The Role of Nano-SiC on Characteristics of Mg-Al-Sr/Nano-SiC Composites Produced by Stir Casting Route

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Abstract. Magnesium composite with Mg-Al-Sr matrix reinforced by nano-SiC has successfully fabricated by stir casting process. Addition of nano-SiC into magnesium matrix varies by 0.05; 0.10; 0.15; 0.20; and 0.25 in percent of volume fraction (Vf-%). The optimum mechanical properties are found in composition of 0.15 Vf-% nano-SiC. The number of hardness, impact toughness, and wear rate of this composition are 68.4 BHN, 0.065 Joule/mm², 1.033×10⁻⁵ mm³/mm respectively. Addition of 0.15 Vf-% nano-SiC enhances the hardness by 26%, impact toughness by 23.57%, and wear resistance by 38.40% respectively. Furthermore, the existence of nano-SiC in Mg-Al-Sr matrix modify the microstructure of composite by dispersing the intermetallic compounds. However, it is observed that higher nano-SiC content tends to agglomerate thus the strengthen mechanism cannot effectively occur. Microstructure analysis using OM and SEM reveals that the addition of nano-SiC transforms the dendritic matrix to globular equiaxed. EDX result predicts the phases formed are α-Mg, Al₄Sr, Mg₁₇Al₁₂, MgAlSr, and XRD analysis finds the existence of SiC, SiO₂.

Keywords: magnesium composite, Mg-Al-Sr, nano-SiC, stir casting

1. Introduction
The increasing demand for economical use of energy resources and stricter control over emissions to lower environmental effect, the industries are always seeking for new materials as alternatives to conventional materials. This is one of the greatest challenge especially for automotive industry. In this context, the use of lightweight materials is an unavoidable solution to increase energy efficiency of vehicle. Magnesium is one such promising light metals for this problem [1].

Magnesium has the high specific strength with low density and good mechanical properties[2]. The strengthening process of magnesium is commonly by adding alloys or other elements[1] since this metal has hexagonal close-packed (HCP) crystal structure which make it difficult to do a cold working process[3]. The most common element used as a magnesium alloy is aluminum. The addition of strontium to Mg-Al alloys enhances the strength at elevated temperature [4]. Compared to monolithic alloys, the addition of various types of reinforcement to magnesium alloys increases their mechanical properties such as fatigue and wear resistance [5]. SiC particles as amplifiers can be uniformly distributed and have a good SiC-matrix interface [6]. Among the various types of metal matrix composite manufacturing processes, stir casting methods are commonly used. The stirring process has an advantage because it is simple and can be used for massive production [7].
In this paper, Mg-Al-Sr/nano-SiC composite is fabricated by stir casting process. The use of Mg-Al-Sr matrix is based on Addition of nano-SiC into magnesium matrix varies by 0.05; 0.10; 0.15; 0.20; and 0.25 Vf%. Nano-SiC used since it is particularly small, and it has large surface area which enhance the possibility of strengthening mechanism. The mechanical properties and microstructures are investigated.

2. Experimental Methods

2.1. Synthesis of Mg/SiC Composites
Magnesium composite was produced through stir casting process. Al-15Sr as master alloy was added as alloying elements. Ingot magnesium (99.9 wt%) was selected as the matrix and combined with 0.05; 0.10; 0.15; 0.20; and 0.25 Vf% of nano-SiC. 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 minutes and coated with zirconia to reduce thermal shock. The Mg, Al and Sr content of all casts were analyzed using Optical Emission Spectroscopy (OES) (the chemical compositions are shown in Table 1).

| Element | 0% | 0.05% | 0.10% | 0.15% | 0.20% | 0.25% |
|---------|----|-------|-------|-------|-------|-------|
| Mg      | 96.3 | 96.1 | 96.5 | 95.8 | 95.3 |       |
| Al      | 3.54 | 3.73 | 3.83 | 3.89 | 4.48 |       |
| Be      | <0.001 | 0.001 | <0.001 | <0.001 | 0.002 | 0.001 |
| Cu      | <0.001 | 0.003 | <0.001 | <0.001 | 0.018 | 0.015 |
| Mn      | 0.0645 | 0.0591 | 0.0477 | 0.0485 | 0.0524 | 0.0259 |
| Zn      | <0.001 | 0.0345 | 0.0264 | 0.0339 | 0.0789 | 0.028 |
| Ag      | <0.001 | <0.001 | <0.001 | <0.001 | 0.0047 | <0.001 |
| Ca      | 0.0024 | 0.0031 | 0.0024 | 0.0031 | 0.0066 |       |
| Cd      | <0.001 | 0.0011 | <0.001 | <0.001 | 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. Mechanical Properties
All casts were prepared and tested to measure mechanical properties (hardness, wear resistance, impact strength). Some parts are cut into 2×3(cm) and ground to get a fine surface then tested by Brinell Hardness at room temperature. Wear test of the composite were conducted using the pin-on-disk machine by sliding a specimen under dry condition. Impact test was performed using a Charpy method at room temperature.

