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Magnificent Grain Refinement of Al-Mg₂Si Composite by Hot Rolling

Rashadoddin Zamani, Hamed Mirzadeh*, Massoud Emamy

School of Metallurgy and Materials Engineering, College of Engineering, University of Tehran, Tehran, Iran.

> Recieved: 17 April 2017; Accepted: 9 October 2017 * Corresponding author email: hmirzadeh@ut.ac.ir

ABSTRACT

The effect of chemical composition and the hot rolling operations on the microstructure and mechanical properties of in situ aluminum matrix composite with Mg₂Si phase as the reinforcement was studied. It was revealed that the modification by phosphorous results in the rounder (more spherical) primary and secondary (eutectic) magnesium silicide intermetallics. During hot rolling, the primary particles underwent mechanical fragmentation and the fragmented particles moved along the rolling direction. Moreover, the eutectic Mg₂Si fragmented and uniformly dispersed in the microstructure. By increasing the reduction in thickness, it was almost impossible to distinguish primary particles from eutectic ones due to excessive fragmentation of particles. These observations were related to the brittleness of Mg₂Si phase and the elongation of the matrix grains during rolling. The grain size of the matrix also changed due to the occurrence of recrystallization and the average grain size decreases from ~ 90 μ m to 7 μ m for the 98% rolled sample. The change in mechanical properties was related to the fragmentation of particles, destroying the eutectic network, magnificent grain refinement of the matrix, the retardation of recrystallization by the dispersed particles at grain boundaries of aluminum grains, and the fast cooling of thin sheets at high reductions in thickness.

Keywords: In situ composite; Hot rolling; Microstructure; Tensile properties.

1. Introduction

Aluminum-matrix composites (AMCs) have received considerable attention from automotive and aerospace industries due to their high specific strength and specific stiffness, high hardness and wear resistance, and good elevated temperature resistance [1,2]. Composites produced by adding particles suffer from various problems like thermodynamic instability of the reinforcement in the matrix, weak matrix-reinforcement interface, inhomogeneous distribution of reinforcement particles, and lack of good elevated temperature mechanical properties. To overcome these problems, the in-situ processing of composites has been put forward as a viable remedy [2].

Among in-situ composites, Al-Mg₂Si system showed promising results, where the blockytype primary Mg₂Si reinforcements and flakelike eutectic or pseudo-eutectic Mg₂Si inside the eutectic cell form during solidification of Al alloy containing Si and Mg [2].

The effects of several modifiers such as phosphorous [3-5], lithium [6,7] and rare earth metals [8-10] on the morphology of Mg₂Si in Al-Mg-Si system have been investigated. For instance, with the addition of phosphorus, the morphology of primary Mg₂Si particulates becomes polyhedrons with more faces and their size decrease [3].

The effect of hot extrusion [11-13] and hot compression [14] on the metallurgical behavior of Al-Mg₂Si composites have been studied before and fragmentation of primary and eutectic Mg₂Si phase, their distribution, and the recrystallization of the matrix adjacent to the particles have been investigated. However, there is no report in the literature on the rolling of these composites to produce them in the sheet form. The present work reports the preliminary results obtained from hot rolling and consequent grain refinement of Al-Mg,Si composites.

2. Experimental materials and procedure

Aluminum matrix composites with 15 wt% and 20 wt% Mg₂Si were prepared in an induction furnace using 99.8% Al, 99.9% Mg and Al-30 wt% Si master alloy. Moreover, Al-15wt% Mg,Si-0.5wt% P was prepared by the addition of Cu-17 wt% P masteralloy. It should be noted that this masteralloy has gained popularity because it causes little gas pollution and has a stable and lasting effect [4,15,16]. Due to the applied Cu-P master alloy for phosphorus addition, some Cu intermetallic compounds might form in the microstructure. However, it has been reported that Cu addition has a marginal effect on the size and morphology of both primary and secondary Mg,Si particles [17,18]. Therefore, the effect of Cu was simply neglected in the present work and more experimental work is required to elucidate its effects.

The melts were heated up to 770 °C followed by pouring in a metallic mold to produce a slab with the dimensions of 147^{L} , 97^{W} , and 12.7^{T} (mm³). Table 1 shows the chemical composition of the as-cast composite. The slabs were homogenized at 520 °C for 4 h followed by furnace cooling [6,11,13]. Hot rolling operations on the Al-15Mg₂Si-0.5P at 400 °C with reductions in thickness of 60% (named as "Rolled" sample) and 98% (named as "Severely Rolled" sample) followed by air cooling were used for microstructural refinement.

