The Role of Heat Treatment on Characteristics of Mg-Al-Sr Reinforced Nano-SiC Composite

Vioni Dwi Sartika, Achmad Fauzi Trinanda, Rama Aditya Syarif, Ivan Orlando Limouswan, Anne Zulfia Syahrial*

1Department of Metallurgy and Materials Engineering, Faculty of Engineering Universitas Indonesia, Kampus Baru UI Depok 16424, Indonesia

anne@metal.ui.ac.id

Abstract. The effect of heat treatment on microstructure and mechanical properties of SiC/Mg-Al-Sr alloy matrix composite was investigated. The heat treatment involved annealing at 425, 375, and 325°C for 4 hours, water quenching then ageing at room temperature for 48 hours. The heat treatment process caused dissolution of Mg$_2$Si precipitated and decomposition of nano-SiC particles. The composites with 0.15 Vf-% nano-SiC which was treated at 425°C has reached the optimum hardness due to precipitate of the intermetallic phase. Besides, the hardness of the treated composites still has good ductility. The fracture surface of the composite consisted of numerous dimples as a result of micro voids nucleated at the interface between the matrix and SiC particles and second-phase particles as well. The fracture of heat treated composite was similar to microvoids which were nucleated mainly on nano-SiC particles as obtained during fractography analysis.

Keywords: Magnesium composite, Mg-Al-Sr, Nano-SiC, Heat treatment

1. Introduction

The transportation industries are facing some challenges, such as the increasing demand for economical use of energy resources and stricter control over emissions to lower the climate change effect. This condition triggered the industries to seek lightweight materials as an alternative to conventional materials. The use of lightweight materials in vehicles become a solution to cut the energy consumption because it takes less energy to accelerate a lighter object than a heavier one, lightweight materials offer great potential for increasing vehicle efficiency. One of promising material to overcome this problem is magnesium[1]. Magnesium is material which has high specific strength with low density and good mechanical properties[2]. Commonly, magnesium being strengthened by adding alloys of other elements[1]. It is hard to do a cold-working process due to the HCP crystal structure of this metal[3]. Aluminum is an element that is commonly used as magnesium alloy. Another element like strontium can be added to Mg-Al alloys to enhance the strength at elevated temperature [4]. The mechanical properties of this alloy can be improved with the addition of various types of reinforcement such as SiC particles[5]. This particles have a good interface with the magnesium alloy matrix and can be distributed uniformly[6]. Another way to improve the mechanical properties of this alloy is through a heat treatment process to modify the intermetallic phases in the matrix. Intermetallic
phases such as Mg17Al12, Al5Sr, Mg2Sr, MgSr and Mg17Sr2 in magnesium matrix could enhance the strength[7]. These intermetallic phases can retard the movement of dislocations, so it needs higher energy for deformation to occur.

In the previous work, magnesium composite was produced using stir casting route. The chemical composition of matrix is Mg-1.25Al-0.75Sr wt% and addition of nano-SiC was varied by 0.05; 0.10; 0.15; 0.20; 0.25 Vf%[8]. In this work, the composites were heat treating in 3 variable temperatures, 325, 375, 425°C for 4 hours. The aims of this experiment are to analyse the effect of age-hardening heat treatment on microstructure, mechanical properties and fracture mechanism of composites.

2. Experimental method

2.1 Synthesis of Mg-Al-Sr reinforced nano-SiC composite

The stir casting process is used to make the specimens. Magnesium ingot (99.9 wt%) was used as the matrix of the composite. The alloying element was added by using Al-15Sr alloy, then combined with various compositions of the nano-SiC particles (0.05; 0.10; 0.15; 0.20; and 0.25 Vf-%). In the making, magnesium was heated to its molten state in 860°C, then Al-15Sr in the form of a rod was added and stirred for 2 minutes. Before poured in the molten magnesium, nano-SiC was preheated in a muffle furnace for an hour in 900°C to remove moist and increase surface tension. The composite is poured into the mould which had been preheated in 900°C for 1 minute and coated with zirconia to reduce thermal shock. The Mg, Al and Sr content of all casts were analysed using Optical emission spectroscopy (OES) (the chemical compositions are shown in Table 1).

| Table 1. Chemical composition of Mg-Al-Sr/Nano-SiC composite[8]. |
|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|
| Element         | 0%              | 0.05%           | 0.10%           | 0.15%           | 0.20%           | 0.25%           |
| Mg              | 96.3            | 96              | 96              | 96.5            | 95.8            | 95.3            |
| Al              | 3.54            | 3.78            | 3.83            | 3.28            | 3.89            | 4.48            |
| Be              | <0.0001         | 0.0001          | <0.0001         | <0.0001         | 0.0002          | 0.0001          |
| Cu              | <0.0010         | 0.003           | <0.0010         | <0.0010         | 0.0168          | 0.0015          |
| Mn              | 0.0645          | 0.0391          | 0.0477          | 0.0485          | 0.0524          | 0.0259          |
| Zn              | <0.0010         | 0.0343          | 0.0264          | 0.0339          | 0.0789          | 0.026           |
| Ag              | <0.0010         | <0.0010         | <0.0010         | <0.0010         | 0.0047          | <0.0010         |
| Ca              | 0.0024          | 0.0031          | 0.0024          | 0.003           | 0.0031          | 0.0096          |
| Cd              | <0.0010         | 0.0011          | <0.0010         | <0.0010         | 0.0019          | 0.001           |
| Sn              | 0.0327          | 0.0543          | 0.0291          | 0.0279          | 0.0834          | 0.0545          |
| Sr              | >0.0008         | >0.0008         | >0.0008         | >0.0008         | >0.0008         | >0.0008         |

2.2 Heat Treatment

Each composite that is obtained through stir casting process then being heat treated at three different temperatures (325°C, 375°C and 425°C). The holding time for each temperature variable is 4 hours and the temperature rises at 10°C/minute with preheating for about 15 minutes.

