% have been used in this study. Isothermal holding experiments were conducted at 585 °C for 140 min. Results showed that, upon heat treatment, grain size of dendrites was reduced while the degree of nodularity was increased. Results of nodularity were obtained using an image-analyzing software which gives the distribution of radius of curvature for different phases of particles. According to the results, in contrast to Al–15 and 25% Mg_2Si, isothermal holding significantly influenced the microstructure of Al–20% Mg_2Si composites. Two companion mechanisms have been proposed for the generation of globular grains. SEM investigations were also employed to confirm the optical observations.

1. Introduction

Superior mechanical properties, as well as increased possibility to control the production process have been the main challenge with which manufacturing of engineering materials is faced. Semisolid processing is claimed to be one of the best approaches which can fulfill such properties. The aim of the semisolid processing is to achieve a fine globular structure. Thus, it is clear that controlling the microstructure of semisolid material is of great importance [1–3]. Possessing a fine globular (nondendritic) microstructure which can be obtained through semisolid-forming processes yields superior advantages over conventional casting and solid-forming methods among which one can mention high quality components capable of full-heat treatment to maximize properties, reduction of macrosegregation, solidification shrinkage, and forming temperature [1–4].

Different methods are classified in semisolid-processing techniques such as magnetohydrodynamic (MHD) stirring [7], spray forming [8], strain-induced melt-activated (SIMA)/recrystallization and partial melting (RAP) [9, 10], and liquidus/near-liquidus casting [11]. The key feature that permits the shaping of alloys in the semisolid state is the absence of dendrites [1, 4]. Hence, partial remelting and isothermal holding recently have been widely used specially for Al–Mg_2Si composites which are also the subject of the current study. Partial remelting and isothermal holding provide a minimum total interfacial energy which is a key factor among composite materials [1, 12].

The Mg_2Si-reinforced Al composites are great potential as automobile brake disc material because the Mg_2Si intermetallic compound exhibits a high melting temperature of 1085°C, low density of $1.99 \times 10^3\ \mathrm{kgm^{-3}}$, high hardness of $4.5 \times 10^9\ \mathrm{Nm^{-2}}$, a low-thermal expansion coefficient (TEC) of $7.5 \times 10^{-6}\ \mathrm{K^{-1}}$, and a reasonably high elastic modulus of 120 GPa [13–16]. However, the primary Mg_2Si phases in normal as-cast Al/Mg_2Si composites are usually very coarse which in turn leads to poor mechanical properties. Therefore, composites with predominantly coarse primary Mg_2Si crystals must be modified to ensure adequate mechanical strength and ductility [1–3, 12, 13].

Current study investigates the effect of isothermal holding in ternary zone (Figure 1) at a temperature slightly higher than that of solidus on the microstructure of the Al–Mg_2Si in

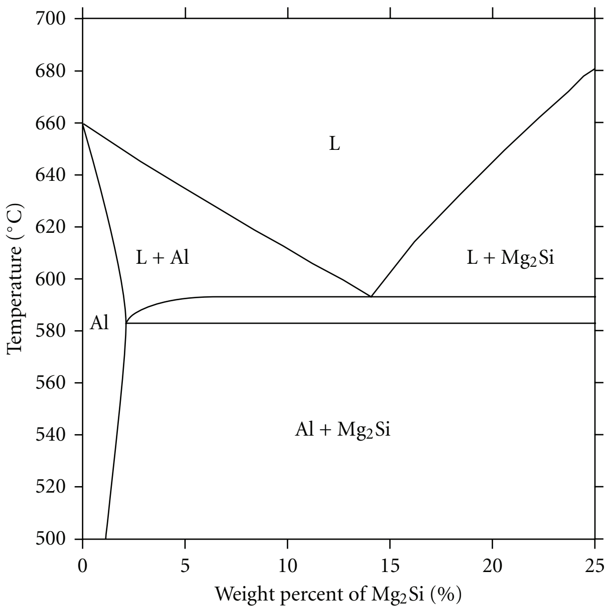


Figure 1: Calculated equilibrium phase diagram of Al–Mg_2Si pseudobinary section. The pseudoeutectic point is at Al–13.9 wt.% Mg_2Si, the temperature range of the ternary region at this composition is between 594 and 583.5°C, and the solubility of Mg_2Si in Al at 583.5°C is 1.91 wt.% [2, 5, 6].

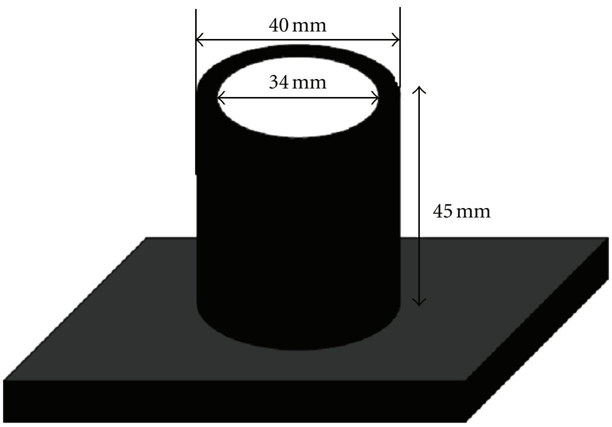


Figure 2: Schematic drawing of cylindrical cast iron mould.

