RESEARCH ARTICLE

Development of Composite Materials Using Magnesium Matrix with Variations in the Addition of Volume Fractions of Nano-Al₂O₃ Reinforcement Results of the Stir **Casting Method**

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ABSTRACT

Magnesium matrix composites were developed as a form of material selection that can save fuel use due to excess magnesium, which has very low specific gravity and still has good mechanical properties. In this study, a magnesium matrix composite with nano-Al₂O₃ reinforcement was successfully fabricated using the stir casting method. When compared with magnesium monolithic, the addition of nano-Al₂O₃ particles of 0.05, 0.10, 0.15, 0.20, and 0.25 %Vf in magnesium composite casting was investigated to improve the mechanical properties of Mg/nano-Al₂O₃ composites. Magnesium composites with 0.20% Vf reinforcement were found to be the best composition of impact price, wear rate, density, and porosity. This is because the more reinforcement given, the more mechanical properties increase, but the agglomeration tendency of nano-Al₂O₃ particles is higher so that at a composition of 0.25 %Vf, there is a mechanism anomaly because the reinforcement carried out is less homogeneous. In addition to the number of amplifiers, the improvement of mechanical properties is influenced by the fabrication process, i.e. stir casting. This study uses chemical characterization of OES, EDS, and XRD, hard damage, impact, wear, density and porosity testing, and metallographic observations using OM and SEM.

KEYWORDS

Magnesium Composite, nano-Al₂O₃, stir casting.

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1. Introduction

Indonesia is a country with population growth that has an increasing trend. This can be seen from the expansive population pyramid, which has a higher number of births than the number of deaths. This expansive population trend is the attraction of transportation-related industries, especially motor vehicles or automotive and aerospace, both abroad and domestically, to improve and develop their industries and products in Indonesia. This can be seen from the growth data of the four-wheeled market, which will increase sales in the country by 50 thousand in 2017. The Association of Indonesian Automotive Industries (Gaikindo) is confident that it will achieve the target of 1.1 million units of cars from all brands (Purnama, 2017). However, along with the increasing growth of the automotive market, so does the demand for economical use of energy/fuel sources that are difficult to renew. In addition, soaring crude oil prices and tighter controls on emissions to reduce environmental impact are fueling industries today to aggressively seek new and cutting-edge materials as alternatives to conventional materials. Soaring world crude oil prices and the use of fuels that have a difficult renewable source encourage studies related to the development of materials for transportation applications, especially automotive and aerospace, that can reduce energy and fuel consumption. One way that energy conservation in the automotive and aerospace fields is becoming a concern is the choice of light metals.

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Magnesium is the lightest metal structural material. Magnesium has a specific gravity of 1.74 g/cm3, which is lighter than aluminum, which has a density of 2.7 g/cm3, and is close compared to plastic, which is 0.97-2.17 g/cm3 [Baldwin 2004].

Metal matrixed composites made from magnesium would provide an attractive alternative to aluminum metal matrixed composites. This is because, in addition to being the lightest metal structural material, magnesium matrix composites exhibit many advantages over monolithic magnesium or magnesium alloys, such as high elasticity modulus, high strength, creep resistance and superior wear at high temperatures. However, their ductility is reduced, which limits their wide application. The desired properties can be achieved by careful selection of the type and size of reinforcing particles. So based on this exposure, in this final project, the author tries to examine composite materials with a magnesium base matrix reinforced with nano-Al₂O₃ particles using the stir casting method as a development in improving mechanical properties of cutting-edge materials for automotive or aerospace application materials as well as reducing harmful emissions, fuel consumption and energy efficiency.

