

## THE MECHANICAL PROPERTIES OF $\text{Al}_2\text{O}_3$ -REINFORCED ALUMINUM A356 WITH GRAIN REFINER Al-5Ti-1B FABRICATED USING THE STIR CASTING METHOD

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### ABSTRACT

$\text{Al}_2\text{O}_3$  reinforced aluminum A356 has been successfully fabricated using the stir casting method. The development of current technology requires a material that is light, strong, tough, and corrosion and wear resistant, in addition to various other advanced properties. A composite material was therefore developed. Composite materials can be used in a wide range of strategic sectors such as the automotive, military, aerospace, and electrical industries. This study aims to develop a composite material that consists of aluminum A356 as the matrix and micro  $\text{Al}_2\text{O}_3$  as the reinforcement, with 8 wt% magnesium as the wetting agent with the addition of grain refiner TiB at 0; 0.01; 0.0347; 0.0362; 0.0622; and 0.0689 wt% using the stir casting method. The material characterization comprises tensile testing, hardness testing, wear testing, chemical composition testing (OES and XRD), and microstructure testing (OM, SEM, and EDX). The test results revealed that the addition of 0.0347 wt% TiB was capable of reducing the size and changing the shape of a long and coarse grain to become round and fine, thereby significantly increasing its tensile strength, hardness, and wear resistance, but decreasing the elongation and ductility.

*Keywords:*  $\text{Al}_2\text{O}_3$ ; Aluminum A356; Composite; Grain refiner TiB; Stir casting

### 1. INTRODUCTION

Current technology requires materials that are light, strong, tough, and corrosion, wear, and high temperature resistant, in addition to various other advanced properties. As a result, the need exists for composite materials, which are a combination of several types of material. Such composite materials can be used in a wide range of strategic sectors that require high performance, including the automotive, military, aerospace, and electrical industries. As a metal, aluminum and its alloys can be used as a composite matrix due to the advantages of being light in weight and easy to combine with other elements to obtain certain mechanical properties, in addition to its high elasticity, corrosion resistance, and good electrical conductivity. It is also anti-magnetic and has good machinability and a low melting point, thus making it easy to recycle and reuse. Pure aluminum and some aluminum alloys have sufficient strength and low hardness. To gain optimum strength, hardness, and wear resistance, it is necessary to strengthen it with a harder material such as ceramic material. Ceramic material is generally used to reinforce chemical compounds such as SiC,  $\text{Al}_2\text{O}_3$ , MgO,  $\text{B}_4\text{C}$ , TiC, BN, and  $\text{ZrO}_2$  conducted research on the effect of reinforcing with the addition of SiC (50  $\mu\text{m}$ ),  $\text{Al}_2\text{O}_3$

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(60  $\mu\text{m}$ ), and MgO (50  $\mu\text{m}$ ) to varying degrees from 0 wt% to 20 wt% of the aluminum matrix (Kheder et al., 2011). The results revealed that the tensile strength and hardness of materials increased significantly from 80 MPa to 140–160 MPa and from 25 HB to 35–45 HB, but that the ductility of the material decreased. Singh et al. (2012) investigated the effect of the addition of 2.5, 5, 7.5, and 10 wt% Al<sub>2</sub>O<sub>3</sub> particles on the mechanical properties of composite aluminum LM 6 using semisolid casting (Singh et al., 2012). The addition of Al<sub>2</sub>O<sub>3</sub> particles increased the tensile strength, hardness, and impact resistance significantly, but decreased elongation and ductility. The optimum mechanical properties were the addition of 10 wt% Al<sub>2</sub>O<sub>3</sub> with a yield strength of 140 N/mm<sup>2</sup>, rockwell hardness of 95.8 with a load of 200 grams for 20 seconds, and an impact force of 7.22 Nm.

