

Fabrication and characteristics of ADC-12 reinforced nano-SiC and nano-Al₂O₃ composites through stir casting route

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Abstract. Aluminum ADC-12 reinforced with nano-SiC and nano-Al₂O₃ have been successfully produced by stir casting method. The effect of addition nano SiC and Al₂O₃ into matrix has been studied with variation of 0.05, 0.10, 0.15, 0.20, 0.30 vf-% for each composites. The addition of Mg was introduced with 10 wt-% to promote wetting between molten Al and ceramic particle reinforcement. The OES result shows decreasing amount of excess Si and Mg in the composite compared to as-cast ADC-12. One of the main intermetallic phases present evenly in aluminum matrix is Mg₂Si which was confirmed by EDS and XRD. The highest tensile strength and hardness of composites were achieved by the addition of 0.15 and 0.30 %vf SiC and Al₂O₃, respectively, with the values of 111 and 115 MPa and 42 and 41 HRB. The increasing hardness generated significant improvement in wear resistance of composites. The higher the reinforcement content, the higher the porosity formed, due to the tendency of dewetting as well as nano particles agglomeration.

1. Introduction

Aluminum and its alloys are metals that are presently widely used in industries. There are around 8.1% aluminum in Earth's crust alone, making it the most abundant metal and third most abundant element after oxygen and silicon. Compared to other metals, aluminum is economically preferred in many industries, due to its light weight, ease of fabrication, good physical and mechanical properties, and good resistance to corrosion. The ADC-12 aluminum alloy, known as A383.0 in ANSI (American National Standards Institute) standard, is commonly used in transportation due to good cast-ability, low density, low chance of shrinkage and high strength [1, 2] due to its main alloying elements which are Si and Cu.

A composite is a system which is composed of reinforcement constituent distributed in a matrix which then makes up combined properties on the composite, taking specific properties from both the reinforcement and the matrix [3]. Good ductility of aluminum combined with good hardness of ceramic reinforcement results in a composite having excellent mechanical properties [4]. Two types of materials that are commonly used as particulate reinforcement in aluminum matrix are Al₂O₃ and SiC. Both of them can be used in many sizes, and nano-sized particulates have been used in recent studies to enhance the hardness properties in aluminum matrix using only small amount of reinforcement particulates. Manu [5] and Mazahery [6] shows that adding nano-sized reinforcement particulates; whether SiC or Al₂O₃, increase the mechanical properties in aluminum based composites.



Ceramics like Al_2O_3 and SiC particles generally have poor wettability on aluminum matrix. Therefore, it is needed to add magnesium as wetting agent to improve the wettability of matrix to generate the formation of MgAl_2O_4 phase. This spinel decrease the surface tension of molten aluminium to improve the wettability between particles reinforced and aluminium matrix [7]. In this work, effect of Al_2O_3 and SiC nano particles addition on ADC12 matrix to the mechanical properties of the composite are studied.

2. Experimental method

Variation of reinforcement particles of nano-SiC and nano- Al_2O_3 used were 0.05, 0.1, 0.15, 0.2, and 0.3 vf-%. Aluminum was melted at $800^\circ\text{C}^{\pm 20}$ in the electric furnace, then 10 wt-% Mg was added to increase wettability, followed by one minute stirring and 2 minutes degassing process using argon (Ar). Meanwhile, reinforcement particles were pre-heated for 1 h at $800^\circ\text{C}^{\pm 20}$ to remove moisture, then added to liquid matrix, followed by 1 minute stirring at ± 600 rpm, and then by 2 minutes degassing again. Casting process was conducted into a mold which were pre-heated at $500^\circ\text{C}^{\pm 20}$ for ± 8 minutes and coated at the insides using water-based zirconia sand coat to increase mold's thermal resistance and prevent products stick to the mold.

The composites were characterised by both microstructural analysis and mechanical properties analysis. Their chemical compositions were tested using Optical Emission Spectroscopy (OES); then, they were prepared by metallography procedures to analyse their microstructure using optical microscope and Scanning Electron Microscope (SEM) linked to Energy Dispersive Spectroscopy (EDS) to analyse phases present in composites and confirmed by X-Ray Diffraction (XRD). Mechanical tests such as tensile, hardness, wear, and density-porosity test were also performed, using the ASTM E8 standard for tensile test, ASTM E18 for hardness test with Rockwell B, wear test using Ogoshi refers to ASTM G99, and density-porosity test using Archimedes method.