2.3. Metallography
All casts microstructurally characterized 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 analysis is conducted for each sample and XRD analysis is conducted to confirm the phases formed in the cast.of FT 1000 Varian instrument at 450-4000 cm⁻¹. Vibrational mode of local structure collected with by Bruker senterra of Raman spectrometer at wavenumber range 30-1560 cm⁻¹ and resolution ~3-5 cm⁻¹ using green laser (532 nm) as the source of
excitation. The reflectance properties were measured by UV-Vis Diffuse Reflectance spectroscopy (DRS) Shimadzu UV-2450 at wavelength range 100-900nm. Collected reflectance data (R) converted to Kubelka-Munk function ($F(R) = (1-R^2)/2R$). The band gap energy determined from threshold energy ($h\nu_0$) between $(F(R)h\nu)^{1/2}$ versus photon energy ($h\nu$) graph.

3. Results and discussion

3.1. Material and Microstructure
XRD Pattern of Indonesian Natural Zeolite has agreement with structural characteristic of mordenite topology (MOR Zeolite). Refinement process accomplished by using mordenite standard from IZA Structure. Refinement result revealed that mordenite’s zeolite crystallized in orthorombic lattice with Ccmc space group (Figure 1). Unit cell parameter of modernite were $a=18.1100$ Å, $b=20.5300$ Å, $c=7.5279$ Å, $a=90^\circ$, asymmetric unit ($Z$)=1 and cell volume ($V$) = 2798.8979 Å$^3$. XRD pattern of Ti$_{0.997}$V$_{0.003}$O$_2$ on zeolite at various w/w ratio presented in Figure 2 along with undoped TiO$_2$ and vanadium doped TiO$_2$ (Ti$_{0.997}$V$_{0.003}$O$_2$). Undoped TiO$_2$ shown characteristic structure of anatase phase of TiO$_2$. Several peaks related to rutile phase of TiO$_2$ was detected while vanadium dopant introduced in TiO$_2$ lattice. The rutile phase formation may led by preparing condition including the nature of vanadium dopant, sonochemical method and temperature employed.
Fig. 1 shows the phases formed based on liquidus projection in the ternary phase diagram of the Mg-4.25Al-0.75Sr alloy. Fig 2(a) and (b) show the liquidus projection of sample 0% and 0.15% nano-SiC which have the highest and lowest Sr content. The three diagrams show the intersecting line at U5-E4 (as the arrow showed in fig. 1b) with the transformation phase following the reactions (1) and (2)[8]. Based on the equation, the final phases formed are Mg_{17}Al_{12}, Mg_{58}Al_{38}Sr_{4}, α-Mg.

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\begin{align*}
U5 &: \text{L} + \text{Al}_4\text{Sr} \rightarrow \text{Mg}_{58}\text{Al}_{38}\text{Sr}_4 + \alpha-\text{Mg} \\
E4 &: \text{L} \rightarrow \text{Mg}_{17}\text{Al}_{12} + \text{Mg}_{58}\text{Al}_{38}\text{Sr}_4 + \alpha-\text{Mg}
\end{align*}
\]

Figure 3. The microstructure of Mg-Al-Sr composites with Vf% of nano-SiC (a)0.00%; (b)0.05%; (c)0.10%; (d)0.15%; (e)0.20%; (f)0.25%
The microstructure of all composites in Fig. 3 consists of α-Mg, Mg$_{17}$Al$_{12}$, MgAlSr. The Mg$_{17}$Al$_{12}$ and MgAlSr phases mainly distribute along a grain boundary of α-Mg with the morphology of discontinuous net. According to Zhang et al. [9], the two intermetallic compounds, Mg$_{17}$Al$_{12}$ and MgAlSr were found along the grain boundary. Intermetallic MgAlSr was observed in continuous network. This report is supported by Nayeri et al. [10] stated that Mg$_{17}$Al$_{12}$ in magnesium matrix found along dendritic regions in an irregular shape.

Fig. 3(a) observes in the dendrites form of the matrix. The addition of nano-SiC affects the transformation of the dendrites form into globular equiaxed as reported by Choi et al. [11]. These particles also modify the continuous intermetallic compounds into finer precipitates. Moreover, the twinning effect is found in all composites because of shear stress in the cutting process. Magnesium which has the HCP crystal structure is susceptible to twinning effect. Nano-SiC in the Mg matrix plays a role as a barrier to twinning deformation at grain boundaries, so it needs more energy to move.

Visual observation does not show the significant change of grain size. The average grain size of all samples in fig.3(a)-3(f) is 59.74, 58.32, 60.70, 50.40, 52.27 μm respectively. The constant value of grain size is caused by the agglomeration of nano-SiC, and the grain strengthening is not effectively occurred.