Besides combining it with reinforced particles, the strength and hardness of aluminum composite can be improved by adding grain refiner. The utilization of Titanium (Ti) as a grain refiner in the aluminum alloy casting process is often combined with Boron (B) in small quantities. Ti and B together form a very effective TiB<sub>2</sub> compound as a grain refiner. Li et al. investigated the effect of the ratio of Ti/B on the grain refining ability of Al-Si alloys with variations of Al-6Ti, Al-5Ti-1B, and Al-2.5Ti-2.5B grain refiner (Li et al., 1997). The ideal ratio of Ti/B for forming the TiB<sub>2</sub> compound as a nucleating agent is 2.2, but this ratio is rarely used in commercial grain refiner. Generally, a higher Ti content is used to obtain finer grains. TiB<sub>2</sub> is a compound that is stable and does not dissolve in liquid aluminum, which makes it very effective in the nucleation phase of primary Al. The results showed that Al-2.5Ti-2.5B is an optimum grain refiner with a Ti content of 0.03 wt%. The addition of 0.2 wt% Al-5Ti-1B reduces the size of Mg<sub>2</sub>Si but does not alter its still dendritic, whereas the addition of 1 wt% Al-5Ti-1B is capable of reducing the size of Mg<sub>2</sub>Si from 100  $\mu\text{m}$  to 20  $\mu\text{m}$  and modifying the original dendritic shape into one that is polygonal (Li et al., 2008).

Stir casting is a metal composite forming method that is simple, flexible, inexpensive, and suitable for use on a massive scale (Hashim et al., 1999; Zulfia et al., 2017). Precipitation or settling is one of the most common problems regarding the distribution of reinforcing particles in an aluminum matrix during the process of melting and casting (Sajjadi et al., 2011). This is due to the density difference between the matrix and the particles. Good dispersion is influenced by the casting speed, casting temperature, and inlet system of the casting products. Rapid solidification provides better particle distribution due to the formation of fine dendrites giving little time for the particles to settle.

The purpose of this study is to characterize the microstructure and mechanical properties of composite aluminum A356 as a matrix, 10% volume fraction of micro Al<sub>2</sub>O<sub>3</sub> as a reinforcer, and 8 wt% magnesium as a wetting agent with the addition of variations of TiB as a grain refiner with 0, 0.01, 0.0347, 0.0362, 0.0622, and 0.0689 wt% through the stir casting process.

## 2. EXPERIMENTAL METHOD

The chemical composition of the aluminum alloy A356 is used as a matrix, micro Al<sub>2</sub>O<sub>3</sub> with size variations of < 63  $\mu\text{m}$  (28%) and > 63  $\mu\text{m}$  (72%) is used as a reinforced particle at 10% volume fraction, 8 wt% pure magnesium is used as a wetting agent, and TiB with variations of 0 wt% (aluminum A356), 0.01 wt% (composite #1), 0.0347 wt% (composite #2), 0.0362 wt% (composite #3), 0.0622 wt% (composite #4), and 0.0689 wt% (composite #5) is used as a grain refiner. The steps involved in preparation of the casting process include the preparation of tools and materials, cutting ingots, balancing the appropriate material formulation, coating the mold with a mixture of thinner and zirconia powder, and heating the mold in a muffle furnace for four minutes at a temperature of 1000°C. The melting process is started by gradually placing the A356 aluminum ingots into a tilting furnace at a temperature of 800°C. After the aluminum has

fully melted, the alloying process is carried out by incorporating Mg and Al-5Ti-1B followed by a stirring and degassing process with argon gas for two minutes to remove any oxygen from the molten aluminum. This is followed by the disposal of any slag that has formed on the surface of the molten aluminum. Al<sub>2</sub>O<sub>3</sub> particles that have been heated for one hour in a muffle furnace are then put into the molten aluminum alloy and stirred for two minutes at 300–500 rpm followed by disposal of the slag, then poured into the mold. The mold is allowed to cool naturally. In the final stage, the casting product is removed from the mold and cut to fit the testing process.

The mechanical properties of the composites were measured using tensile strength testing, hardness testing, and wear resistance testing. The entirety of the mechanical testing was conducted at the Center of Materials Processing and Failure Analysis Universitas Indonesia (CMPFA UI). Tensile testing was carried out using GoTech 27-7000 LA 10 based on ASTM E8 standards with three specimens of each variable. Hardness testing refers to the ASTM E18-11 standards using Brinell hardness. For each sample, five hardness tests were carried out on randomly selected regions in order to eliminate the segregation effects and obtain a representative value of the material hardness. Wear resistance testing was carried out based on the Ogoshi method with the following parameters: sliding distance (x) = 600 m, thickness of ring (B) = 3 mm, load = 6.32 kg, sliding speed = 2.91 m/s, and radius of ring (r) = 1.47 cm.