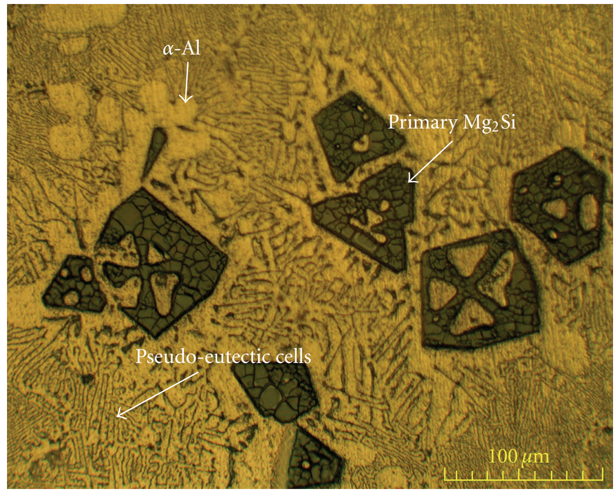


Figure 3: As-cast microstructure of Al–20% Mg_2Si composite.

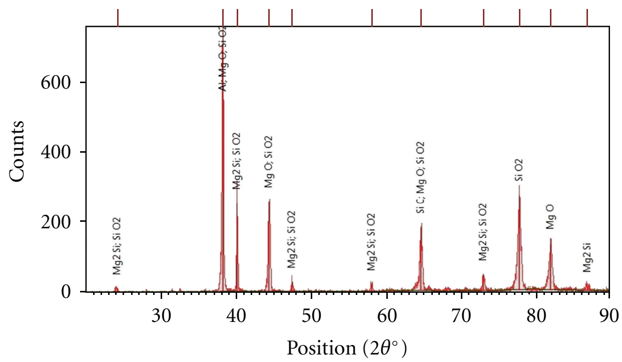


Figure 4: XRD pattern of as-cast Al–Mg_2Si composite.

situ composites with different reinforcement contents. This study aims to achieve a higher processability and performance in this composite via the mentioned treatment.

2. Material and Methods

Commercial pure Al (99.8%), Mg (99.9%), and Si (99.5%) have been used to produce Al–Mg_2Si primary ingots (8 kg). Due to the importance of elemental loss during the preparation of the melt and according to previous works [12, 13, 16], amount of weight loss was selected 3, 10, and 15% for Al, Si, and Mg, respectively. Note that the amount of weight loss for Mg (15%) was due to the high amount of oxidation of this element in the temperature rang used for preparation of the melt. First, pure Al (7 kg) was melted at 750°C in a graphite crucible with the capacity of 10 kg using an electrical resistance furnace. Then, Si and Mg were added to the melt. In order to avoid Mg weight loss, small amount of Sulphur was added to the molten alloy surface. Then molten metal matrix composite (MMC) was maintained at 750°C, 800°C, and 850°C for about 15 minutes for Mg_2Si contents of 15, 20, and 25%, respectively (in accordance to phase diagram). Table 1 shows the chemical composition of hypereutectic Al–15, 20, and 25% Mg_2Si in situ metal matrix composites (MMCs). The primary ingots were cut in small pieces, with the approximate dimensions of 40 mm × 30 mm × 20 mm, appropriate for a 2 kg SiC crucible. Then the cut ingots were remelted in another electrical resistance furnace. A flux powder was used to ensure that the melt surface was covered during melting period. Degassing was conducted by using dry tablets containing C_2Cl_6 powder (0.3 wt.% of the molten MMC) by wrapping in an aluminum foil before submerging into the melt using a graphite plunger. The melt was degassed for about 5 min. After stirring and cleaning off the dross, molten MMC was poured into a cylindrical cast iron mould (34 mm diameter and 45 mm height, Figure 2) and left to cool in the air.