3. Results and discussion

3.1. Chemical composition of ADC-12/ nano SiC and ADC 12/ nano Al_2O_3 composites

Examination results of chemical composition using OES of unreinforced ADC 12, ADC 12/0.15% Vf SiC and ADC 12/0.30% Vf Al_2O_3 is presented in Table 1. There were differences in chemical composition between ADC-12 and both reinforcing with SiC and Al_2O_3 .

Table 1. The chemical composition of the composite using OES.

Materials	Elements (at.%)						
	Al	Si	Fe	Cu	Mn	Mg	Zn
ADC 12	84.80	10.50	0.864	2.33	0.223	0.221	0.637
Composite SiC	77.80	7.16	0.821	1.01	0.196	> 12	0.518
Composite Al_2O_3	77.90	7.17	0.827	1.05	0.191	>12	0.520

Mg content was intentionally added at about 10 wt.% into a matrix to avoid evaporation because operation condition of melting furnace was at elevated temperature ($800^\circ\text{C}^{\pm 20}$). However, no evaporation effect of Mg occurred; even on the final composite sample, Mg content exceeds up to 12 wt.%. The presence of Mg affects the formation of the MgAl_2O_4 spinel phase and tends to form Mg_2Si . Mn content of 0.191% by weight is likely to form an intermetallic phase of α -AlMnSi or better known as α -cubic [8]. This phase was considered might lead material into brittle structure especially when the content is high.

The presence of Mg_2Si phase which is clearly seen in the microstructure is confirmed through XRD testing of the as-cast composite. Mg will react with Si to form Mg_2Si phase during solidification process and takes several forms: primary, secondary and tertiary. In addition, the presence of other phases is also confirmed by XRD testing as seen in Figure 1.

The addition of Mg pushes oxygen to create a bonding MgO which subsequently binds with the aluminum oxide coating on the Al₂O₃ and SiC to form a spinel layer (MgAl₂O₄) due to following reaction [7] :

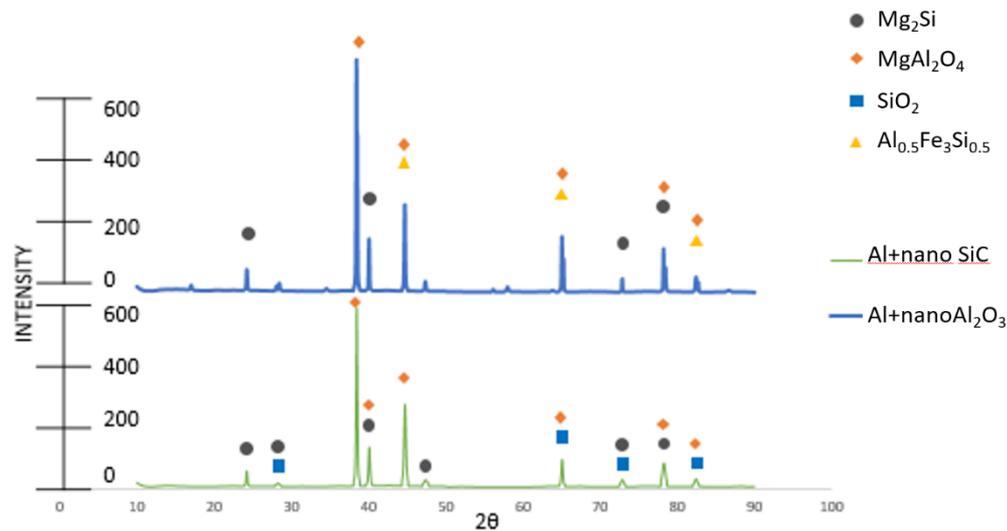


Figure 1. XRD pattern of ADC-12 reinforced nano-SiC and nano-Al₂O₃ composites.