Metallographic preparation was based on ASTM E3-11 standards. Samples were ground using SiC paper #80, #120, #240, #400, #600, #800, #1000, #1200, and #1500, prior to polishing using a TiO<sub>2</sub> solution. Furthermore, a sample was etched using a Keller reagent (95 ml of water, 2.5 ml of HNO<sub>3</sub>, 1.5 ml of HCl, and 1.0 ml of HF) for 15 seconds. The whole sample preparation was observed using an Olympus AH Optical Microscope (OM) at the Department of Metallurgy and Materials Engineering Universitas Indonesia and a Scanning Electron Microscope – Energy Dispersive X-Ray Spectroscopy (SEM-EDX) at the Center for Metrology and Microanalysis Surya University. Composition and phase identification were examined using Optical Electron Spectroscopy (OES) and X-Ray Diffraction (XRD) at CMPFA UI.

### 3. RESULTS AND DISCUSSION

#### 3.1. Chemical Composition of A356/Al<sub>2</sub>O<sub>3</sub> Composite

There was a difference between the target composition and actual composition of casting due to the increasing amount of impurity element (Fe) in the composite material. Impurities can emanate from casting tools that are soluble in liquid aluminum, such as stir bars and degasser rods.

Table 1 Chemical composition of A356/Al<sub>2</sub>O<sub>3</sub> composite

Samples	Elements (wt%)								
	Al	Si	Mg	Fe	Mn	Cr	Ti	Ni	Cu
Aluminum A356	Bal.	6.82	0.32	0.185	0.0047	0.0045	0.119	0.009	0.0065
Composite #1	Bal.	6.81	8.66	0.469	0.0263	0.1020	0.126	0.171	0.0108
Composite #2	Bal.	7.05	8.27	0.459	0.0261	0.0935	0.151	0.227	0.0009
Composite #3	Bal.	6.76	7.95	0.520	0.0192	0.0993	0.154	0.156	0.0031
Composite #4	Bal.	7.04	7.03	0.575	0.0329	0.1640	0.168	0.318	0.0162
Composite #5	Bal.	6.78	7.32	0.800	0.0296	0.1780	0.178	0.259	0.0074

The existence of a high Fe content can form intermetallic compounds with poor mechanical properties. Intermetallic compounds include  $\alpha$ -Al<sub>8</sub>Fe<sub>2</sub>Si (chinese script),  $\alpha$ -Al<sub>15</sub>(Fe,Mn)<sub>3</sub>Si<sub>2</sub> (chinese script, polyhedral, atau star-like crystals),  $\beta$ -Al<sub>5</sub>FeSi (needle-like), Al<sub>3</sub>Fe,  $\pi$ -

Al<sub>8</sub>Mg<sub>3</sub>FeSi<sub>6</sub>, and  $\beta$ -Fe (platelet-like) (Fortini et al., 2016).  $\beta$ -Fe and  $\beta$ -Al<sub>5</sub>FeSi compounds have needle- or platelet-like forms that can act as a stress raiser, increase porosity, increase fracture initiation, lower ductility, and render the material brittle. The addition of Mn is able to change the morphology of Fe compounds in a platelet-like form into (FeMn)Al<sub>6</sub> and (FeMn)<sub>3</sub>Si<sub>2</sub>Al<sub>15</sub> that are more cubic or globular shaped, thereby increasing the strength, elongation, and ductility of the material (Shabestari, 2004).

### 3.2. Microstructural Observation of A356/Al<sub>2</sub>O<sub>3</sub> Composite

The microstructures of composites with different additions of grain refiner TiB are shown in Figure 1 using a 500 $\times$  magnification, with (a) 0 wt%, (b) 0.01 wt%, (c) 0.0347 wt%, (d) 0.0362 wt%, (e) 0.0622 wt%, and (f) 0.0689 wt%.