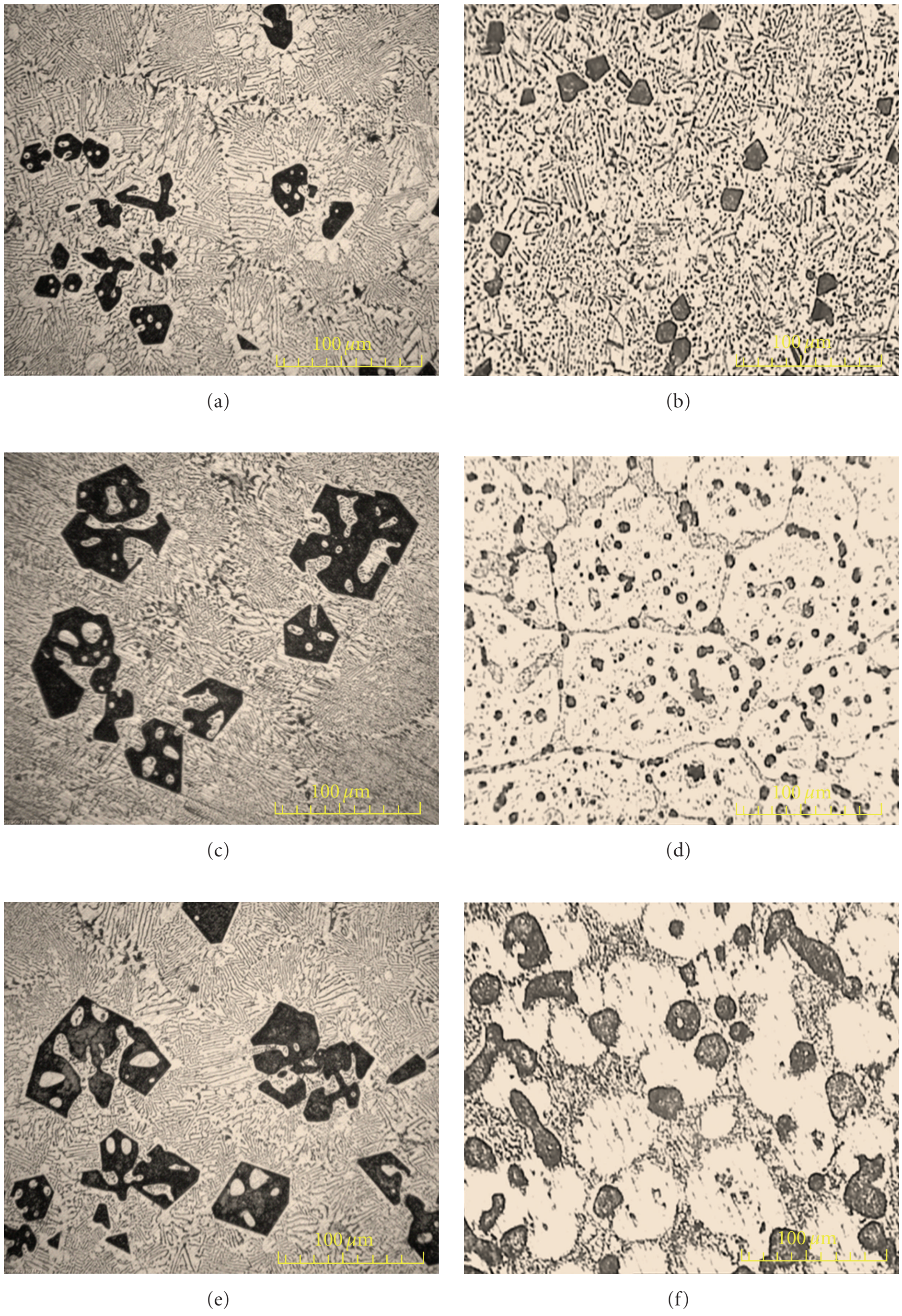


FIGURE 5: Microstructure of Al–Mg_2Si samples before and after heat-treatment in (a) 15% (as cast) (b) 15% (heat treated) (c) 20% (as cast) (d) 20% (heat treated) (e) 25% (as-cast) (f) 25% (heat treated).

TABLE 1: Chemical composition of hypereutectic Al–15, 20 and 25% Mg_2Si in-situ metal matrix composites.

Material	Si	Fe	Cu	Mn	Mg	Cr	Ni	Zn	Ti	Al
15% Mg_2Si	5.72	0.163	0.01	0.01	9.82	0.01	0.0168	0.128	0.01	Base
20% Mg_2Si	7.7	0.12	0.01	0.012	12.8	0.02	0.02	0.1	0.01	Base
25% Mg_2Si	9.23	0.134	0.01	0.01	16.1	0.01	0.012	0.09	0.011	Base

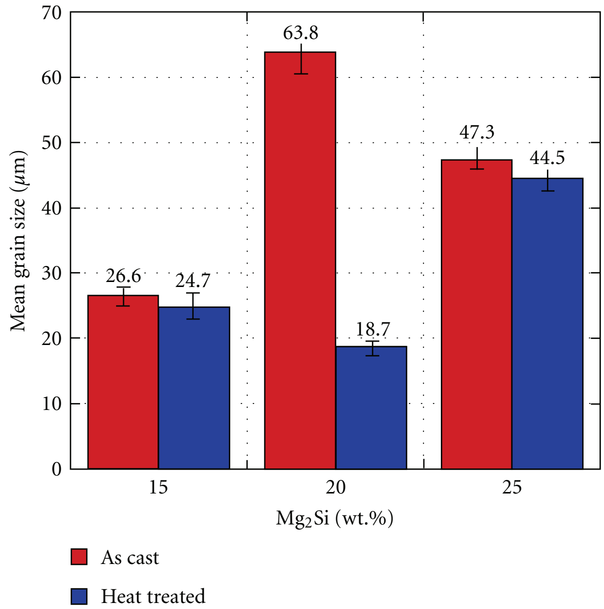


Figure 6: Comparison between primary Mg_2Si mean grain size for as-cast and heat-treated samples at different Mg_2Si wt.%.

