Effect of Cooling Rate on Microstructural and Microhardness Properties of Al-(Mg2Si + Al3Ni) Matrix Composite

Hossein Ramezanalizadeh*

Department of Materials and Polymer Engineering, Faculty of Engineering, Hakim Sabzevari University, Iran

Corresponding author:

Hossein Ramezanalizadeh

Department of Materials and Polymer Engineering, Faculty of Engineering, Hakim Sabzevari University, PO. Box 397, Sabzevar, Iran. Tel: 985144012779
Email: h.ramezanalizadeh@hsu.ac.ir

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ABSTRACT

Among the high-tech industries like automotive, aerospace, electronics, etc., aluminum matrix cast composites (AMCCs) are widely applied for the fabrication of accountable and especially acute pieces. During the present study, hybrid aluminum base composites containing Mg2Si and Al3Ni particles were fabricated successfully in casting moods and their structural characteristics were evaluated under different solidification conditions. A variety of microstructural measurements were performed on the composite microstructure in this study, including X-ray diffraction (XRD) and optical microscope (OM). Furthermore, a hardness test was conducted to evaluate the mechanical properties of the material. Results indicate that increasing the cooling rate during solidification reduces the average size of the Mg2Si initial phases, improves their distribution uniformity and increases their final amount whereas the average size of the Al3Ni particles decreases greatly but their content remains the same. In comparison to base alloys, hybrid composite with Mg2Si and Al3Ni particles shows the highest hardness.

Keywords: Al-based in-situ composites, hybrid intermetallic reinforcement, microstructural analysis, hardness, solidification rate, particle size.

INTRODUCTION

One of the most significant issues in materials science and engineering is the manufacture of materials with predicted properties. Currently, standard alloys cannot compete adequately with advanced structural and functional materials in terms of their mechanical and useful properties [1,2]. It is possible to achieve such goals by using aluminum (Al) matrix composites (AMCs) containing particles of oxides, carbides, silicides, borides, and other refractory materials [3]. This kind of compound can be designed at the manufacturing step by adopting particle size dispersion, morphological
characteristics, and volume or weight percent of reinforcing phases. Depending on the requirements, it can produce structural, heat-resistant, antifriction, electrotechnical, and other useful materials with bold properties.

In addition to the low degree of realization of the physico-mechanical properties of the second phase in the matrix, the technological challenges that limit the wide application of AMCs are the most significant factors in limiting the wide application of AMCs in the industry. This problem is primarily caused by poor wettability of the reinforcing particles by the matrix melt [3]. AMCs have been achieved through several technological routes [4]. Considering quality and economic standards, as well as the feasibility of metallurgical processing during the fabrication, liquid-state processes such as infiltration of porous preforms with matrix melts [5]; mechanical stirring of disintegrated particles into metallic melts [6]; chemical reactions at high-temperature that produce in-situ reinforcing compounds [7] and others are preferred.

Stir casting [8] is the most widely used method for making cast composites by mechanically mixing the melt with reinforcing particles. The process, in spite of its numerous advantages such as being simple and most economical method, has several rigid disadvantages: oxidation and gas glut of the matrix alloy during active stirring (leading to elevated porosities in castings [9]), poor bonding between the matrix and reinforcement, agglomeration of reinforcement particles. This process produces composites that are not at equilibrium, and the reinforcing materials and the matrix alloy can react severely, resulting in damage to the reinforcing materials and formation of unwanted products [10]. As a result, stir casting methods are difficult to achieve continuous and full contact between second phases and matrix, resulting in unstable mechanical and functional properties.

As an alternative to stir casting, liquid-state reactionary synthesis (in-situ process) produces novel endogenous reinforcing components through controlled exothermic reactions between the constituents of Al matrix composites prior to processing [7]. Those composite materials achieved by in-situ methods have improved thermodynamic stability and reinforcement dispensing, plus enhanced adhesion bonds along the interface between the matrix and reinforcing phases, resulting in better mechanical and operational properties. By selecting the technology of mixing the phases imported in the in-situ reactions [11], the distribution of the new compounds can be adopted. There is no need for a special rig for most routes of endogenous reinforcing. Therefore, making endogenous ceramic compounds directly in the matrix melt is more economical than making exogenously-reinforced composites with ready-made ceramic powders.