2. Method

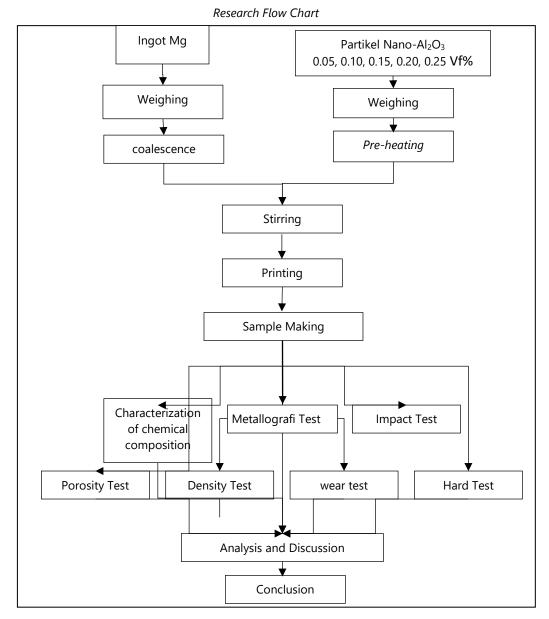


Figure 1 Research Flow Chart.

3. Results and Discussion

3.1 Analysis of Density and Porosity Test Results of Magnesium/nano-Al₂O₃ Composites with %Vf Variation of Nano-Al₂O₃ Reinforcement

Density and porosity tests were carried out to determine the effect of the casting process and the addition of nano-Al₂O₃ reinforcement on the difference in actual density and theoretical density of magnesium composites and porosity obtained by magnesium composites. The calculation of theoretical density is carried out by applying *the calculation of the Rule* of Mixture (ROM) and the calculation of actual density using the principle of Archimedes. The results of the test can be seen in Figure 4.6 and Figure 4.7.

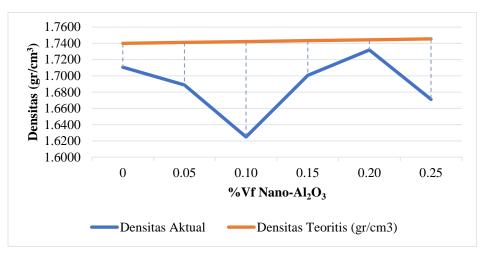


Figure 4. 1 Graph of Comparative Value of Actual Density with Theoretical Density of Nano-Al₂O₃ Reinforced Magnesium Composite with %Vf Variation of Nano-Al₂O₃ Reinforcement

From Figure 4.6 and Figure 4.7, it can be concluded that according to theory, the density produced by composite materials increases as the amount of nano-Al₂O₃ reinforcement given increases. This is due to the higher density of nano-Al₂O₃ (3.94 gr/cm3) compared to pure magnesium (1.74 gr/cm3) [Hassa et al., 2007; Hassan et al., 2008]. While the actual density is obtained at a lower level than the theoretical density. This is due to the porosity produced both from the poor casting process and the addition of the number of nano-Al₂O₃ reinforcement particles. The casting process is one of the largest porosity contributions due to the trapping of gases in the molten magnesium metal when it enters the mold due to turbulence from the pouring process. Porosity can also be seen from the resulting casting sample, namely the number of holes both on the surface and inside the magnesium composite so that it does not match its theoretical density. In addition to the poor casting process, the addition of nano-Al₂O₃ reinforcing particles also plays a role in increasing the amount of porosity because the addition of reinforcing particles in the semi-solid state and proper stirring causes an increase in viscosity and a decrease in melt fluidity. Gas bubble trapping causes porosity to increase with an increase in the percentage of reinforcement volume (Aravindan et al., 2015). In addition, it is also due to the low wetability and agglomeration tendency in the number of nano-Al₂O₃ reinforcement, increase porosity due to the presence of interstitial cavities in the agglomeration pool (Yadav et al., 2017).