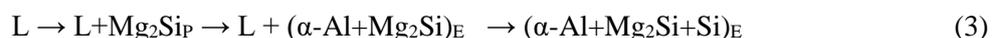
Reaction (1) commonly occurred with SiC and reaction (2) with Al₂O₃ particles. For some cases, reaction (2) may occur on SiC reinforcement, in which the Al₂O₃ comes from oxidized Al melt surface that sticks to SiC particles when the particles were added. Spinel affluence the infiltration of molten aluminium into reinforcing particle so that effectiveness of wettability can be achieved.

3.2 Microstructure analysis of ADC-12/ nano SiC and ADC-12/ nano Al₂O₃ composites

The microstructure of unreinforced ADC-12 analysed by optical microscope showed needle-like shaped aluminum, which makes it hard to analyse the grain boundaries. The Si element on ADC-12 is eutectic, but during the process, the addition of nano Al₂O₃ and SiC reduce the presence of Si. It is proven by the results of the OES in Table 1, wherein the original Si content of 10.5 wt.% in ADC-12 was reduced to about 7% in the composition of composites with 0.15 vf-% SiC and 0.30 vf-% Al₂O₃ respectively, because Si element binds Mg to form Mg₂Si compound in various forms and is not in a free eutectic state.

Nano SiC and Al₂O₃ will reduce grain size as they act like a grain refiner. As the content of reinforcer increases, the aluminum grain gets finer. Figure 2 shows various appearances in microstructures which are signed by the arrow. The arrow 1 indicates shrinkage porosity, arrow 2 is a β -Al₅FeSi phase, arrow 3 is a π -Al₈Mg₃FeSi₆ phase, arrow 4 is gas porosity, and arrow 5 is particle porosity.

Mg content of up to 12 wt.% in a chemical composition will lead shifting in a major constituent of a matrix into Al-Mg-Si which results in the formation of Mg₂Si. According to Li *et al.* [9], the Mg₂Si phase can be formed in several morphologies: primary Mg₂Si with polyhedral or dendritic morphology, binary eutectic Mg₂Si in flake-like form surrounded by α -Al, and ternary eutectic Mg₂Si which has a small round shape between binary eutectic phases Mg₂Si, thus forming an archipelagic region together with a binary eutectic Mg₂Si phase. The formation of Mg₂Si follows reaction (3) as follows:



Primary Mg_2Si can improve the mechanical properties of composite [10, 11] due to its morphology: blocky or snow flake with sharp edges, which can induce localized stress concentration that might increase the tensile strength of the composite. The Fe content which reaches 0.80 wt.% causes Fe to react to form an intermetallic phase by the reaction:

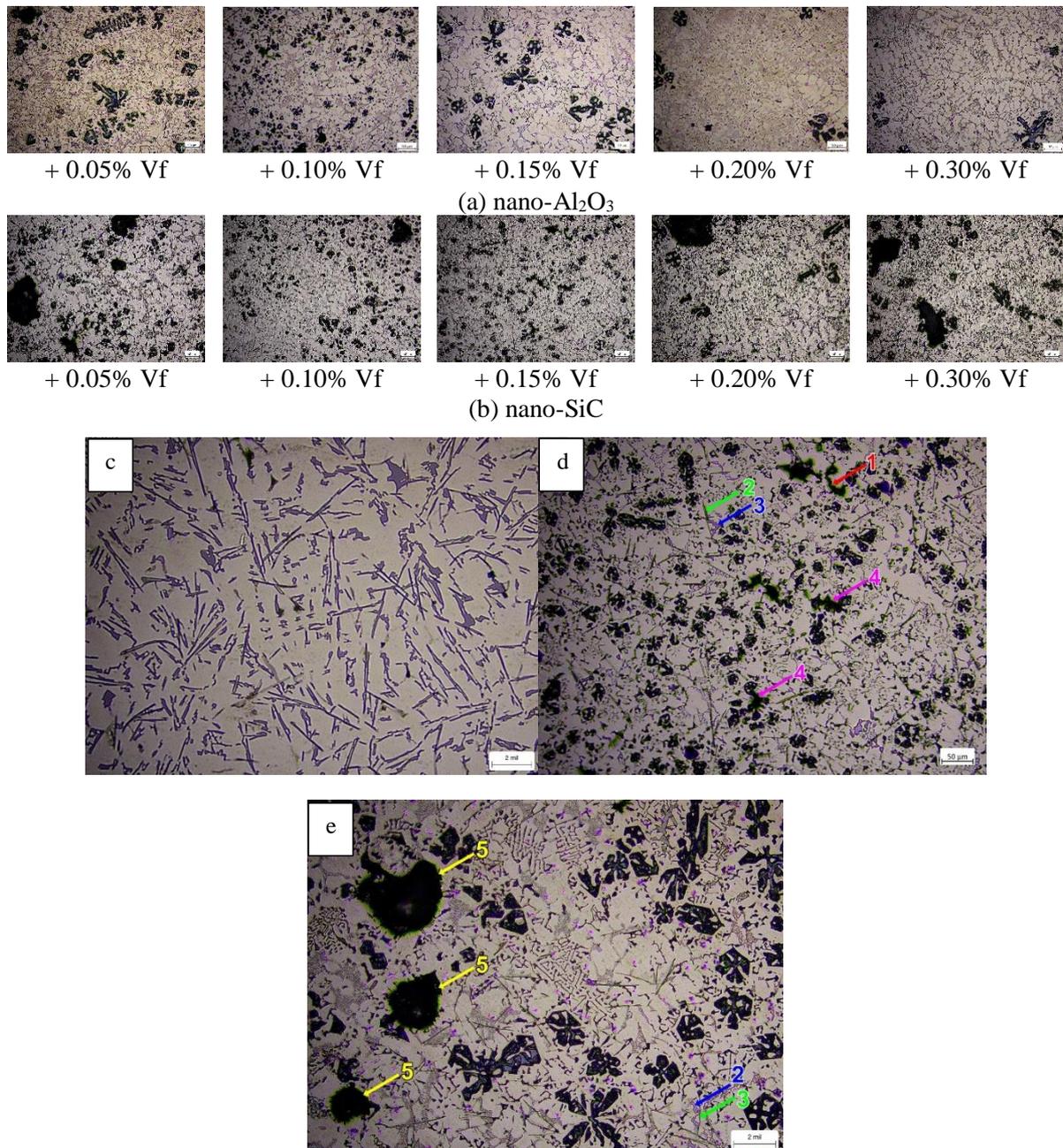


Figure 2. Microstructure of composites; (a) ADC-12/nano-Al₂O₃, (b) ADC 12/nano-SiC, (c) ADC 12 (d) ADC 12 + 0.15% Vf SiC, and (e) ADC-12 + 0.30% Vf Al₂O₃. Keller etchant, 10s.

The β - Al_5FeSi phase will have a needle/plate-like morphology. The higher Mg content, the greater the possibility of quaternary phase which is π - $\text{Al}_8\text{Mg}_3\text{FeSi}_6$ formed after Mg_2Si [12]. The further observation of these phases is shown with SEM link to EDS in Figure 3 and indicated points within the Table 2 and Table 3 are also depicted from Figure 3.