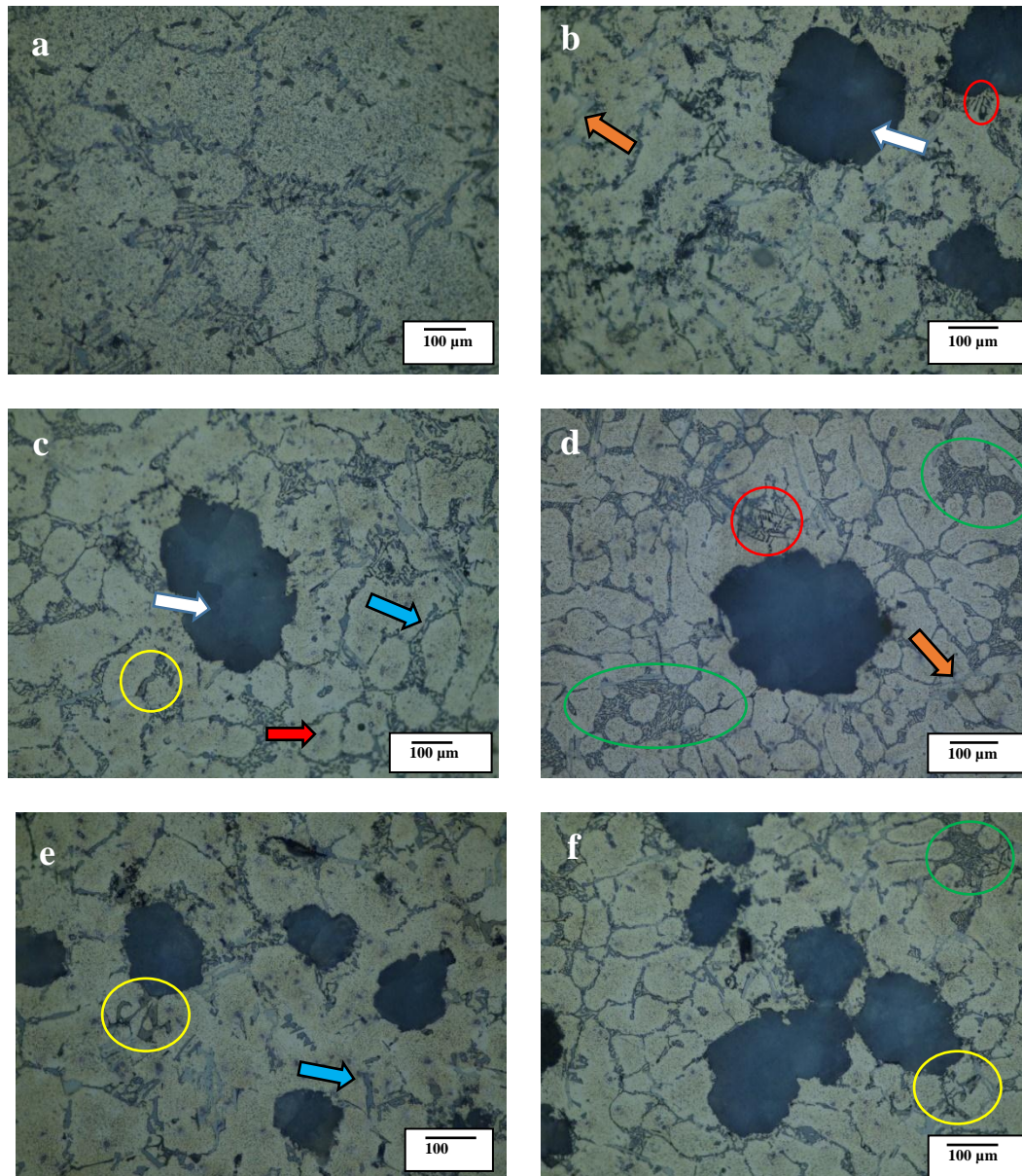


Figure 1 Microstructure of A356/Al<sub>2</sub>O<sub>3</sub> composite

Figure 1a is an aluminum A356 casting result without reinforcement Al<sub>2</sub>O<sub>3</sub> particles and grain refiner Al-5Ti-1B. In this figure, there is an  $\alpha$ -Al (colored light gray) phase and a silicon eutectic phase (colored dark gray). In Figure 1f, Al<sub>2</sub>O<sub>3</sub> particles formed aggregates and clusters. The blue arrows show the eutectic silicon (Si) with irregular and plate shapes. The yellow, red,