The calculated equilibrium diagram of the Al–Mg_2Si system as a vertical section of the ternary Al–Mg–Si system is shown in Figure 1. The figure indicates that there is a narrow ternary phase region of (liquid + α-Al + Mg_2Si) between 583.5 and 594°C at the pseudoeutectic point. The composition of the pseudoeutectic is 13.9 wt.% Mg_2Si, and the solubility of Mg_2Si in the α-Al at 583.5°C is 1.91 wt.% [2, 5, 6]. Figure 1 was used as a guide to select the proper treatment temperature.

Heat treatment experiments were performed in a high accurate temperature-controlled electrical resistance furnace (±2°C). The specimens were isothermally kept at 585°C for 140 min followed by quenching in cold water.

Microstructural investigations were conducted using samples each cut from upper 1 cm of the primary heat treated and as-cast samples. The cut sections were polished and then etched by HF (1%) to reveal the structure. The microstructural characteristics of the specimens were examined by scanning electron microscopy performed in a Vega Tescan SEM. Finally, computer software (Clemex Vision Pro. Ver. 3.5.0.25) was used to calculate the grain size and nodularity of the Mg_2Si phase.

3. Results and Discussion

Figure 3 shows the typical as-cast microstructure of Al–20% Mg_2Si composite. It is clear from the phase diagram (Figure 1) that the compositions of all samples are located in the hyper section of the diagram. Basically, this means that the microstructure is consisted of coarse primary Mg_2Si particles in a matrix of α-Al and pseudoeutectic cells. Figure 4 shows the XRD pattern of the as-cast Al–Mg_2Si composite. The result reveals that the components of the composite consist of Al, Mg_2Si, SiO_2, and MgO phases, as expected. The microstructure of the Al–Mg_2Si specimens before (as cast) and after the heat treatment are shown in Figure 5. Although the microstructure of the sample with 15% of Mg_2Si prior to the heat treatment (Figure 5(a)) reveals a nondendritic and coarse morphology for reinforcement particles, but the dendritic and also coarse morphology is obvious for the samples with 20 and 25% Mg_2Si (Figures 5(c) and 5(e)). As seen in Figure 5, heat treatment has rendered the morphology to a finer and globular structure for all specimens.

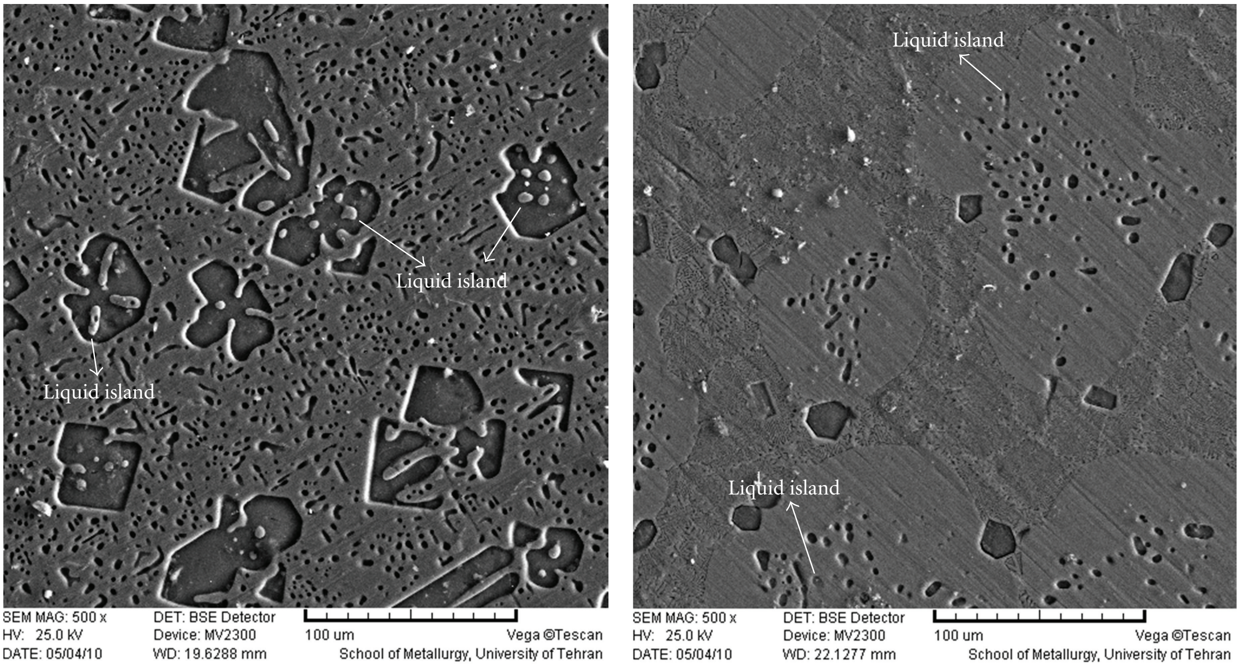
Results of quantitative investigations for the primary Mg_2Si grain size obtained by image analyzer software are listed in Figure 6. The grain size was generally reduced after the heat treatment. As seen in Figure 6, heat treatment has more significant effect for the Al–20% Mg_2Si composite which shows a 70.75% reduction in the grain size.