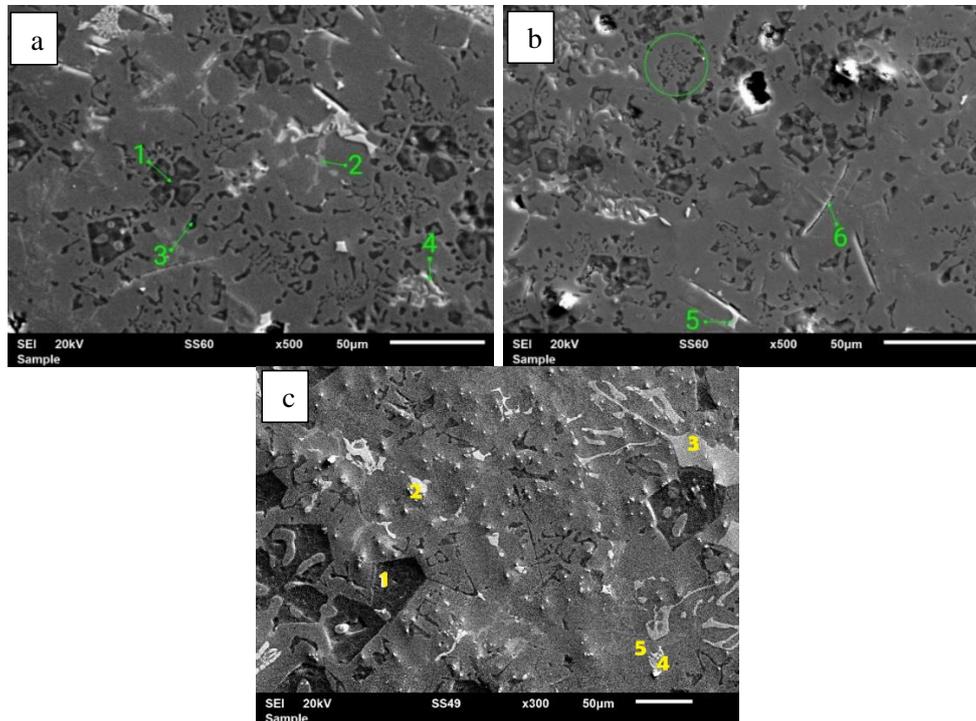


Figure 3. SEM images of ADC-12 with 0.15vf-% SiC composite at magnification of (a) 500x (b) 3000x and (c) ADC-12 with 0.30 vf-% Al_2O_3 composite.

Table 2. EDS result on a composite of ADC-12 with 0.15 vf-% nano-SiC.

Point	Amount of Element (at.%)						Possible phase formed
	Al	Mg	Si	Fe	Cu	O	
1	84.86	1.60	6.08	-	-	7.46	Mg_2Si (primary)
2	86.37	1.92	2.79	3.71	2.44	2.77	π - $\text{Al}_8\text{Mg}_3\text{FeSi}_6$
3	18.32	32.53	26.54	1.38	-	15.11	$\text{Al-Mg}_2\text{Si}$ (binary)
4	12.80	36.46	27.75	-	-	17.27	$\text{Al-Mg}_2\text{Si}$ (binary)
5	80.41	-	-	-	8.59	-	Al_2Cu
6	64.44	1.43	9.73	7.00	-	6.14	β - Al_5FeSi

Table 3. EDS result on a composite of ADC-12 with 0.30 vf-% nano- Al_2O_3 .

Point	Amount of Element (at.%)						Possible phase formed
	Al	Mg	Si	Cu	Fe	C	
1	-	28.78	28.74	-	-	12.37	Mg_2Si (Primary)
2	52.11	-	-	30.98	-	11.16	Al_2Cu
3	60.48	-	6.96	-	18.98	-	Al-Fe-Si
4	67.10	4.61	4.64	23.64	-	-	Al_2Cu
5	63.10	10.84	19.97	-	6.09	-	$\text{Al} + \text{Mg}_2\text{Si}$ (Binary)

3.3. Mechanical properties of ADC 12/nano SiC and ADC 12/nano Al₂O₃ composites

Figure 4 shows the Ultimate Tensile Strength (UTS), for both composites with SiC and Al₂O₃ reinforcement. The UTS of composites greater than that of its matrix. ADC-12 unreinforced has UTS of 101 MPa, while composites with 0.15 vf-% SiC is 111 MPa and 115 MPa for composites with 0.30 vf-% Al₂O₃.

The strengthening mechanism at work in the application of SiC and Al₂O₃ nano-sized reinforcement are load transfer into reinforcer, dislocation density, Hall-Petch and Orowan Strengthening [13, 5]. A decline of UTS is predicted to be the result of agglomeration of nano particles. These particles have a greater surface-to-volume-ratio, leading to agglomeration with each other. Moreover, the increase of porosity level further enhanced by agglomeration of SiC particles leads to the decline of UTS [6]. As the porosity level increases, more crack initiation presents to ease the failure take place.