2.3 Mechanical Properties

The composites, which had been heat treated, were prepared and mechanical properties tested (hardness& wear resistance). Someparts are cut into 2×3(cm) and ground to obtain a fine surface then tested by Brinell Hardness at room temperature. Weartest is conducted using the pin-on-disk machine by sliding a specimen under dry condition.
2.4 Metallography
All casts microstructurally characterised to observe the grain size of matrix alloy using an optical microscope (OM) and scanning electron microscope (SEM). They were cut, polished using alumina and ethanol, then etched using Nital reagent. EDX and XRD analysis are conducted to confirm the phases formed in the cast.

2.5 Fractography
The impact test sample was further analysed the fracture surface by a scanning electron microscope (SEM). The composite with 0.15 vf% nano SiC is selected as a comparative date to see the fracture behaviour of the composite after heat treatment.

3. Results and discussion

3.1 Hardness and Wear Resistance
In previous work, it was stated that the increasing of nano-SiC could enhance the hardness of composites. However, after certain addition, there is no significant effect since the tendency of particles to agglomerate[8]. In this work, heat treatment process improved the hardness value of composites, as a temperature increased as seen in figure 1. The highest hardness was found in the composite which had been heat treated at 425°C with the addition of 0.15 Vf% nano-SiC, that is 75 BHN as present with the blue line in figure 1. This hardness is 47.67% greater than magnesium alloy without reinforcement and heat treatment.

![Figure 1. The hardness value of composites.](image)

Usually, the hardness of material relating to its wear resistance. Abrasive testing gives the wear rate which shows the lowest value. It is matched with the hardness given in figure 1. In some conditions, it is still observed the fluctuation wear rate in figure 2. Another contributing factor which increases the wear rate is the debris formed and stick on the rotating pin, so it could erode the sliding surface of the composite.
3.2 Microstructure

The observation of microstructure using optical microscope is conducted on Mg-Al-Sr/0.15 Vf% nano-SiC before [8] after heat treatment process, since it gives the highest hardness value and wear resistance. The microstructure of the two composites consists of $\alpha$-Mg, Mg$_{17}$Al$_{12}$, MgAlSr, which shown in Figure 3.

![Microstructure Images]

**Figure 3.** The microstructure of Mg-Al-Sr/0.15 Vf% nano-SiC, (a) before [8] and; (b)after heat treatment process.
Microstructural analysis using SEM-EDX.

Microstructure characterisation using SEM found that the average size of intermetallic phases before heat treatment is about 7.777μm[8]. On the other hand, there is significant change observed after heat treatment process which reduces the average size of intermetallic phases. The measurement shows the size is about 2.990 μm. EDX analysis which conducted on composite after heat treatment reveals the phases formed at point A, B, C, D in figure 4 are α-Mg, Al, Sr, Mg2Si, MgAl. According to Xia et al[9], Mg2Si could found in magnesium composite as the result of a reaction between Mg and SiC particles. It indicates that the improvement of hardness after heat treatment is caused by the finer precipitates and existence of Mg2Si phase.

3.3 XRD analysis
Another way to see the phases that are formed after heat treatment is through XRD (X-ray Diffraction) analysis as shown in Figure 5.
XRD analysis in figure 5 found the MgAl, Mg$_2$Sr$_2$ compound. This compound has resulted after heat treatment process. This precipitate gave strengthening effect to the matrix, so that the hardness of the composite enhanced after the heat treatment process[10]. Then, the XRD analysis found Mg$_2$Si compound at 2 theta position of 78.1. Mg$_2$Si compound is formed from the interfacial reaction between nano-SiC and Mg matrix[11]. Another compound such as SiC and SiO$_2$ are found at 2 theta position of 71 and 63.2 respectively. SiO$_2$ compound is formed on the surface of SiC within the pre-heating process before casting. Li et al[9] reported that pre-oxidation of nano-SiC at 973-1273K in 2 hours could produce the SiO$_2$ layer on the surface. This layer could enhance the adhesion force between the matrix and reinforce.

3.4 Fractography

Figure 6 shows the fracture surface of the non-heat treated and heat treated composite. Numerous dimples of various size are seen on the fracture surface of both composites. The nucleation of
microvoids were resulting the dimples at the interface between matrix and SiC-particles and second-phase particles and/or by SiC-particles irregular multiphase particles cracking and its coalescence[12].

4. Conclusion
The effect of Heat Treatment on Mg-Al-Sr/nano-SiC to its microstructure and mechanical properties were investigated. From work discussed in this paper, the heat treatment enhances the hardness of composites. The optimum hardness is reached by the composite which contained 0.15Vf% reinforcement and treated at 425°C. The highest value of hardness is 75 BHN. Furthermore, heat treatment decreases wear rate of composites with the optimum is reached by composite with composition is 0.15Vf% reinforcement which was treated at 425°C, that is 5.46×10⁻⁶ mm³/mm. The fracture surface of the heat treated composite consisted of dimples of various sizes. The dimples were a result of the microvoids nucleation at the interface between the matrix and SiC particles and second-phase particles. Microstructural observation after heat treatment shows the existence of some phases such as α-Mg, Al₆Sr, Mg₅Si, MgAl. This result is confirmed by XRD analysis which found the existence of MgAl, MgAlSr,Mg₅Si, Mg₁₀Sr₂, SiC, SiO₂ phases.

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