Table 2 summarizes the results of nodularity for the samples before and after the heat treatment in terms of distribution in the radius of curvature (RC). Equation (1) was used to calculate the RC values [17] as follows:

$$\mathrm{RC} = \frac{4\pi A}{p^2}, \tag{1}$$

where A is area, and p is perimeter of the particle. Generally the RC values are laid between 0 and 1. Lower values of this parameter represent a coarse morphology with less globularity while higher values correspond to a spherical structure. According to Table 2, for Al–15% Mg_2Si, the portion of the structure corresponds to RC values lower than 0.65 which represent a coarse structure and have been reduced from 40 to 22.73% after the heat treatment, while other portions of structure with RC values of 0.65–0.8 and 0.8–1.0, respectively, have shown an increase in magnitude. Same trend is also concluded for both other samples (Table 2). Thus, it is concluded that the heat treatment has enhanced the nodularity of the Mg_2Si phase which is more significant for the Al–20 and 25% Mg_2Si composite samples. From the stand point of energy, sharp corners are preferential sites to melt and therefore melt faster in comparison to others. This fact was employed to explain the observed decrease in the portion of structure with RC values of lower than 0.65 in Al–15% Mg_2Si samples which primarily possessed a nondendritic structure. On the other hand, fracture of Al–Mg_2Si dendrites in 20 and 25% samples is claimed to be the reason behind the increase in the nodularity and refining of grains.

According to the results, it is concluded that the effect of heat treatment was more significant for the Al–20% Mg_2Si sample while its effect for Al–15 and 25% samples was not conspicuous. Indeed, in Al–15% Mg_2Si, heat treatment has only partially melted the sharp corners of the nondendritic particles and though has not greatly enhanced the grain size and nodularity. Higher content of reinforcement in Al–25% Mg_2Si is also thought to necessitate more time and temperature of treatment to yield a globular and fine structure. Since fracture of dendrites is the dominant mechanism for increase in the nodularity of the Al–20 and 25% Mg_2Si composite, agglomeration of fractured dendrites becomes possible within treatment period. This in turn can



(a) (b)

Figure 7: SEM micrograph of Al–20% Mg_2Si composite in (a) as cast and (b) after heat treatment that showing the fine and globular grains of α-Al and Mg_2Si.

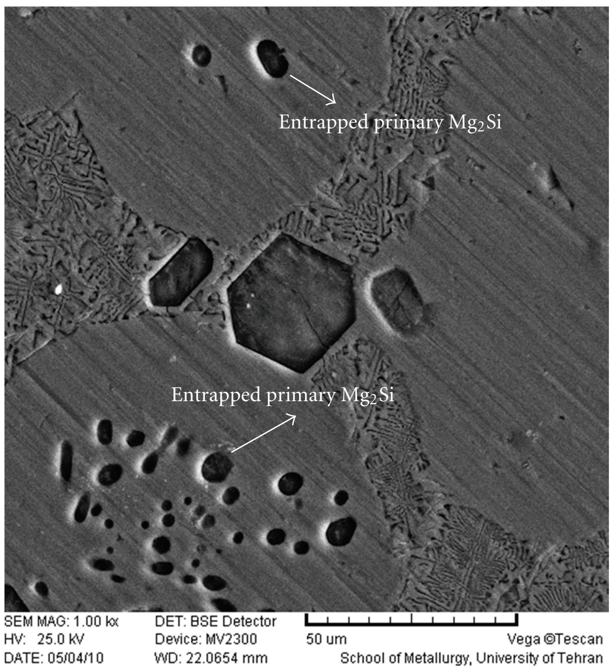


Figure 8: High magnification SEM micrograph of Al–20% Mg_2Si composite after heat treatment.

explain the rather coarse globular structure of heat-treated Al–25% 7(Mg_2Si sample, as observed in Figure 5(f).