and green circles show the primary  $Mg_2Si$  compound, Al- $Mg_2Si$  binary, and Al- $Mg_2Si$ -Si ternary forms according to research conducted by Trostein Grostad (Grostad et al., 2014). The orange arrows indicate the formation of  $\beta-Al_5FeSi$  compounds that can degrade the mechanical properties of the composite. The red arrow indicates a compound surrounded  $Al_3Ti$  Aluminum phase (Qin et al., 2004).  $Al_3Ti$  compound was formed in two different morphologies, equiaxed/blocky and plate or needle/flaky (Wang et al., 2004). Flaky  $Al_3Ti$  formed by growth along the direction of the dendrite arms  $\langle 1\ 1\ 0 \rangle$  at a high temperature and moderate cooling rate, while blocky  $Al_3Ti$  formed in either direction  $\langle 1\ 1\ 0 \rangle$  and  $\langle 0\ 0\ 1 \rangle$  under conditions of supersaturated titanium in the molten Al-Ti-B.

The addition of reinforced  $Al_2O_3$  particles and grain refiner Al-5Ti-1B has a significant impact on grain size, with the grains becoming smaller and smoother. Al-5Ti-1B serves as a nucleating agent to form compounds  $TiB_2$  and  $Al_3Ti$ . The composite grain sizes are shown in Table 2. Besides reducing the grain size, the role of grain refiner Al-5Ti-1B is to distribute second phase, microporosity, and the reinforcement particles (Emamy et al., 2008).

Table 2 Grain size of A356/ $Al_2O_3$  composite

Samples	A356	#1	#2	#3	#4	#5
Grain Size ( $\mu m$ )	153.11	83.92	78.61	80.38	96.80	90.23

SEM-EDX testing was conducted to determine the phase and compound shape in the composite A356/ $Al_2O_3$ . The SEM-EDX observation is shown in Figure 2.

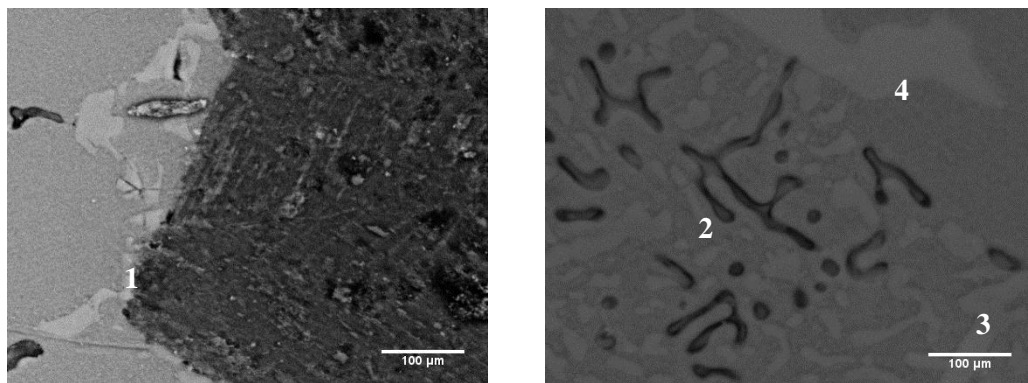


Figure 2 SEM-EDX images of A356/ $Al_2O_3$  composite with 0.0347 wt% TiB

Point 1 in Figure 2 that originally predicted the  $MgAl_2O_4$  spinel layer interface between the aluminum matrix with  $Al_2O_3$  particles is not proven by EDX testing. The dominant elements at point 1 are Al (57.4%) and Si (18.7%), indicated as A356 aluminum matrix containing aluminum and Si eutectic.

Table 3 EDX result of A356/ $Al_2O_3$  composite with 0.0347 wt% TiB

Point	Element Content (At%)						
	Al	Si	Mg	O	C	Ti	Fe
1	57.4	18.7	-	14.3	9.2	0.4	-
2	75.3	7.7	-	7.7	5.0	-	-
3	69.9	13.2	5.0	4.5	7.1	-	0.4
4	65.8	12.4	6.1	4.7	9.1	-	1.9