Among the many reinforcing phases, the particular attention of researchers is paid to the Mg2Si intermetallic compound, as it can be easily created in-situ via ingot metallurgy at high volume fraction [7]. The possibility for utilizing Mg2Si as a reinforcing agent is related to the set of high physical and mechanical properties of this phase, such as low coefficient of thermal expansion (7.5×10⁻⁶ K⁻¹), high melting point (1358 K), high hardness (4.5×10⁹ N.m⁻²), low density (1.88 g/cm³) and high elastic modulus (120 GPa) [7]. Although, Al/Mg2Si composites have not yet acquired a vast industrial application, due to the obtained degree of mechanical properties is relatively low due to the structural characteristics of these compounds [12]. By adding more than one reinforcing agent to a composite material, the mechanical properties can be improved. A promising choice for use along with Mg2Si for the reinforcing of an Al matrix is the intermetallic compound Al3Ni, which have a low density (~4.03 g/cm³), high melting point (~1127 K), considerable high-temperature mechanical stability up to 773 K, high Young's modulus, attractive chemical stability, low coefficient of thermal expansion, high bulk modulus (~113 GPa), and is capable to be as heterogeneous nucleation sites for the α-Al grains leading to more increase of mechanical properties of the composites [13]. In addition, the structure of endogenously-reinforced composites could be controlled by changing the cooling rate during crystallization and finally achieving a determined degree of properties [14].

It is the objective of this study to develop hybrid aluminum matrix composites (HAMC) reinforced with in-situ formed Mg2Si and Al3Ni particles and evaluate their morphology and size distribution under different thermal conditions.

**EXPERIMENTAL METHOD**

Composite materials were prepared in a 6 Kg SiC crucible in an electric resistance furnace. Pure Al ingot (≥99.99 wt.% Al), magnesium ingot (≥99.9), silicon block (≥99.0) and nickel powder (≥99.5) were used during the melting. Because the importance of elemental loss during the melt preparation, amount of weight loss was considered as 5, 5, 10 and 15% for Al, Ni, Si and Mg, respectively. Note that the amount of weight loss for Mg (15%) was due to the high level of oxidation of this element in the used temperature range for preparation of the melt. Firstly, crucible was filled by Al ingot and heated up to melt state. Then silicon and foil-wrapped magnesium...
preheated to 150°C were added to Al melt at 750°C. After melting the charge components, it was manually stirred by a graphite rod, followed by overheated to 800°C and the canned nickel powder was added. The melt temperature was then increased to 900°C and hold for 15 min. The temperature modes of the test were controlled by K-type thermocouple with a precision of ± 1.5°C. The provided melt was cast at a temperature of 750°C into cold copper and steel molds to achieve ingots with a diameter of 45 mm and a length of 70 mm. Table 1 shows the chemical composition of the in situ AMCCs obtained by Quantometer analysis.

| Materials | Si   | Mg   | Ni   | Fe  | Zn  | Mn  | Cu  | Ti  | Cr  |
|-----------|------|------|------|-----|-----|-----|-----|-----|-----|
| %Wt.      | 6.5  | 12.3 | 3.1  | 0.01| 0.01| 0.01| 0.01| 0.01| 0.01|

Sections of casting rods were used to characterize microstructural properties. Metallographic specimens were polished using standard methods and etched with 5% HF for near 10 s at room temperature. Microstructural parameters were determined using an optical microscope equipped with an image analysis system (Clemex Vision. Pro. Ver. 3.5.025). Phase analysis was identified by X-ray diffractometry (XRD, Philips PW 1730, 40 kV and step of 0.02o) with Cu Kα radiation (λ = 0.15406 nm). Phase identification was performed in the High X’Pert software complex using the Crystallography Open Data database. The hardness was estimated by Brinell hardness (Wilson® BH3000 Brinell Hardness Tester) using 5-mm indenter at 2500-N load. The cubic sample size for hardness test was chosen to be 2cm×2cm×1cm. The hardness values were average of at least ten measurements. It should be noted that for simplicity, henceforth AMN word applies instead of Al/(Mg2Si + Al3Ni) (A, M and N letters refer to Al, Mg2Si and Al3Ni, respectively).

RESULTS AND DISCUSSION

Figure 1 shows the microstructure of synthesized AMN composites under different solidification rate. In addition, the histograms of the size distribution of reinforcing particles is drawn in Figure 2. Primary Mg2Si particles formed in the steel mold have an irregular, coarse and dendritic morphology or turns into a hole phases and its sizes can attain over of 50 μm. The early Mg2Si particles solidify into incomplete octahedrons that wax quickly along the direction <100> to create the primary solid dendrites under common solidification conditions [15]. Due to the anisotropy growth of the Mg2Si phases, it is feasible the advent of complicated and dendritic-like systems with large sizes, which recognition stresses at their horned corners and planes.