The samples were electropolished at 50 V in a 20% perchloric acid - 80% ethanol electrolyte and then etched with the Keller's reagent (5 ml HNO₃

- 3 ml HCl - 2 ml HF - 190 ml H₂O) to reveal the microstructural features. An optical microscope was used for microstructural investigations. The tensile specimen was prepared according to the ASTM E8 standard with a gage length of 32 mm (Fig. 1). Tensile testing was carried out at room temperature by a computerized testing machine (SANTAM STM-20) at the constant cross-head speed of 0.1 mm/min. The diameter of Mg₂Si particles was reported based on the equivalent diameter (D_{eq}) concept defined as the diameter of a circle having the

same area as that of the particles: $D = \sqrt{4 \times area / \pi}$. The roundness of Mg,Si particles was calculated by

Roundness(%) = $100 \times 4\pi \times area / perimeter^2$. The grain size of the matrix was obtained based on the standard lineal intercept method.

3. Results and discussion

3.1. As-cast microstructures

Figure 2 shows the microstructures of the as-cast composites. It can be seen that the microstructures consist of primary Mg₂Si particles in the matrix of Al–Mg₂Si eutectic cells. This can be verified by the XRD pattern and SEM image, shown in Fig. 3, for Al-15Mg₂Si composite. In the hypereutectic Al-Mg₂Si alloys, both primary particles of Mg₂Si and α -Al can be observed besides the pseudoeutectic matrix. During solidification, Mg₂Si particles are formed as a primary phase and α -Al grains are formed at a similar time due to the non-equilibrium solidification, which restricts the diffusion of Mg



Fig. 1- Schematic representation of the tensile specimen.

| Composite | Mg | Si | Р | Cu | Al |
|------------------------------|------|-----|-----|-----|---------|
| Al-15Mg ₂ Si | 9.4 | 5.5 | 0 | 0 | Balance |
| Al-15Mg ₂ Si-0.5P | 9.4 | 5.5 | 0.5 | 2.9 | Balance |
| Al-20Mg ₂ Si | 12.8 | 7.5 | 0 | 0 | Balance |
| | | | | | |

Table 1- Nominal chemical compositions of the as-cast alloys

and Si into the surrounding liquid of the Mg₂Si particles [11,19]. In fact, there is a phase field in the phase diagram where α -Al and the pseudoeutectic co-solidify from the liquid state [10,19]. From Fig. 2, it can be seen that by increasing the weight percent of Mg₂Si, the amount of primary particles increases and the amount of eutectic constituent decreases, which can be easily verified based on the phase diagram of Al-Mg₂Si [19].

The effect of modification with P on the morphology of Mg_2Si can be seen by comparing Al-15 Mg_2Si with Al-15 Mg_2Si -0.5P composites, where the main difference is in the shape of primary and eutectic Mg_2Si phase. For eutectic Mg_2Si , the fibers tended to acquire rounder morphology, and the primary ones also became more spherical (in fact, polyhedrons with more faces). These observations can be related to the poisoning effect



Modified Al-20wt%Mg2Si Fig. 2- Optical micrographs of as-cast structures.

of P that restricts the crystal growth along some crystallographic orientation [3].

3.2. Homogenized microstructures

The homogenized microstructures after heat treating at 520 °C for 4 h are shown in Fig. 4. This treatment tremendously influences pseudoeutectic Mg₂Si structure and alters its shape from continuous eutectic Mg₂Si phase to fine, dot-like and partially round particles. After heat treatment, eutectic Mg₂Si intermetallics distribute more uniformly in the matrix. Beside eutectic structure, the sharp edged primary particles tend to become rounder (more spherical). These behaviors are similar to spheroidization of cementite in steels to reduce surface energy [20]. It can also be seen that the primary particles became coarser as a result of this spheroidization treatment.

3.3. Hot rolled microstructures

Since the Mg₂Si particles are rounder in the Al-15Mg₂Si-0.5P composite, this material was considered as the most appropriate one to conduct high reduction of rolling deformation [4,11]. The hot rolled microstructures of the Al-15Mg,Si-0.5P composite are shown in Fig. 5. It can be seen that the primary particles underwent mechanical fragmentation and the fragmented particles moved along the rolling direction. Moreover, the eutectic Mg₂Si particles also are fragmented and dispersed in the microstructure. By increasing the reduction in thickness, it is almost impossible to distinguish primary particles from eutectic ones. These observations can be easily explained by the brittleness of Mg₂Si phase and elongation of the matrix grains during rolling. For instance, the



Fig. 3- As-cast Al-15Mg2Si composite: (a) XRD pattern (using a PHILIPS X-ray diffractometer with Cu-k α radiation) and (b) SEM image (using a Vega Tescan scanning electron microscope).

average sizes of particles are $\sim 22 \ \mu\text{m}$, 6 μm and 3.9 μm for the as-cast, Rolled, and Severely Rolled samples, respectively. Moreover, the particles become rounder as shown in Fig. 6.

