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Research Paper

Effect of cooling rate on microstructural and microhardness properties of Al-(Mg_2Si + Al_3Ni) Matrix Composite

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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 Mg_2Si and Al_3Ni 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), optical microscope (OM) and scanning electron microscope (SEM). 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 Mg_2Si initial phases, improves their distribution uniformity and increases their final amount, whereas the average size of the Al_3Ni particles decreases greatly but their content remains the same. In comparison to base alloys, hybrid composite with Mg_2Si and Al_3Ni particles shows the highest hardness.

Keywords: Al-based in-situ composites, Hybrid intermetallic reinforcement, Microstructural analysis, Hardness, Solidification rate, Particle size.

1. 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 powder 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, 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 dispersing, 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 reinforcement. 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 Mg_2Si intermetallic compound, as it can be easily created in-situ via ingot metallurgy at high volume fraction [7]. The possibility for utilizing Mg_2Si 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 (4.5×10^{9} $N.m^{-2}$), low density (1.88 g/cm^3) and high elastic modulus (120 GPa) [7]. However, Al/Mg_2Si composites have not yet acquired a vast industrial application, because 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 Mg_2Si for reinforcement of an Al matrix is the intermetallic compound Al_3Ni, which has a low density (~4.03 g/cm^3), 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 of being heterogeneous nucleation sites for the α-Al grains leading to greater 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 Mg_2Si and Al_3Ni particles and evaluate their morphology and size distribution under different thermal conditions.

2. Materials and Methods

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 ºC. After melting the charge components, the material was manually stirred by a graphite rod, followed by overheating to 800 °C and the canned nickel powder was added. The melt temperature was then increased to 900 °C and held 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.

Table 1 Chemical composition of AMCCs

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 nearly 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). Additionally, a scanning electron microscope (SEM) was used to analyze microstructural revolutions. 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 using 5 mm indenter at 2500 N load. The hardness values were the average of at least ten measurements. It should be noted that for simplicity, henceforth AMN abbreviation is applied instead of Al/(Mg_2Si + Al_3Ni) (A, M and N letters refer to Al, Mg_2Si and Al_3Ni, respectively).

3. Results and Discussion

Fig. 1 shows the microstructure of synthesized AMN composites under different solidification rate. In addition, the histograms of the size distribution of reinforcing particles are drawn in **Fig. 2 a-d**. Primary Mg_2Si particles formed in the steel mold have an irregular, coarse and dendritic morphology or turns into hole phases and its size can reach over 50 µm **((Fig. 1a) and 1c)).** Similarly, the same reinforcing particles formed in copper mold is shown in **Fig. 1b) and 1d)**.The early Mg_2Si 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 Mg_2Si phases, it is feasible that the advent of complicated and dendritic-like systems with large sizes, which recognizes stresses at their horned corners and planes.