Generally, Figure 5 shows an increased hardness number with the addition of SiC and Al₂O₃ and decreased wear rate. The nano-sized particles have significantly higher hardness compared to the matrix and make it helpful to harden the composite. In addition, reinforcers act as a barrier during indentation so that higher force is needed to perform plastic deformation. Nonetheless, higher porosity level contributed from the higher content of reinforcing responsible for declining hardness of the material.

The wear rates of SiC- and Al₂O₃-reinforced composites are given in Figure 6. Unreinforced ADC-12 has a wear rate of $2.51 \times 10^{-5} \text{ mm}^3/\text{m}$ where the most-reduced wear rate is down to $0.98 \times 10^{-5} \text{ mm}^3/\text{m}$ and $1.08 \times 10^{-5} \text{ mm}^3/\text{m}$ that are reached by addition of 0.1 vf-% SiC and 0.3 vf-% Al₂O₃, respectively. The decreased wear rate corresponds to the presence of nano particles reinforcement as they created materials harder followed by slight material's cut [14]. To define wear resistance, the hardness is a profound characteristic to be considered. There is a increased of wear rate due to poor microstructure homogeneity leads to undispersed reinforcement, higher level porosity and weak interface bonding between matrix and reinforce [15].

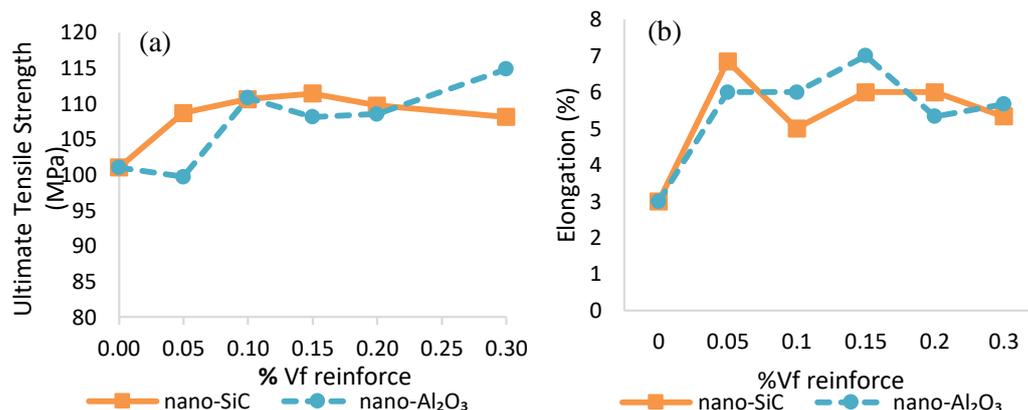


Figure 4. Graphic of (a) UTS and (b) elongation of ADC-12 with an addition of nano-SiC and nano-Al₂O₃ in various compositions.

Generally, the data obtained by impact testing shown in Figure 6 (a), the impact toughness inclined to decrease with the addition of volume fraction of nano-reinforce. Reinforcement hardens matrix material and then decreases impact strength because the material will be broken immediately without absorbing much energy [16]. Current work is in line with the research conducted by Bekir *et al.* [17]. The addition of Al₂O₃ or SiC will lower impact toughness of composite aluminum. However, the increase of impact toughness might result from good distribution of nano particles inside matrix which causes good interface bonding between them [18].