Significant influence of the heat treatment on the Al–20% Mg_2Si sample was confirmed by SEM images of corresponding microstructure shown in Figure 7. Spherical grains of α-Al and Mg_2Si particles obtained after heat treatment are evident in this figure. As seen, Mg_2Si particles size has been reduced while nodularity increased. It is also observed that a few "liquid islands" are entrapped inside light α-Al and dark primary Mg_2Si grains in the samples, as indicated

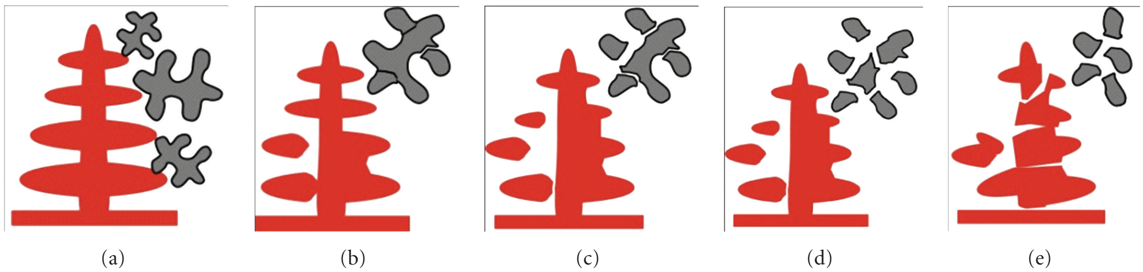


FIGURE 9: Schematic illustration of semisolid structure evolution during heat treatment of (a) original dendrites, (b) and (c) melting, liquid penetration and combining, (d) removing and (e) coalescence, ripening and spheroid formation.

TABLE 2: Results of nodularity for samples before and after heat treatment.

Type	RC* (<0.65) (%)	RC (0.65–0.8)	RC (0.8–1)
Al–15% Mg_2Si (as cast)	40	36	24
Al–15% Mg_2Si (heat treated)	22.73	40.91	36.36
Al–20% Mg_2Si (as cast)	90	0	10
Al–20% Mg_2Si (heat treated)	13.46	17.31	69.23
Al–25% Mg_2Si (as cast)	90.91	4.55	4.55
Al–25% Mg_2Si (heat treated)	37.21	25.58	37.21

* Radius of curvature.

by arrows in Figure 7. Figure 8 shows the microstructure of Al–20% Mg_2Si composite after the heat treatment in higher magnification. The eutectic Mg_2Si particles that were diffuse in the as-cast structure (Figure 7(a)) are gathered in intercellular regions after the heat treatment (Figure 8). It can be also observed that the most of globular primary Mg_2Si particulates are present in the liquid phases distributed at the grains boundaries; however, a few of ones are present inside the α-Al grains [2], as shown in Figures 7(b) and 8.

Figure 9 depicts the underling behavior of Mg_2Si and α-Al particles during the heat treatment. Dissolution of last solidified phases with low melting point occurs during the initial stages of heat treatment. The boundaries of these original phases, which were formed due to segregation, are further penetrated by the surrounding melt in a temperature above the solidus (Figure 9(b)). Dendrite arms are then remelted at their roots which lead to normal ripening of dendrite arms (Figure 9(c)). At next step, the fragmented arm renders to a spheroidal or ellipsoidal grain (Figure 9(d)). It is obvious that the size of this new grain is dependent on the size of its original dendrite arm. At next step and in case of short spacing between ripened dendrite arms, joining of adjacent particles occurs, and as a result a little amount of liquid known as "Liquid Islands" will be entrapped inside the yielded grain which is also identified as a new globular grain (Figure 9(e)). When heating continuous to the semisolid temperature and holding for a predetermined time, it also evolves toward spheroidal morphology. There are several coarsening mechanisms during the isothermal holding. One coarsening mechanism is the coalescence of the grains, namely, two grains join together and leave some bigger entrapped "liquid islands" in between the two grains, as mentioned above [18]. Another grain-coarsening mechanism is likely to the Ostwald ripening [18, 19], in which the large grains grow and the small grains remelt. Until the holding time and temperature or reinforcement content increase, the amount of the liquid phase between the grains increases to attain the equilibrium condition, and the two apparent mechanisms are shown for the liquid phase inside the grains, which coalesces to become larger in size, and also become spheroidal in shape to reduce the surface energy. Therefore, it should be noted that further increase in time and temperature of the treatment or reinforcement content can lead to more joining of grains or fragmented arms, and this in turn yields a coarser morphology and probably undesired properties [1, 2, 18–21].

4. Conclusions

The effect of isothermal holding on the microstructure and nodularity of the Al–Mg_2Si in-situ composites was studied. The following conclusions can be drawn.

(1) Microstructure of all tested samples prior to the heat treatment is consisted of coarse Mg_2Si phases in a matrix of α-Al and pseudoeutectic cell.

(2) The morphology of Mg_2Si particles is nondendritic for the sample with 15% of reinforcement, while it is dendritic for the samples with 20 and 25% of reinforcement. Heat treatment resulted in a finer and globular structure in all samples but was more significant for Al–20% Mg_2Si.