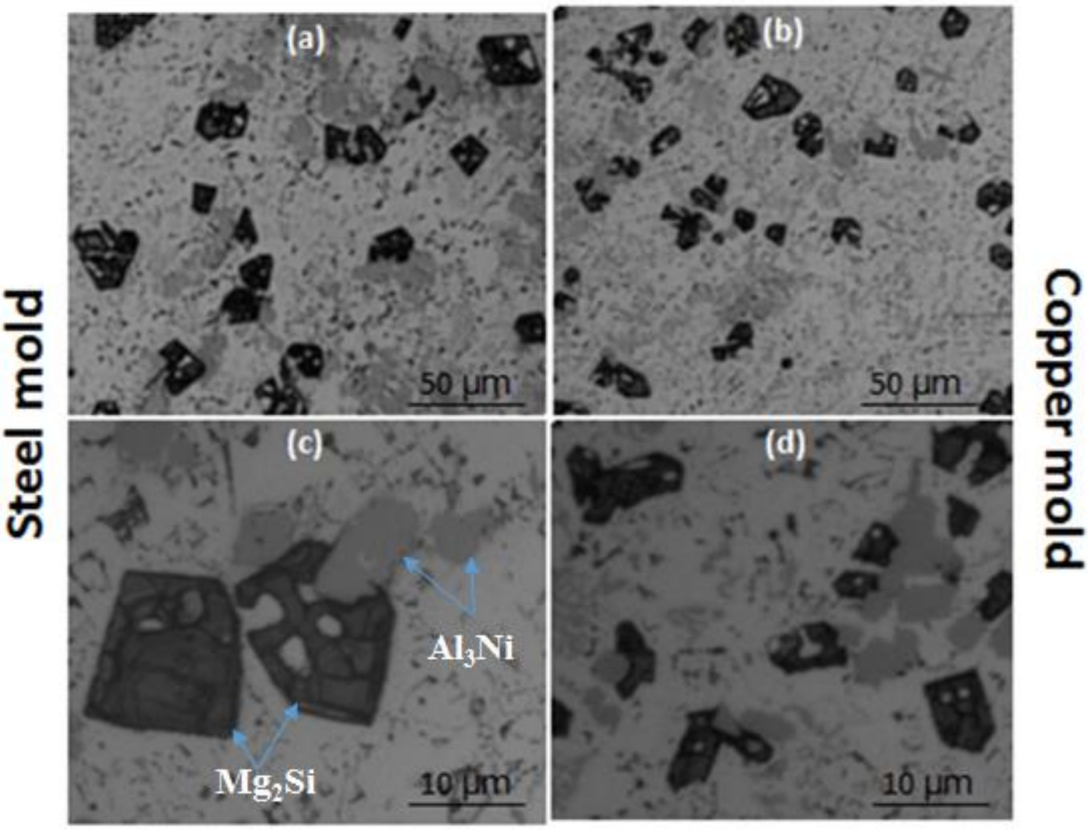

Fig.1 Microstructures of as-cast AMN composite samples obtained using different types of molds: a), c) in steel mold, and b), d) copper mold

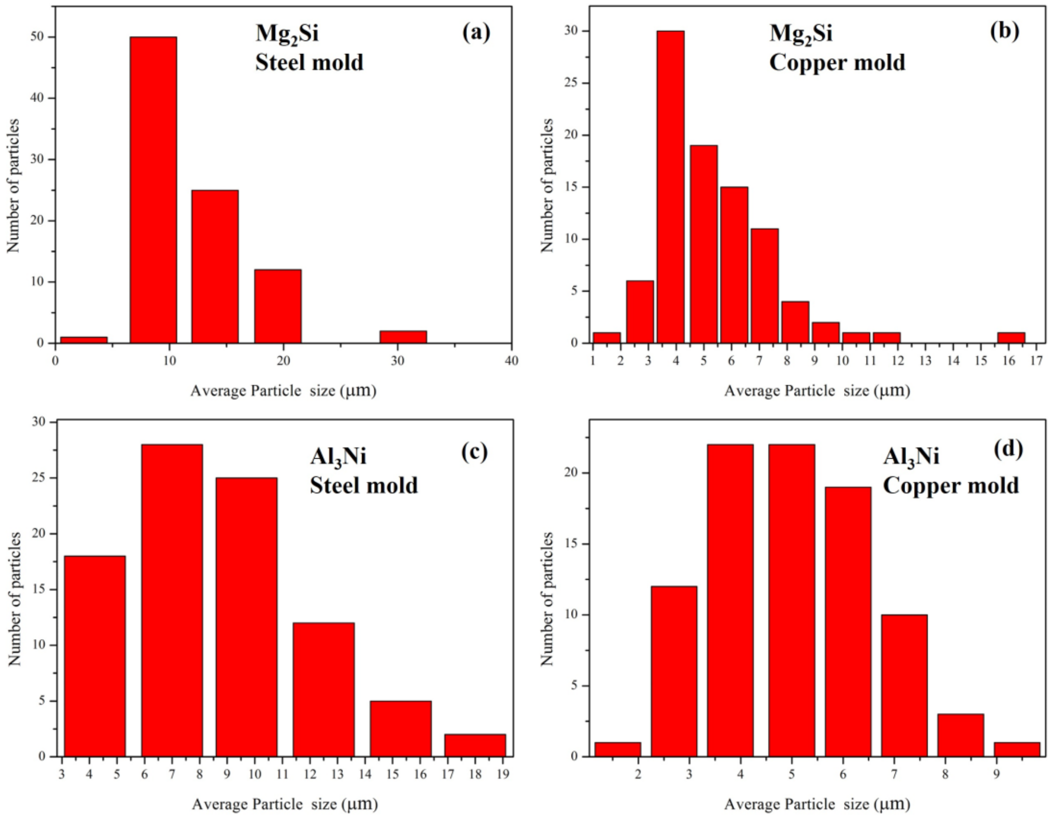

Fig. 2 Size distribution graphs of reinforcing phases in AMN composites specimens obtained at different cooling rates: a), b) in copper mold, and c), d) in steel mold