Fig. 5 and Fig. 6 also show that the grain size of the matrix changes, which might be related to the occurrence of recrystallization during hot rolling. The microstructural refinement of Al-Mg₂Si composite during hot compression has been reported by Shafieizad et al. [14], and similar effect is expectable for hot rolling. In fact, the average grain size decreases from 90 μ m to 7 μ m for the 98% rolled (Severely Rolled) sample, which is 1/13 that of the as-cast microstructure.

3.4. Mechanical properties

The summary of mechanical properties is shown in Fig. 7. It can be seen that the tensile strength of



Modified Al-20wt%Mg2Si

Fig. 4- Optical micrographs of homogenized composites.

the as-cast and Rolled samples are nearly the same. Two microstructural parameters are important in this respect: (1) the grain size of the rolled sample is much smaller (90 vs. 26 μ m) and (2) the network of eutectic Mg₂Si has been destroyed in the rolled sample. The former is in favor of but the latter is detrimental to strength. It seems that the balance of these effects have resulted in the observed behavior. Fig. 7 shows that the elongation of the rolled sample is much higher than that of the ascast specimen, which reveals that the redistribution of Mg,Si particles and the absence of eutectic network are in favor of ductility. By comparing the microstructures of the Rolled and Severely Rolled samples (Fig. 5), it can be seen that the aluminum grains are elongated in the latter. This shows that this microstructure is essentially a deformed one



Fig. 5- Optical micrographs of the hot rolled composites. RD represents the rolling direction for the rolled and severely rolled samples.



Fig. 6- The equivalent size of Mg2Si particles, the grain size of the aluminum matrix, and the roundness of Mg2Si particles versus rolling reduction.

and the recrystallization did not complete in this microstructure during processing. The latter can be related to the following facts:

(a) The fast cooling of thin sheets at high reductions in thickness prevents the occurrence of full recrystallization. The similar has been reported by El-Sabbagh et al. [21] in Al-SiCp composite

(b) The retardation of recrystallization occurs by highly dispersed Mg₂Si particles at grain boundaries of aluminum grains as it is evident in Fig. 5. This effect is also a known fact during recrystallization of metallic materials [22].

Figure 5 also reveals that the grain size of the Severely Rolled sample is smaller. As a result, by consideration of the deformed nature of this sample, its tensile strength is much higher (more than 2 times) and its total elongation is lower.

4. Conclusions

The effect of chemical composition and the hot rolling operations on the microstructure and mechanical properties of in situ aluminum matrix composite with Mg₂Si phase as the reinforcement was studied. The following conclusions can be drawn from this work:

(1) The modification by phosphorous results in the rounder primary and secondary (eutectic) magnesium silicide intermetallics.

(2) During hot rolling, the primary particles underwent mechanical fragmentation and the fragmented particles moved along the rolling direction. Moreover, the eutectic Mg₂Si fragmented and uniformly dispersed in the microstructure. By increasing the reduction in thickness, it was almost impossible to distinguish primary particles from eutectic ones due to the excessive fragmentation of particles. These observations were related to the



Fig. 7- Comparison between the ultimate tensile strength and total elongation of the as-cast, rolled, and severely rolled samples.

brittleness of Mg₂Si phase and the elongation of the matrix grains during rolling.

(3) The grain size of the matrix also changed due to the occurrence of recrystallization and the average grain size decreases from ~ 90 to 7 μ m for the 98% rolled sample.

(4) The change in mechanical properties was related to the fragmentation of particles, destroying of the eutectic network, magnificent grain refinement of the matrix, the retardation of recrystallization by the dispersed particles at grain boundaries of aluminum grains, and the fast cooling of thin sheets at high reductions in thickness.

References

1. Lloyd DJ. Particle reinforced aluminium and magnesium matrix composites. International Materials Reviews. 1994;39(1):1-23.

2. Pramod SL, Bakshi SR, Murty BS. Aluminum-Based Cast In Situ Composites: A Review. Journal of Materials Engineering and Performance. 2015;24(6):2185-207.