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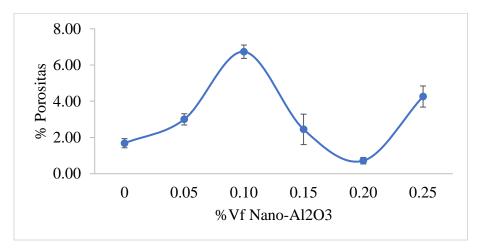


Figure 4. 2 Graph of Actual Porosity Amount in Magnesium and Nano-Al₂O₃ Reinforced Magnesium Composites with %Vf Variation of Nano-Al₂O₃ Amplifier

3.2 SEM-EDS-Mapping Analysis of Magnesium/nano-Al₂O₃ Composites with %Vf Variation of Nano-Al₂O₃ Reinforcement

Tests for SEM-EDS analysis were carried out on samples derived from magnesium without reinforcement and Mg/nano-Al₂O₃ composites that had optimum and minimum mechanical and physical properties values to be compared, namely in composites with %Vf nano-Al₂O₃ of 0.15%, 0.25%, and 0.05%. Shooting using SEM was carried out with two shots at 500x magnification and once at 1000x magnification, shown in figure 4.19, figure 4.20, figure 4.21, and figure 4.22, to clarify the topography to be observed. Then, EDS testing was carried out with the aim of determining the possibility of elements formed in the phase seen in this Mg/nano-Al₂O₃ composite, in reference to the results of the previously carried out element mapping.

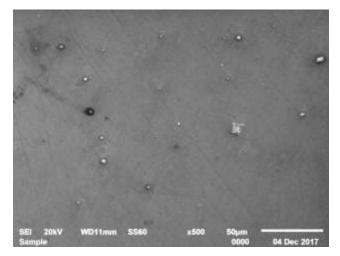
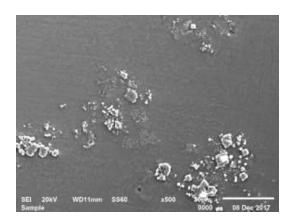


Figure 4. 3 Results of SEM Magnesium Without Reinforcement and Mg/Nano-Al2O3 Composite Using 500x Magnification.

When compared with nano-Al₂O₃-reinforced magnesium in Figures 4.18, 4.19, and 4.20, magnesium materials without reinforcement showed no collection of nano-Al₂O₃ reinforcing particles.



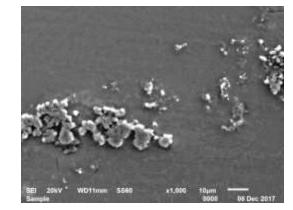
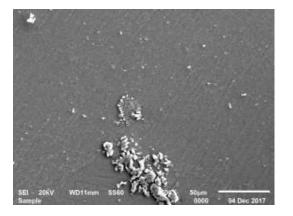


Figure 4. 4 Results of SEM Composite Mg/nano-Al₂O₃ with Amplifier of 0.05 %Vf Using 500x (left) and 1000x (right) magnifications.



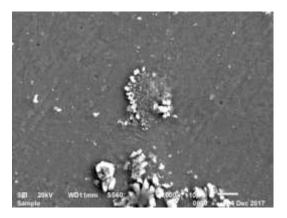
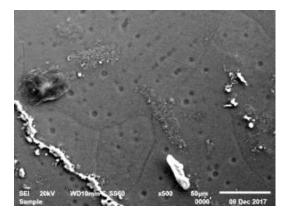


Figure 4. 5 Results of SEM Composite Mg/nano-Al₂O₃ with Amplifier as Much as 0.15 %Vf Using Magnification 500x (left) and 1000x (right).