Therefore, the modification of Mg_2Si particles may be a vital way to enhance 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 Mg_2Si phases to 8.5 µm, enhancement of the dispersion uniformity and significant increase of their final amount (**Fig. 3**). Concurrently, there was no clear change in the morphology of the Mg_2Si particles during the crystallization with an increased cooling rate. Intermetallic compound Al_3Ni crystallizes mostly in the form of dense and block phases, and their content is almost the same for both molds. When using the copper mold, the average size of Al_3Ni particles reduced to 5 µm. In addition, the size distribution of Mg_2Si and Al_3Ni particles is more uniform and near the Gaussian distribution, that is seen from the effects of distribution histograms (**Fig. 2, c,d)**.

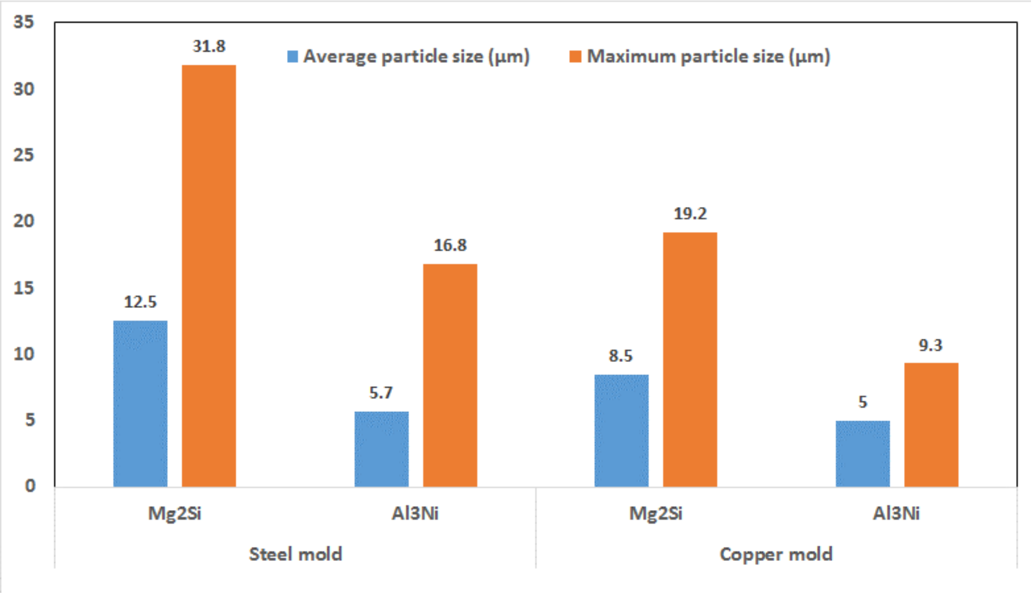

Fig. 3 Quantitative parameters of reinforcing particles in AMN composite

For more investigation, the microstructural revolution of AMN hybrid composite was done by SEM and the result is shown in **Fig. 4a**. As seen, the composite structure consists of Al as matrix and Mg_2Si and Al_3Ni as reinforcement particles. The corresponding elemental mapping as well as the XRD patterns of AMN hybrid composite is illustrated in **Fig. 4b** and **Fig. 5** respectively, which indicate the existence of the Al, Mg_2Si and Al_3Ni in the HAMC.