3. Qin QD, Zhao YG, Zhou W, Cong PJ. Effect of phosphorus on microstructure and growth manner of primary Mg2Si crystal in Mg2Si/Al composite. Materials Science and Engineering: A. 2007;447(1-2):186-91.

4. Nasiri N, Emamy M, Malekan A, Norouzi MH. Microstructure and tensile properties of cast Al–15%Mg2Si composite: Effects of phosphorous addition and heat treatment. Materials Science and Engineering: A. 2012;556:446-53.

5. Yeganeh SEV, Razaghian A, Emamy M. The influence of Cu-15P master alloy on the microstructure and tensile properties of Al-25wt% Mg2Si composite before and after hot-extrusion. Materials Science and Engineering: A. 2013;566:1-7.

6. Razaghian A, Bahrami A, Emamy M. The influence of Li on the tensile properties of extruded in situ Al–15%Mg2Si composite. Materials Science and Engineering: A. 2012;532:346-53.

7. Khorshidi R, Honarbakhsh Raouf A, Emamy M, Campbell J. The study of Li effect on the microstructure and tensile properties of cast Al–Mg2Si metal matrix composite. Journal of Alloys and Compounds. 2011;509(37):9026-33.

8. Emamy M, Jafari Nodooshan HR, Malekan A. The microstructure, hardness and tensile properties of Al-15%Mg2Si in situ composite with yttrium addition. Materials & Design. 2011;32(8-9):4559-66.

9. Khorshidi R, Honarbakhsh-Raouf A, Mahmudi R. Microstructural evolution and high temperature mechanical properties of cast Al–15Mg 2 Si– x Gd in situ composites. Journal of Alloys and Compounds. 2017;700:18-28.

10. Zhang J, Fan Z, Wang YQ, Zhou BL. Microstructural development of Al-15wt.%Mg2Si in situ composite with mischmetal addition. Materials Science and Engineering: A. 2000;281(1-2):104-12.

11. Emamy M, Vaziri Yeganeh SE, Razaghian A, Tavighi K. Microstructures and tensile properties of hot-extruded Al matrix composites containing different amounts of Mg2Si. Materials Science and Engineering: A. 2013;586:190-6.

12. Soltani N, Jafari Nodooshan HR, Bahrami A, Pech-Canul MI, Liu W, Wu G. Effect of hot extrusion on wear properties of Al–15wt.% Mg2Si in situ metal matrix composites. Materials & Design. 2014;53:774-81.

13. Emamy M, Khodadadi M, Honarbakhsh Raouf A, Nasiri N. The influence of Ni addition and hot-extrusion on the microstructure and tensile properties of Al–15%Mg2Si composite. Materials & Design. 2013;46:381-90.

14. Shafieizad AH, Zarei-Hanzaki A, Abedi HR, Al-Fadhalah KJ. The Mg2Si phase evolution during thermomechanical processing of in-situ aluminum matrix macro-composite.

Materials Science and Engineering: A. 2015;644:310-7.

15. Zhang Q, Liu X, Dai H. Re-formation of AlP compound in Al–Si melt. Journal of Alloys and Compounds. 2009;480(2):376-81.

16. Schmid EE, von Oldenburg K, Frommeyer G. Microstructure and properties of as-cast intermetallic Mg2Si-Al alloys. Zeitschrift für metallkunde. 1990;81(11):809-15.

Enamy M, Nemati N, Heidarzadeh A. The influence of Cu rich intermetallic phases on the microstructure, hardness and tensile properties of Al–15% Mg2Si composite. Materials Science and Engineering: A. 2010;527(12):2998-3004.
 Emamy M, Emami AR, Tavighi K. The effect of Cu addition and solution heat treatment on the microstructure hardness

and solution heat treatment on the microstructure, hardness and tensile properties of Al-15%Mg2Si-0.15%Li composite. Materials Science and Engineering: A. 2013;576:36-44.

19. Zhang J, Fan Z, Wang YQ, Zhou BL. Equilibrium pseudobinary Al-Mg2Si phase diagram. Materials Science and Technology. 2001;17(5):494-6.

20. Krauss G. Steels: processing, structure, and performance. ASM International; 2015 Mar 1.

21. El-Sabbagh AM, Soliman M, Taha MA, Palkowski H. Effect of rolling and heat treatment on tensile behaviour of wrought Al-SiCp composites prepared by stir-casting. Journal of Materials Processing Technology. 2013;213(10):1669-81.
Humphreys FJ, Hatherly M. Recrystallization Textures. Recrystallization and Related Annealing Phenomena: Elsevier;

2004. p. 379-413.