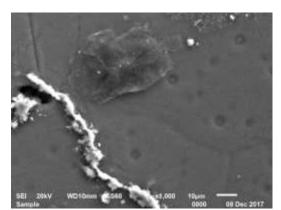


Figure 4. 6 Results of SEM Composite Mg/nano-Al₂O₃ with Amplifier of 0.25 %Vf Using 500x (left) and 1000x (right) magnification

This is in accordance with the previous research conducted by M. Gupta et al. in Figure 4.21. Meanwhile, the results of the Mg/nano-Al₂O₃ composite SEM in Figures 4.18, 4.19, and 4.20 all found white objects indicated as a collection of reinforcing particles and magnesium oxide reacted during casting. It is this collection of reinforcing particles that makes the improvement of mechanical properties not so significant. The phenomenon of collecting reinforcing particles is caused by poor methods and parameters of stirring and *pretreatment* of nano-Al₂O₃ reinforcing particles.

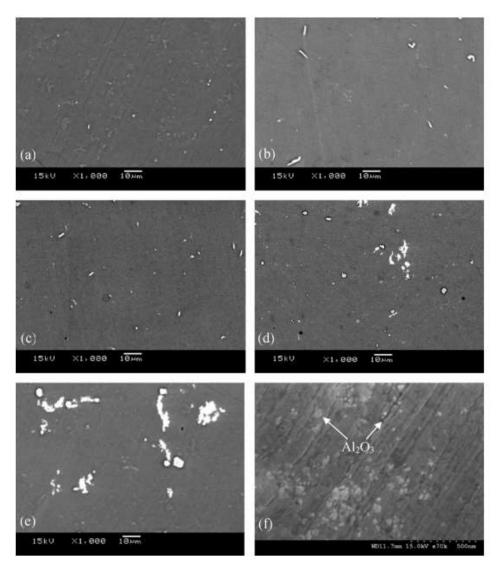


Figure 4. 7 SEM Results Show Second Phase Distribution and Reinforcement Particles on (a) AZ31, (b) AZ41, (c) AZ51, (d) AZ41-Al2O3 and (e) AZ51-Al2O3 Samples. (f) FESEM Results of Nano-Sized Alumina Distribution in AZ41-Al₂O₃ Samples (Hassan &; Gupta, 2007)

Figure 4.22 shows the results with 500x magnification on the entire sample with 4 arrows as a clue to the location to be carried out EDS. Before EDS is carried out, element mapping is carried out first to determine the distribution of elements at that location, which can be seen in Figure 4.23.

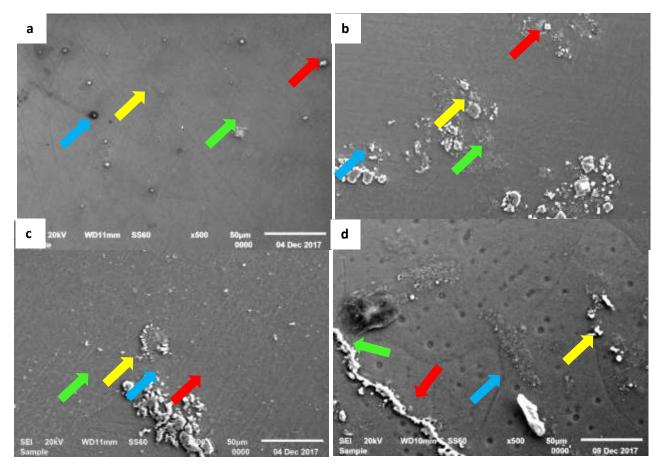


Figure 4. 8 SEM Results on (a) Magnesium Without Reinforcement and Mg/Nano-Al₂O₃ Composite with Amplification as Much as (b) 0.05 %Vf, (b) 0.15 %Vf, and 0.25 %Vf Using 500x Magnification

The results of the EDS test can be seen in Table 4.4 below. Based on the EDS results in all samples, almost the main elements were found, namely Mg, Al, O, and C. Mg, Al and O elements are predicted as a result of the formation of phases MgO, Al_2O_3 , and Mg2Al2O4 derived from the reaction between nano- Al_2O_3 reinforcement particles with Magnesium based on reactions (4.1) and (4.2) (Lloyd, 1994) and as a reaction of molten magnesium with highly oxidative oxygen gas to form MgO.