(3) Partial remelting of sharp corners is thought to be the underling mechanism for increased nodularity in the sample with 15% of Mg_2Si phase, while for the samples with 20 and 25% of Mg_2Si, fragmentation and/or further joining of adjacent particles is claimed to be dominant. Inordinate increase in the treatment

time and temperature or reinforcement content can result a coarser globular grains which may yield an improper properties.

Acknowledgment

The authors would like to thank University of Tehran for financial support of this work.

References

[1] Q. D. Qin, Y. G. Zhao, P. J. Cong, W. Zhou, and Y. H. Liang, "Functionally graded Mg_2Si/Al composite produced by an electric arc remelting process," *Journal of Alloys and Compounds*, vol. 420, no. 1-2, pp. 121–125, 2006.

[2] Q. D. Qin, Y. G. Zhao, K. Xiu, W. Zhou, and Y. H. Liang, "Microstructure evolution of in situ Mg_2Si/Al–Si–Cu composite in semisolid remelting processing," *Materials Science and Engineering A*, vol. 407, no. 1-2, pp. 196–200, 2005.

[3] Q. D. Qin, Y. G. Zhao, P. J. Cong, W. Zhou, and B. Xu, "Semisolid microstructure of Mg_2Si/Al composite by cooling slope cast and its evolution during partial remelting process," *Materials Science and Engineering A*, vol. 444, no. 1-2, pp. 99–103, 2007.

[4] H. V. Atkinson, "Modelling the semisolid processing of metallic alloys," *Progress in Materials Science*, vol. 50, no. 3, pp. 341–412, 2005.

[5] J. Zhang, Z. Fan, Y. Q. Wang, and B. L. Zhou, "Microstructural development of Al–15wt.%Mg_2Si in situ composite with mischmetal addition," *Materials Science and Engineering A*, vol. 281, no. 1-2, pp. 104–112, 2000.

[6] J. Zhang, Z. Fan, Y. Q. Wang, and B. L. Zhou, "Effect of cooling rate on the microstructure of hypereutectic Al–Mg_2Si alloys," *Journal of Materials Science Letters*, vol. 19, no. 20, pp. 1825–1828, 2000.

[7] M. P. Kenney, J. A. Courtois, R. D. Evans et al., *Metals Handbook*, vol. 15, ASM International, Metals Park, Ohio, USA, 9th edition, 1988.

[8] P. J. Ward, H. V. Atkinson, P. R. G. Anderson et al., "Semi-solid processing of novel MMCs based on hypereutectic aluminium-silicon alloys," *Acta Materialia*, vol. 44, no. 5, pp. 1717–1727, 1996.

[9] K. P. Young, C. P. Kyonka, and J. A. Courtois, "Fine grained metal composition," United States Patent US 441537430, 1982.

[10] D. H. Kirkwood, C. M. Sellars, and L. G. Elias-Boyed, "Thixotropic materials," European Patent 0305375 B1, 1992.

[11] A. Mitsuru, S. Hiroto, H. Yasunori, and S. Tatsuo, "Method and apparatus of shaping semisolid metals," European Patent 0745694A1, UBE Industries Ltd., 1996.

[12] A. Malekan, M. Emamy, J. Rassizadehghani, and A. R. Emami, "The effect of solution temperature on the microstructure and tensile properties of Al–15%Mg_2Si composite," *Materials and Design*, vol. 32, no. 5, pp. 2701–2709, 2011.

[13] R. Hadian, M. Emamy, N. Varahram, and N. Nemati, "The effect of Li on the tensile properties of cast Al–Mg_2Si metal matrix composite," *Materials Science and Engineering A*, vol. 490, no. 1-2, pp. 250–257, 2008.

[14] L. Lu, K. K. Thong, and M. Gupta, "Mg-based composite reinforced by Mg_2Si," *Composites Science and Technology*, vol. 63, no. 5, pp. 627–632, 2003.

[15] M. Mabuchi and K. Higashi, "Strengthening mechanisms of Mg–Si alloys," *Acta Materialia*, vol. 44, no. 11, pp. 4611–4618, 1996.

[16] M. Emamy, H. R. Jafari Nodooshan, and A. Malekan, "The microstructure, hardness and tensile properties of Al–15%Mg_2Si in situ composite with yttrium addition," *Materials and Design*, vol. 32, no. 8-9, pp. 4559–4566, 2011.