![Figure 1](https://via.placeholder.com/150)

**Figure 1.** Microstructures of as-cast AMN composite samples obtained using different types of molds.
Therefore, the modification of Mg2Si particles may be a vital way in enhancing the mechanical properties of composite materials. An increment in the cooling rate during solidification, due to the use of a copper mold, results in decreasing the mean size of the primary Mg2Si phases to 8.5 μm, enhancement of the dispersion uniformity and significant increasing of their final amount (Fig. 3). Concurrently, there was no clear change in the morphology of the Mg2Si particles during the crystallization with a raised cooling rate. Intermetallic compound Al3Ni crystallizes mostly in the form of dense and block phases, and their content is almost the same for both of molds. When using the copper mold, the average size of Al3Ni particles reduced to 5 μm. In addition, the size distribution of Mg2Si and Al3Ni particles is more uniform and nearby the Gaussian distribution, that is seen from the effects of distribution histograms (Fig. 2, c,d).

**Figure 2:** Size distribution graphs of reinforcing phases in AMN composites specimens obtained at different cooling rates.

**Figure 3:** Quantitative parameters of reinforcing particles in AMN composite.
For more investigation, the microstructural revolution of AMN hybrid composite was done by SEM and the result is shown in Figure 4a. As seen, the composite structure consists of Al as matrix and Mg2Si and Al3Ni as reinforcement particles. The corresponding elemental mapping as well as the XRD patterns of AMN hybrid composite is illustrated in Figures 4b and 5 respectively, which indicate the existence of the Al, Mg2Si and Al3Ni in the HAMC.

The XRD graph proves that the structural phases of the received specimens are α-Al (JCPDS card # 04-0787), Mg2Si (JCPDS card # 035-0773) and Al3Ni (JCPDS card # 02-0416) (Fig. 4). Therefore, it can be concluded that in-situ melt exothermic reaction occurred fully between the Ni powder and the Al (3Al + Ni = Al3Ni + 258 kcal/mol), and also Mg and Si in a certain stoichiometric ratio produced Mg2Si phase.

The mean hardness value of AMN composites increases from 70.04 ± 1.23 to 122.91 ± 1.05 HBN, when the copper mold is used. The increasing in the acquired hardness value is basically due to the crushing of the primary Mg2Si particles, decreasing their size and an enhancement in the tedium of their distribution thorough the composite materials.

On the other hand, the space among the reinforcing particles reduces with the aid of lowering their length. This issue become schematically proven in Fig. 5 and can be defined with the aid of using Equation (1), because the reinforcement particle size decreases, the space among the particles may even decreases ($\lambda_2 < \lambda_1$) [16].

$$\lambda = \frac{4(1-f)}{2f}$$

Where in $\lambda$ is the space among the reinforcement particles, f is the particle extent fraction and r is the particle radius, assuming them spherical. In different words, in line with Equation (2) lowering the space among the Mg2Si particles will boom the specified stress for dislocations motion among them, ensuring in a boom withinside the composite strength. The shear stress needed to overcome the obstacle is:

$$\tau = \frac{Gb}{\lambda}$$

where G is shear modulus, b is burger’s vector and $\lambda$ is space between obstacles [16].

![Figure 4: XRD pattern of AMN composite](image)

![Figure 5: Schematic of reducing the distance between the particles by decreasing the particle size.](image)

Therefore, HAMCs can be manufactured through a one-degree casting technique with the simultaneous creation of Mg2Si and Al3Ni intermetallic compounds. Alternatively, the mix of different types of reinforcing phases in one material could prolong the potentials of meaning structure control to achieve the required mechanical and functional properties of cast counterparts.

CONCLUSION

In current research, HAMC materials reinforced with in-situ particles Mg2Si and Al3Ni were produced successfully by melt state production process in different thermal conditions during solidification. The main results could be listed as follow: The XRD pattern proves that the structural phases of the gained in-situ composites are α-Al, Mg2Si and Al3Ni.

An increase in the cooling rate of solidification, by the use of a copper mold alternated steel mold, results in decreasing the mean size of the primary Mg2Si phases from 12.5 to 8.5 μm and enhancement of the distribution homogeneity; synchronously, the mean size of Al3Ni particles reduces from 5.7 to 5 μm but their amount is almost the same for both molds.
With increasing the solidification rate, the size histogram of Mg2Si and Al3Ni particles becomes more homogeny and near the Gaussian distribution.

The mean hardness value of AMN in-situ composites raises from 70.04 ± 1.23 to 122.91 ± 1.05 HBN when casting into the copper mold, which shows a 75.5% increase.

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