3 Mg + Al2O3 ↔ 3MgO + 2Al		(4.1)
3 Mg + 4Al2O3 ↔ 3Mg Al2O4 + 2Al	(4.2)	

While element C is predicted from inclusions derived from the fall of the crucibels used, namely graphite, the crucibels used fall out due to the unsuitable conditions of metal melting with crucibel properties that are alkaline, while molten magnesium tends to be acidic. The crucibel condition is also porous, allowing slag penetration from the magnesium fuse to enter the crucible and react to erode the crucibels and mix into the metal melt.

Matarial	A	Element Content (wt.%)						Possible Phases
Material	Arrow	Mg	To the	Or	Yes	Cr	С	
Mg	Blue	80.46	-	3.50	-	-	16.05	MgO
	Yellow	94.12	-	2.07	-	-	3.81	MgO
	Green	88.15	-	7.56	0.24	-	4.05	MgO
	Red	68.82	4.51	19.19	0.24	-	7.44	MgO, Al ₂ O _{3,} MgAl ₂ O
Mg/0.05 %Vf Nano-Al ₂ O ₃	Blue	49.95	14.39	6.25	0.66	-	4.96	MgO, Al ₂ O _{3,} MgAl ₂ O
	Yellow	66.95	-	24.57	0.31	-	8.15	MgO
	Green	87.99	-	8.21	-	0.31	3.66	MgO
	Red	50.53	-	41.10	0.07	-	8.15	MgO
Mg/0.15 %Vf Nano-Al ₂ O ₃	Blue	81.63	-	14.22	0.12	0.03	4.01	MgO
	Kuning	58.23	1.28	34.84	0.43	-	4.78	MgO, Al ₂ O _{3,} MgAl ₂ O ₄
	Green	84.98	0.90	5.52	0.10	-	8.50	MgO, Al ₂ O _{3,} MgAl ₂ O
	Red	89.43	-	0.82	-	-	9.75	MgO
Mg/0.25 %Vf Nano-Al ₂ O ₃	Blue	59.49	-	39.17	-	0.02	1.32	MgO
	Yellow	59.23	-	39.0	-	0.03	1.74	MgO
	Green	52.94	6.29	40.62	0.15	-	-	MgO, Al ₂ O _{3,} MgAl ₂ O,
	Red	59.29	-	39.08	0.04	0.01	1.58	MgO

Table 1 EDS Results on Magnesium and Magnesium Composites Reinforced 0.05, 0.15, and 0.25%Vf Nano-Al2O3, 500xMagnification.

Based on Figure 4.23, the results of element mapping in the four samples show that the distribution of Al and O elements is only present in certain parts and not spread across the SEM observation surface. This confirms that the agglomeration phenomenon of nano-Al₂O₃ reinforcing particles occurs and supports the cause of the lack of effectiveness in improving the mechanical properties of Mg/nano-Al₂O₃ composites.

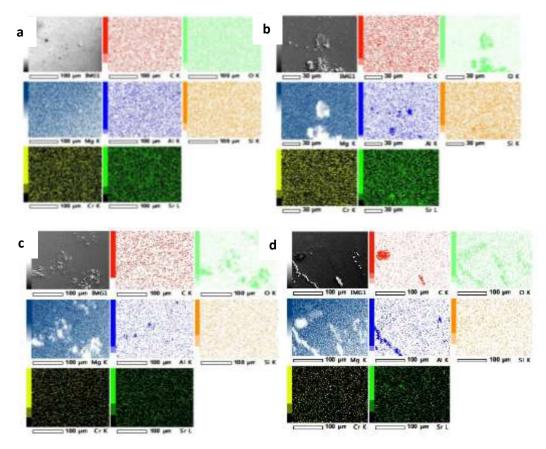


Figure 4. 9 Results of Element Mapping on (a) Magnesium Without Reinforcement and Mg/Nano-Al₂O₃ Composites with Amplifiers as Much as (b) 0.05 %Vf, (b) 0.15 %Vf, and 0.25 %Vf Using 500x Magnification