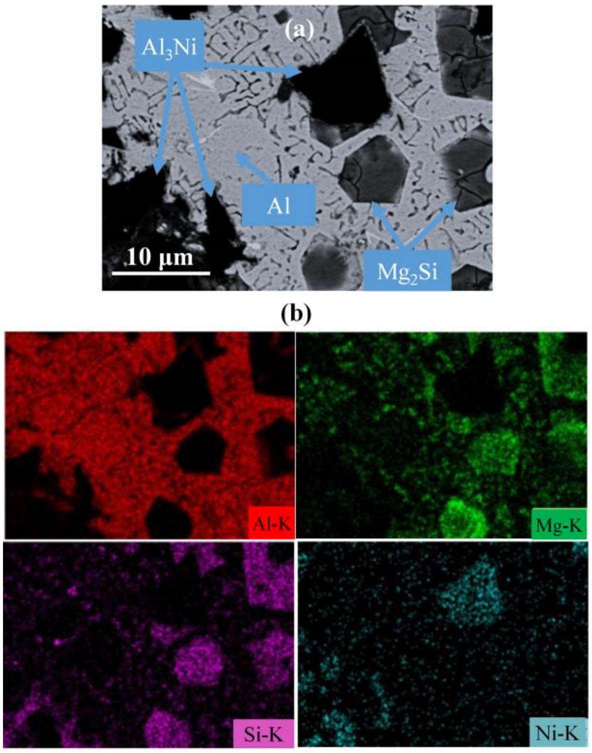

Fig.4 a) SEM image of AMN composite and, b) corresponding elemental mapping analysis

The XRD graph proves that the structural phases of the received specimens are α-Al (JCPDS card # 04-0787), Mg_2Si (JCPDS card # 035-0773) and Al_3Ni (JCPDS card # 02-0416) (**Fig. 5**). Therefore, it can be concluded that in-situ melt exothermic reaction occurred fully between the Ni powder and the Al ($3Al + Ni = Al_3Ni + 258$ kcal/mol), and also Mg and Si in a certain stoichiometric ratio produced Mg_2Si phase.

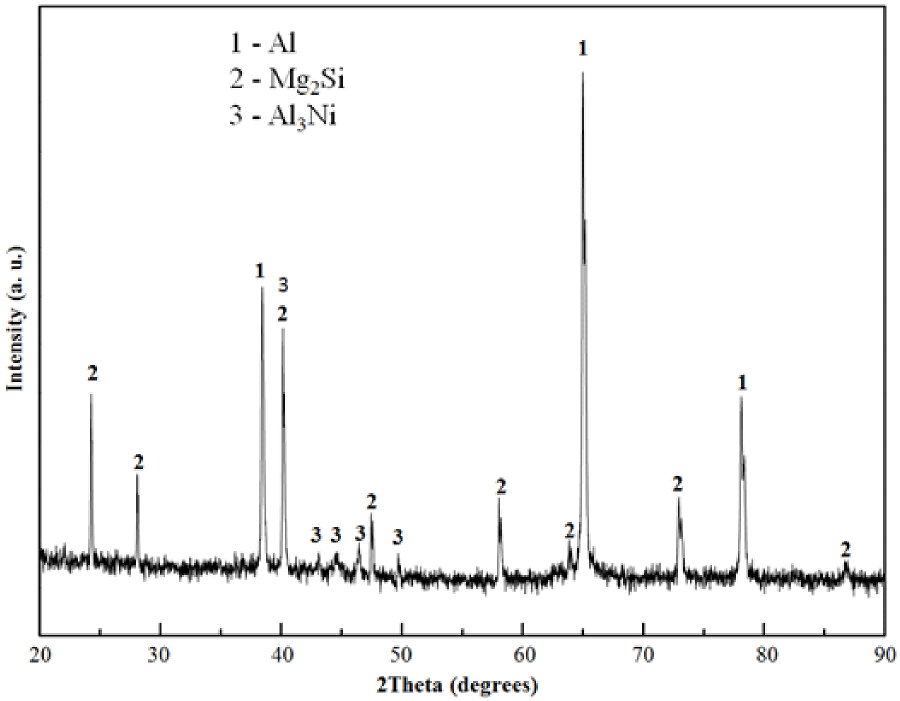

Fig. 5 XRD pattern of AMN composite

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 increase in the acquired hardness value is basically due to the crushing of the primary Mg_2Si particles, decreasing their size and leading to an enhancement in the tedium of their distribution thorough the composite materials.