The MgAl<sub>2</sub>O<sub>4</sub> spinel layer is a very thin layer of about 5 μm thickness, which makes it quite difficult to observe. This layer modifies and damages the aluminum oxide layer on the surface of the liquid, thereby facilitating diffusion, wetting, and sintering, in addition to reducing surface tension and providing a strong chemical bond on the surface of the aluminum and Al<sub>2</sub>O<sub>3</sub> particles (Lurnley et al., 1999). The element Ti enables the formation of TiAl<sub>3</sub> compounds that act as nucleation sites of the α-Al primer. The dominant elements at point 2 are Al (75.3%) and Si (7.7%), indicated as A356 aluminum matrix containing aluminum and Si eutectic. The high content of iron at point 2 may allow the formation of β-Al<sub>5</sub>FeSi compound (needle-like) and π-Al<sub>8</sub>Mg<sub>3</sub>FeSi<sub>6</sub> that can degrade the mechanical properties because of its role as a stress raiser. Based on the solidification reaction, the possibilities for points 3 and 4 are Al + Mg<sub>2</sub>Si ternary + Si phase.

### 3.3. Mechanical Properties of A356/Al<sub>2</sub>O<sub>3</sub> Composite

The tensile strength of the composite will increase with the addition of Al<sub>2</sub>O<sub>3</sub> particles. This is because the Al<sub>2</sub>O<sub>3</sub> particles serve to hold or inhibit dislocation so that the material has increased mechanical properties (Kheder et al., 2011; Singh et al., 2012). Kheder et al. (2011) also showed that Al<sub>2</sub>O<sub>3</sub> particles serve as a grain refiner in which the particles act as nucleation sites for the growth of grains during solidification.

An improvement in tensile strength can be due to the addition of grain refiner Al-5Ti-1B that acts as a nucleating agent to form a compound TiB<sub>2</sub>. Stoichiometrically formed of TiB<sub>2</sub> compounds occurred from the composition of 0.05 wt% Ti and 0.02 wt% B. The excess of Ti also functions as a nucleating agent to form TiAl<sub>3</sub> and establish the following Equation 1:



Casting quality can be improved with the addition of grain refiner Al-5Ti-1B. Figure 1 and Table 2 show the changes in the size and shape of the α-Al primary grain from 153 μm to 78–96 μm and from being columnar in shape to becoming more rounded (equiaxed). This shape offers several advantages such as its high yield strength, high toughness, improved machineability, and the production of a good, deep drawability. The more the grain boundaries were formed, the more dislocations were hampered, thereby improving the mechanical properties of the material.

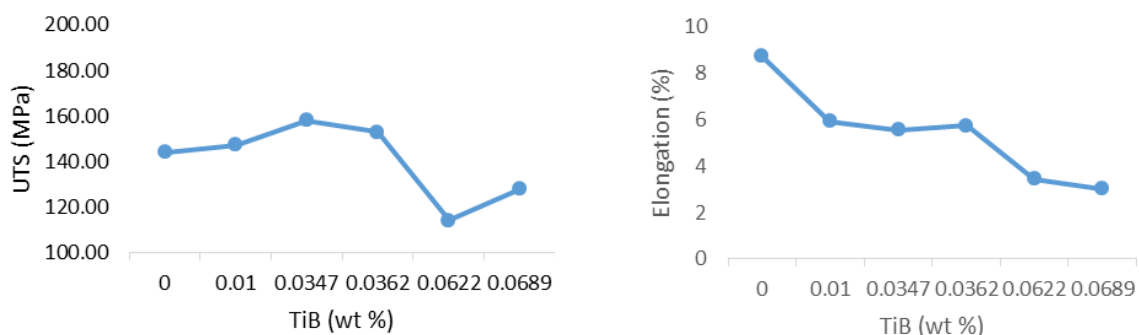


Figure 3 Effect of grain refiner TiB on: (a) ultimate tensile strength; and (b) elongation of A356/Al<sub>2</sub>O<sub>3</sub> composite

The addition of grain refiner TiB can increase the ultimate tensile strength (UTS) of composite A356/Al<sub>2</sub>O<sub>3</sub>. The highest UTS is 158.24 MPa with the addition of 0.0347 wt% TiB. However, based on Figure 3, the addition of 0.0622 wt% and 0.0689 wt% TiB exhibits lower tensile strength than with no addition of grain refiner. This could be due to high levels of grain refiner triggering a poisoning effect. The addition of 0.03 wt% TiB to Al-7Si alloy does not have a

significant impact and tends to give adverse effects (poisoning effect) by binding Si to form titanium silicide that coats the surface of  $\text{TiAl}_3$ , thus the form of nucleation is blocked (Kori et al., 2000). Based on microstructural observations in Figure 1f, there is significant agglomeration of  $\text{Al}_2\text{O}_3$  particles compared to other compositions. This agglomeration tends to have hydrogen porosity that can trigger fracture initiation.