3.3 Analysis of XRD Test Results of Magnesium/nano-Al₂O₃ Composites with %Vf Variation of Nano-Al₂O₃ Reinforcement XRD testing on Magnesium composites reinforced with nano-Al₂O₃ particles was carried out on samples with nano-Al₂O₃ particle composition of 0.15%Vf, where the sample had the highest tensile strength value. To process the data generated after testing, X'Pert HighScore Plus software is used to see the phases indicated to appear in the composite sample. Table 4.5 shows a list of detected phases along with the scores for each.

No.	Ref. Code	Score	Compound Name	Chemical Formula
1	98-016-6274	20	Magnesium Aluminium Oxide (0.25/0.5/1)	MgAl ₂ O ₄
2	96-901-3272	27	Periclase	MgO
3	98-016-1061	1	Aluminium Oxide	Al ₂ O ₃
4	98-005-3767	27	Magnesium	Mg

Tabel 4. 1	Identifikasi Sen	yawa pada Sam	npel Komposit Ma	agnesium Berper	guat 0.15%Vf Nano-Al ₂ O ₃
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Based on the *score* figures in Table 4.5, it can be seen that the most compounds detected are the MgAl₂O₄ phase and periclase or MgO. This condition is in accordance with the previous analysis, where the phases MgAl₂O₄ and MgO will form a lot because these two compounds are stable compounds formed when reacting with Mg according to equations 2.1 and 2.2. In addition, the element Mg is very reactive and easily reacts with Oxygen to form MgO as a compound that is widely found. In addition, the persistence of the Al₂O₃ phase, even in small amounts, indicates that the distribution of reinforcing particles is not good and tends to collect in a certain area or agglomerate as previously predicted and described. Figure 4.24 shows the top ten peaks of the XRD results of a Mg/0.15%Vf nano-Al2O3 composite sample.

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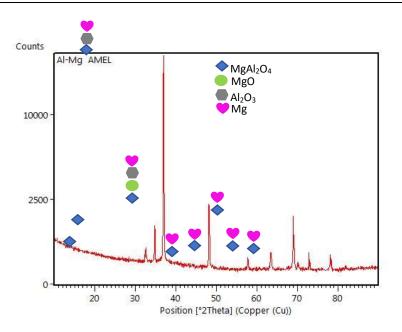


Figure 4. 10 XRD Test Results on Magnesium Composites with the Addition of 0.15%Vf Nano-Al₂O₃ Booster.

Furthermore, a comparison between *the peak list* of composite samples and *reference peaks* can be seen in Figure 4.25. The peak comparison is then complemented by a pattern identification analysis that will produce a *score*. *The score* can be used as a reference to determine the indication of the existence of a phase. The higher the score obtained in a phase, it can be indicated that the existence of the phase is better.

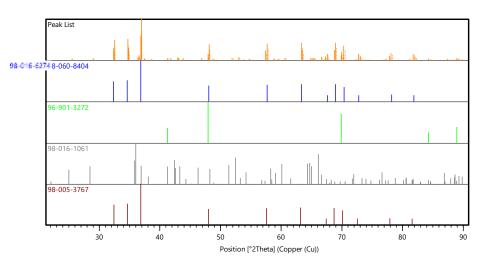


Figure 4. 11 Comparison between *Peak List of* 0.15%Vf Nano-Al₂O₃ Reinforced Magnesium Composite Samples with *Reference Peak*

4. Conclusion

Unstable stirring speeds and less stirring times result in inhomogeneous dispersion of reinforcement particles and turbulenceprone conditions and leave more gases trapped in the composite sampling process, resulting in high amounts of porosity and insignificant increases in mechanical strength.

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Conflicts of Interest: The authors declare no conflict of interest.

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