![Figure 4. Microstructural analysis using SEM (a)0.00% nano-SiC; (b)0.15% nano-SiC](image)

The microstructure analysis using SEM is restricted to composites with addition 0.00 Vf-% and 0.15 Vf-% nano-SiC. SEM analysis shows the dendrites formed in Mg-Al-Sr without the addition of nano-SiC in fig. 4(a). On the other hand, fig. 4(b) shows there is no dendrites phase and intermetallic compounds are finer than fig. 4(a). The average size of intermetallic compounds in composite 0.00% and 0.15% nano-SiC are 12.856 μm and 7.777 μm.

![Figure 5. FTIR spectra of mordenite zeolite, TiO$_2$, Ti$_{0.997}$V$_{0.003}$O$_2$, and Ti$_{0.997}$V$_{0.003}$O$_2$/zeolite at various composition.](image)

### Table 2. EDX Result

| Point | Mg (%6) | Al (%6) | Sr (%6) |
|-------|---------|---------|---------|
| A     | 100.00  | 0.00    | 0.00    |
| B     | 91.20   | 5.05    | 3.75    |
| C     | 88.61   | 6.95    | 4.44    |
| D     | 92.57   | 7.43    | 0.00    |
| E     | 100.00  | 0.00    | 0.00    |
| F     | 86.89   | 7.00    | 6.11    |
| G     | 86.58   | 8.57    | 4.85    |
| H     | 92.75   | 7.25    | 0.00    |
EDX analysis of point E, EDX result is presented in Table 2. predicts the compositions of point A, B, C, D are $\alpha$-Mg, Al$_4$Sr, MgAlSr, and Mg$_{17}$Al$_{12}$ respectively. The same results found at point E, F, G, and H. The present of Al$_4$Sr phase is caused by the difficulties to control cooling rate, so it occurred at the non-equilibrium stage. The agglomeration of nano-SiC is still observed in Mg-Al-Sr/0.15% nano-SiC.

XRD analysis in fig. 5 is found Mg$_{17}$Al$_{12}$ and MgAlSr as predicted in SEM-EDX result. However, XRD data shows SiC and SiO$_2$ compounds at the position of 63 and 72.5 (2θ). SiO$_2$ compound is formed on the surface of SiC within the pre-heating process before casting. Li et al. [12] reported that pre-oxidation of nano-SiC at 973-1273K for 2 hours could produce the SiO$_2$ layer on the surface. This layer could enhance the adhesion force between the matrixes and reinforce.

3.2. Hardness and Wear Resistance

As nano-SiC content increased, the hardness of cast increase as well as seen in fig. 5. After certain addition, it gives no significant effect. The highest hardness value is 68.4 HB which observed in magnesium with addition 0.15% nano-SiC. The hardness of composites increased with higher nano SiC content approximately increased 26% compare to unreinforced Mg-Al-Sr alloys with possessed 50.79 HB in hardness. This hardness related to its wear resistance, as seen at 0.15% nano-SiC generated the highest hardness therefore they have good wear resistance which is indicated by the lowest wear rate. The wear rate of unreinforced is $1.677 \times 10^{-5}$ mm$^3$/mm, then decreased to $1.033 \times 10^{-5}$ mm$^3$/mm at composites with 0.15% nano-SiC.

![Figure 6. Hardness value (a) and wear rate (b) of composites](image)

3.3. Impact Toughness

The impact toughness of composite increased with the addition of nano-SiC into a matrix as seen in fig. 6. The highest toughness is found at composite with 0.05% and 0.15% nano-SiC with the value of 0.067 and 0.065 Joule/mm$^2$. It has been reported by Cao et al. [13] that the addition of nano-SiC into Mg matrix increase the yield, tensile strength and ductility of the composite. The increase in strength and ductility indicate the enhancing of impact toughness which is in line with present work.

![Figure 7. Impact toughness of composites](image)
4. Conclusion
The effect of nano-SiC on characteristics of Mg-Al-Sr was investigated. From work discussed in this paper, conclusions can be drawn. Mg-Al-Sr/nano-SiC composite can be successfully fabricated through stir casting route. The optimum mechanical properties found in Mg-Al-Sr/0.15 Vf% nano-SiC. The number of hardness, impact toughness, and wear rate of this composition are 68.4 BHN, 0.065 Joule/mm², 1.03x10⁻⁵ mm³/mm respectively. Furthermore, microstructures observation showed the existence of some phases, α-Mg, Mg₁₇Al₁₂, MgAlSr, and Al₄Sr. Addition of nano-SiC modified the dendrite phases of α-Mg into equiaxed and refined the intermetallic compounds. XRD analysis found the existence of Mg₁₇Al₁₂, MgAlSr, SiC, SiO₂, SrO.

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