On the other hand, the space among the reinforcing particles reduces with a decrease in their length. This was schematically proven in **Fig. 6** and can be defined in Eq. (1), because the reinforcement particle size decreases, the space among the particles may even decrease ($\lambda_2 < \lambda_1$) [16].

$$\boldsymbol{\lambda} = \frac{\mathbf{4(1-\boldsymbol{f})\boldsymbol{r}}}{\mathbf{3\boldsymbol{f}}} \qquad \text{Eq.1}$$

wherein λ is the space among the reinforcement particles, f is the particle extent fraction and r is the particle radius, assuming they are spherical. In other words, in line with Eq. (2), lowering the space among the Mg_2Si particles will enhance the specified stress for dislocations motion among them, ensuring a rise in the composite strength. The shear stress needed to overcome the obstacle is:

$$\tau = Gb/\lambda \quad \text{Eq. 2}$$

where G is shear modulus, b is Burger's vector and λ is space between obstacles [16].

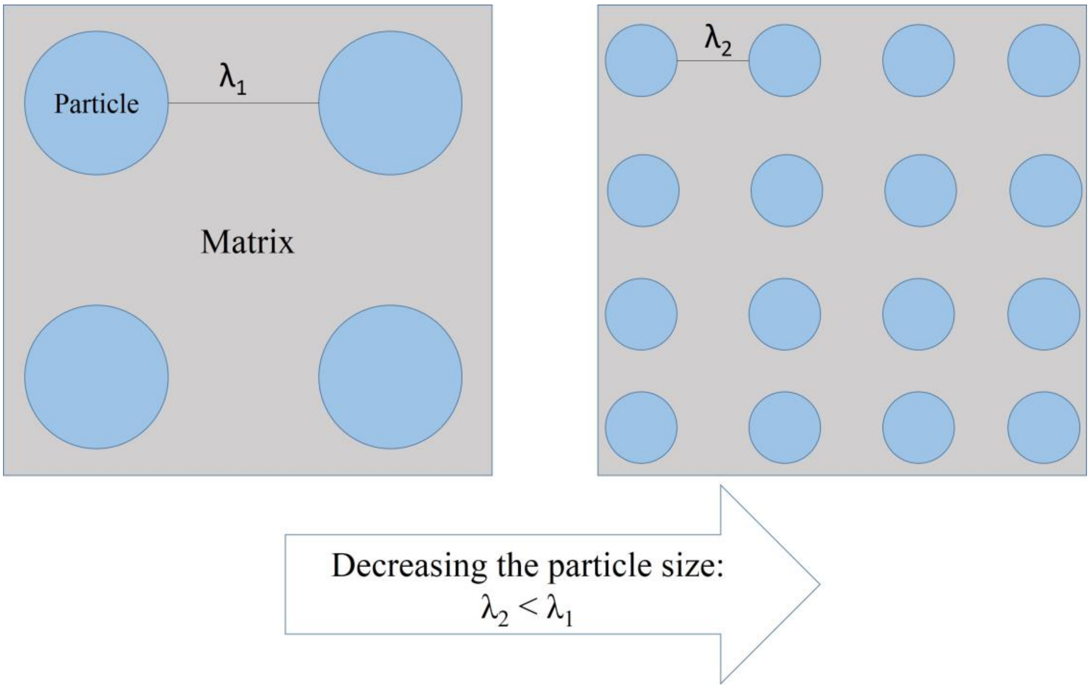

Fig. 6 Schematic illustration of the distance between the particles that affected by the particle size

Therefore, HAMCs can be manufactured through a one-degree casting technique with the simultaneous creation of Mg_2Si and Al_3Ni 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.

4. Conclusions

In current research, HAMC materials reinforced with in-situ particles Mg_2Si and Al_3Ni were produced successfully by melt state production process in different thermal conditions during solidification. The main results could be listed as follows:

The XRD pattern proves that the structural phases of the gained in-situ composites are α-Al, Mg_2Si and Al_3Ni.

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 Mg_2Si phases from 12.5 to 8.5 μm and enhancement of the distribution homogeneity; simultaneously, the mean size of Al_3Ni particles reduces from 5.7 to 5 μm but the amount is almost the same for both molds.

With increasing the solidification rate, the size histogram of Mg_2Si and Al_3Ni particles becomes more homogenous and near the Gaussian distribution.

The mean hardness value of AMN in-situ composites rises 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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