The ductility of composite material from Figure 3b deteriorates significantly with the addition of  $\text{Al}_2\text{O}_3$  particles and grain refiner TiB. Kheder et al. (2011) showed that the elongation and ductility of composite material will decrease with the addition of  $\text{Al}_2\text{O}_3$  particles.  $\text{Al}_2\text{O}_3$  particles will resist any dislocation movement. Porosity and intermetallic compounds in a needle- or platelet-like form are other factors that can also affect the elongation of composite material.

Based on Figure 4, the hardness of composite material increases significantly with the addition of  $\text{Al}_2\text{O}_3$  particles and grain refiner TiB. This is due to the  $\text{Al}_2\text{O}_3$  particles having greater strength and hardness than aluminum A356, thereby increasing the overall hardness properties. The role of grain refiner Al-5Ti-1B is as a nucleation point and to change the shape of  $\alpha$ -Al primary grains from elongated and coarse into equiaxed and fine. These microstructural changes increase the strength and hardness properties of the composite. The greatest hardness of 66.43 HRE was obtained with the addition of 0.0362 wt% TiB.

The microstructures in Figure 2 show the  $\text{Mg}_2\text{Si}$  compound in its primary, binary, and ternary forms.  $\text{Mg}_2\text{Si}$  compounds have excellent mechanical properties, a high melting temperature ( $1085^\circ\text{C}$ ), low density ( $1.99 \text{ g/cm}^3$ ), low coefficient of thermal expansion ( $7.5 \times 10^{-6} \text{ K}^{-1}$ ), high hardness, and high elastic modulus (120 GPa).

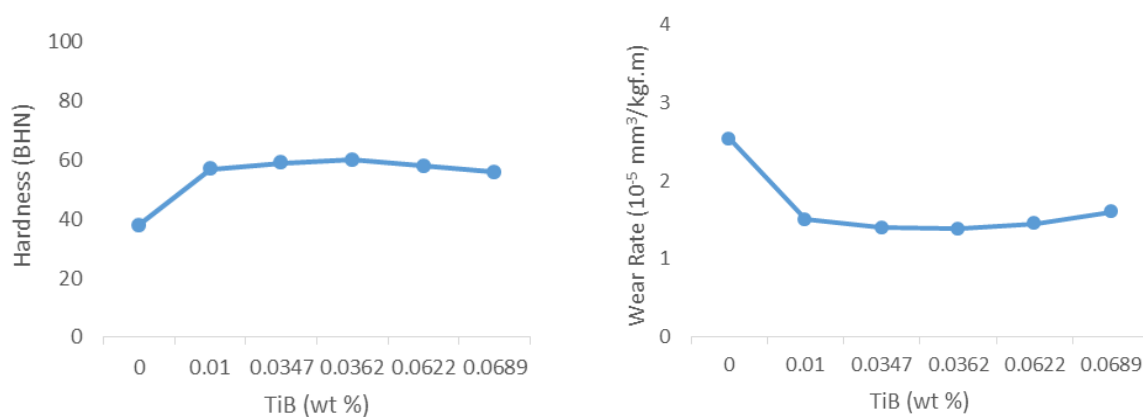


Figure 4 Effect of grain refiner TiB on: (a) hardness; and (b) wear rate of A356/ $\text{Al}_2\text{O}_3$  composite

The addition of 1 wt%, 2.5 wt%, and 5 wt%  $\text{Al}_2\text{O}_3$  to aluminum A356 can increase the hardness from 62 BHN to 68 BHN and decrease the wear rate of the composite (Alhawari et al., 2013). Figure 4b shows the wear rate of A356/ $\text{Al}_2\text{O}_3$  composite with different variations of grain refiner TiB. The addition of  $\text{Al}_2\text{O}_3$  particles and grain refiner TiB improves wear resistance compared to pure aluminum A356. Generally, the wear rate is inversely proportional to hardness. The harder the composite material, the lower the wear rate. The optimum wear rate is  $1.384 \times 10^{-5} \text{ mm}^3 \cdot \text{kgf/m}$  with the addition of grain refiner at 0.0362 wt% TiB.