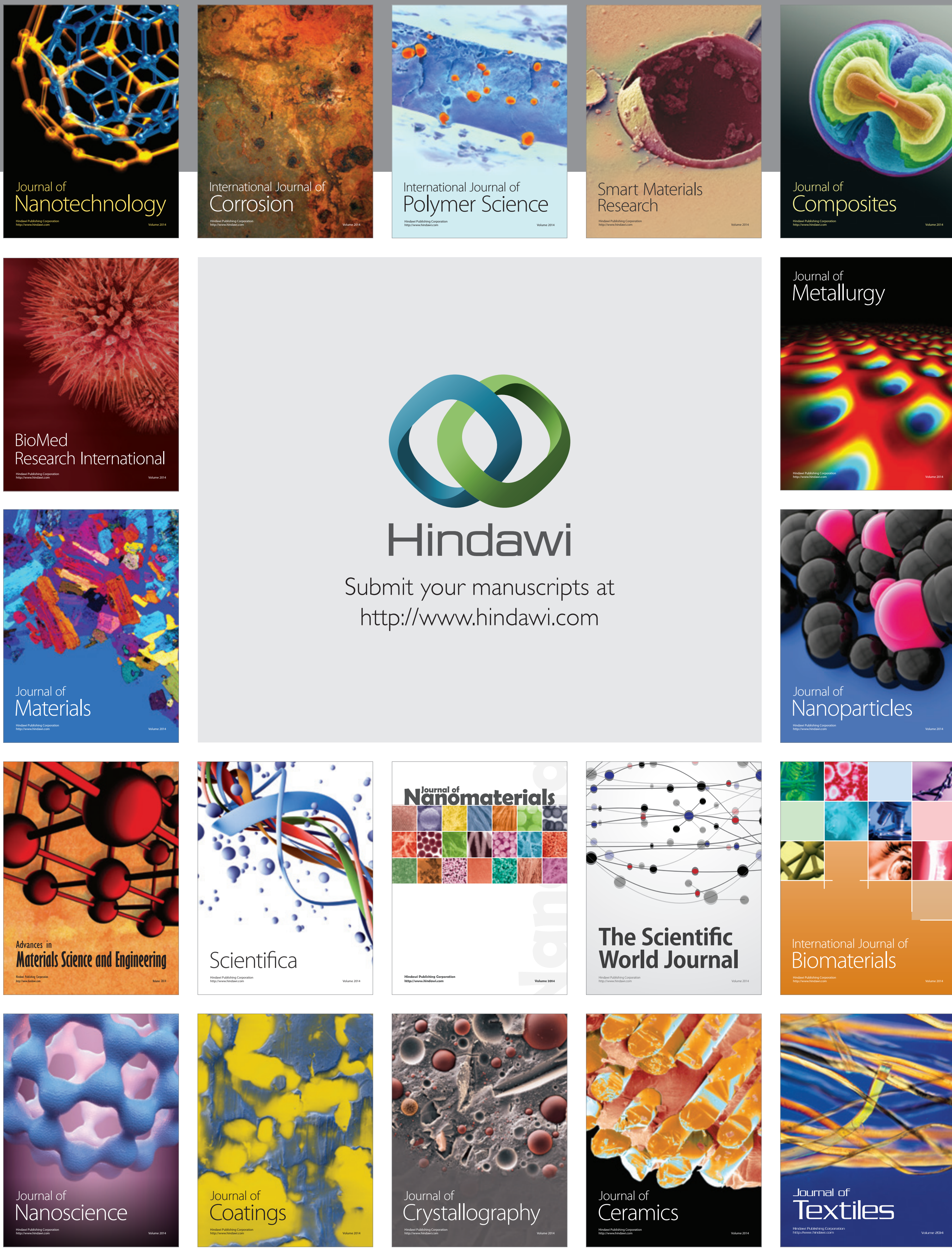
[17] W. R. Loue and M. Suery, "Microstructural evolution during partial remelting of Al–Si7Mg alloys," *Materials Science and Engineering A*, vol. 203, no. 1-2, pp. 1–13, 1995.

[18] J. L. Wang, Y. H. Su, and C. Y. A. Tsao, "Structural evolution of conventional cast dendritic and spray-cast non-dendritic structures during isothermal holding in the semi-solid state," *Scripta Materialia*, vol. 37, no. 12, pp. 2003–2007, 1997.

[19] M. C. Flemings and A. Mortensen, "Solidification of binary hypoeutectic alloy matrix composite castings," *Metallurgical and Materials Transactions A*, vol. 27, no. 3, pp. 595–609, 1996.

[20] E. Tzimas and A. Zavaliangos, "A comparative characterization of near-equiaxed microstructures as produced by spray casting, magnetohydrodynamic casting and the stress induced, melt activated process," *Materials Science and Engineering A*, vol. 289, no. 1, pp. 217–227, 2000.

[21] R. D. Doherty, H. I. Lee, and E. A. Feest, "Microstructure of stir-cast metals," *Materials Science and Engineering*, vol. 65, no. 1, pp. 181–189, 1984.


Journal of Nanotechnology
International Journal of Corrosion
International Journal of Polymer Science
Smart Materials Research
Journal of Composites
BioMed Research International
Hindawi
Submit your manuscripts at http://www.hindawi.com
Journal of Metallurgy
Journal of Materials
Journal of Nanoparticles
Advances in Materials Science and Engineering
Scientifica
Journal of Nanomaterials
The Scientific World Journal
International Journal of Biomaterials
Journal of Nanoscience
Journal of Coatings
Journal of Crystallography
Journal of Ceramics
Journal of Textiles