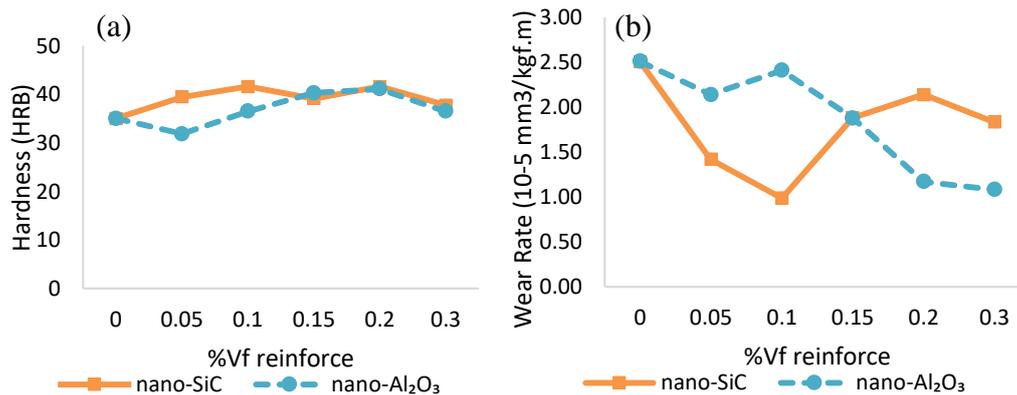


Figure 5. Graphic of (a) hardness and (b) wear rate of ADC-12 with an addition of nano-SiC and nano-Al₂O₃ in various compositions.

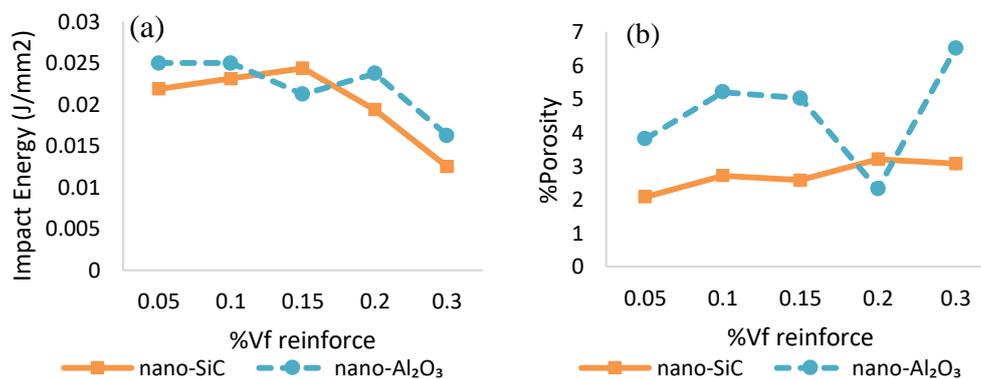


Figure 6. Graphic of (a) impact toughness and (b) % porosity level of ADC-12 with an addition of nano-SiC and nano-Al₂O₃ in various compositions.

3.4. Density and porosity of ADC 12/ nano SiC and ADC 12/ nano Al₂O₃ composites

The density of composite is calculated using Archimedes' test. As-cast composite will have lower density compared to theoretical density due increasing in weight percent of nano particle. In this work, it can be seen in Figure 6 (b). Hydrogen composition arises when metal melting on a temperature fabricated around 800°C^{±20} during stir casting. Even though argon degassing had been applied, hydrogen is remaining in molten aluminum because argon less protective to prevent hydrogen infiltrate into molten aluminum particularly in the open melting furnace [19]. Various kinds of porosity which are present in the microstructure are gas, shrinkage and particle porosity; they significantly reduce the composite's density.

4. Conclusion

Composite ADC-12 with variation of addition 0.05, 0.10, 0.15, 0.20, 0.30% Vf nano-sized SiC and Al₂O₃ were successfully fabricated using stir casting method. The excess of Mg content influences the formation of Mg₂Si which reduces the effectiveness in the formation of spinel layer. In addition, the Fe content is high enough to form an intermetallic phase that affects the mechanical properties of composites. Both of these elements can be seen through SEM-EDS testing. Addition of 0.15vf-% SiC and 0.30 vf-% Al₂O₃ give the optimum UTS of 111 MPa and 115 MPa, respectively. The Orowan mechanism is being the major strengthening mechanism in terms of nano-sized particles addition. The decline of UTS might be a result of agglomeration of reinforcement particles or porosity. The maximum hardness gained by adding of SiC up to 0.1 vf-% is 42 HRB and Al₂O₃ up to 0.2 vf-% is 41 HRB,

respectively. However, as the content of reinforcement arise, the possibility of porosity in the matrix increase, thus decreasing the hardness. Nano-sized SiC and Al₂O₃ were significantly reducing wear rate of an unreinforced matrix as materials become harder and reducing volume of material loss. Increased wear rate might be caused by inhomogeneity of microstructures, and impact toughness reduced due to increasing hardness and decreasing ductility of composites.

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