### 3.4. X-Ray Diffraction of A356/ $\text{Al}_2\text{O}_3$ Composite

The XRD sample used just the highest tensile strength at 158.243 MPa with the addition of 0.0347 wt% TiB. XRD test analysis was performed using X'Pert HighScore Plus software.

From Figure 5, it can be seen that there are seven peaks: 28.2291, 38.2769, 44.5091, 47.2274, 64.8904, 78.1114, and 82.3352.

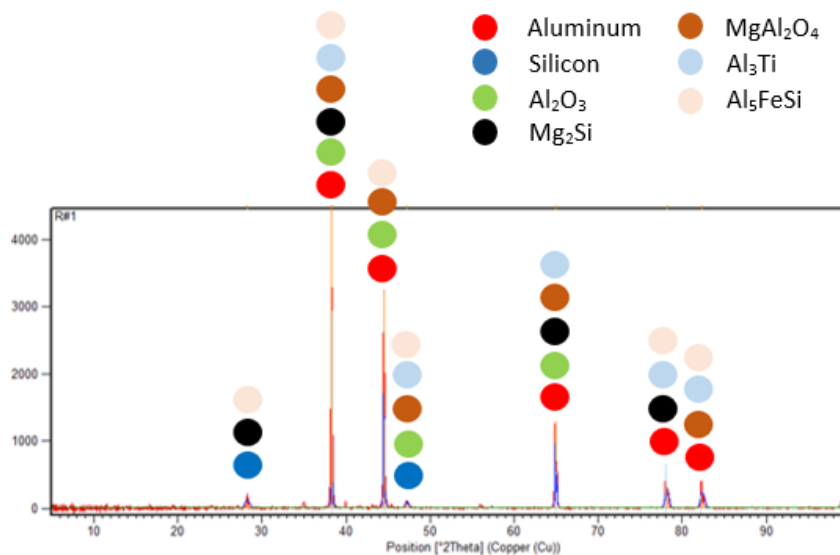


Figure 5 XRD pattern of A356/Al<sub>2</sub>O<sub>3</sub> composite with 0.0347 wt% TiB

Mg<sub>2</sub>Si compound improved the mechanical properties of the A356/Al<sub>2</sub>O<sub>3</sub> composite because it has a high melting point, low density, high hardness, low thermal expansion, and a high Young's modulus. Al<sub>3</sub>Ti compound is stable at high temperatures and acts as a nucleating agent. Spinel MgAl<sub>2</sub>O<sub>4</sub> compound is formed at the interface of the Al<sub>2</sub>O<sub>3</sub> particles and aluminum A356 matrix. Spinel MgAl<sub>2</sub>O<sub>4</sub> can damage the Al<sub>2</sub>O<sub>3</sub> passivation layers and provides good contact between the aluminum matrix and Al<sub>2</sub>O<sub>3</sub> particles. Intermetallic Al<sub>5</sub>FeSi compound is formed due to the high content of Fe in the composite material of 0.575 wt%. Al<sub>5</sub>FeSi compounds may be shown as needle- or platelet-like β-Al<sub>5</sub>FeSi compound that can serve as a stress raiser, increase porosity, increase fracture initiation, lower ductility, and render the material brittle.

#### 4. CONCLUSION

The addition of Al<sub>2</sub>O<sub>3</sub> particles and grain refiner TiB reduces grain size from 153.11 μm to 78.61 μm and changes the shape of elongated and coarse grains into equiaxed and fine grains that increase tensile strength, hardness, and wear resistance but also decrease elongation and ductility. The process is influenced by the mechanism of grain boundary strengthening, chemical composition, the size and shape of the grain, Al<sub>2</sub>O<sub>3</sub> particle distribution, and porosity. The optimum addition of grain refiner TiB was obtained at 0.0347 wt% and 0.0362 wt%. Microstructure observations and XRD testing indicate the formation of aluminum, silicon eutectic, primary Mg<sub>2</sub>Si, binary Mg<sub>2</sub>Si, ternary Mg<sub>2</sub>Si, Al<sub>3</sub>Ti, and β-Al<sub>5</sub>FeSi phase.

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