Characterization of aluminium AC4B/Nano TiC composite with the variation of volume fraction of nano TiC reinforced through stir casting process

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Abstract. Characterization of aluminium AC4B/Nano TiC composite with variation of volume fraction of nano TiC reinforced through stir casting process has been studied. The aluminium matrix used was aluminium alloy AC4B, which contain silicon and copper as its main alloy. Furthermore, the addition of Nano TiC into AC4B composite can increase the tensile strength, ductility, and toughness of AC4B composite by refining the dendrite structure of the α-Al phase and forming super saturated solid phase, θ (Al2Cu). In this study, AC4B/Nano TiC composite were made through stir casting with some variable parameters of Nano TiC reinforce composition of 0.25%, 0.3%, 0.35%, 0.4%, and 0.5% volume fraction to determine the optimum value of the mechanical properties of AC4B/Nano TiC composite. Stir casting process was chosen because it has several advantages, such as easy to use, flexible, and can be used to produce a large number of the products. It is known that AC4B/Nano TiC composite has optimum value of the mechanical properties when Nano TiC composition is 0.3% volume fraction with ultimate tensile strength of 132.31 MPa and the hardness of 55.18 HRB.

1. Introduction
Current technological developments are very fast because of seeing the need for new technologies that can meet human daily life needs. This rapid technological development is open the opportunities to the need for material that has mechanical properties that can compensate the sophistication of technology that is made. A material currently being developed is the composite materials.

Composite is a promising solution as a material that can compensate the sophistication of technology with its mechanical properties because composite is a material that made from two different material with different mechanical properties that can combine and improve the strength of the composite itself. Aluminium composite is one of the composite type that has bigger opportunities to be used as the material for the new technologies in the future because aluminium has the characteristics of low density, good strength, good wear and corrosion resistance, low melting point, and resilience.

In this study, the composite material used was a matrix composite material of aluminium AC4B with a reinforce in the form of nano TiC. The matrix composites of aluminium AC4B will be made through the stir casting process with variable parameter of nano TiC reinforce composition of 0.25%, 0.3%, 0.35%, 0.4% and 0.5% volume fraction so that it is expected to get the optimal value from the reinforce addition of Nano TiC to obtain optimal strength of aluminium matrix composites. The addition of Al-5Ti-1B aims to minimize the formation of dendritic grains by triggering equiaxed fine grain growth [1]. The addition of Al-15Sr aims to change the morphology of silicon eutectic which was originally in the form of a rough needle into a form of fibres that are spread evenly [2]. Beside, The addition of Al-15Sr can also increase the flowability of AC4B alloys [3]. Meanwhile, the addition of magnesium (Mg)
aims to increase the elasticity of the aluminium matrix so that the distribution of particle reinforce is evenly distributed and prevents the occurrence of reinforcing particle agglomeration [4].

2. Methods
The study was begun with the fabrication process of composite samples of aluminium AC4B/Nano TiC. The AC4B alloy is melted at 850°C in the tilting furnace. The Nano TiC reinforce which has been pre-heating at 900°C in the muffle furnace for an hour, and the purification process to remove impurities using an ultrasonic vibrator are mixed into the melting AC4B alloy. Then, the melting AC4B alloy was stirred using a stirrer for 40 seconds and carried out the degassing process using argon gas for 2 minutes. Furthermore, 0.15%wt TiB, 0.04%wt Sr., and 5%wt Mg were added to the melting AC4B alloy. Then, it was stirred using a stirrer for 40 seconds and carried out the degassing process using argon gas for 2 minutes. The melting alloy was then poured into a mould which had been pre-heated at a temperature of 850°C in a muffle furnace for 7 minutes which was then cooled through an air-cooling process. Composites of aluminium AC4B/Nano TiC are made with variations of 0.25%, 0.3%, 0.35%, 0.4%, and 0.5%Vf. The composition of pure AC4B aluminium can be seen in Table 1.

Table 1. Al AC4B composition determined by optical electron spectroscopy

| Element | %  | 86.5 | 7.65 | 1.01 | 2.51 | 1.05 |
|---------|----|------|------|------|------|------|

Furthermore, destructive testing and identification of elements and compounds were carried out to determine the characteristics of the composite samples. Meanwhile, samples of aluminium AC4B/Nano TiC were tested through mechanical testing in the form of a tensile test using the Go Tech 27-7000 LA 10 machine with ASTM E8 standard, hardness test using the Rockwell B method with indenter in the form of a steel ball with a diameter of 1/16 inch according to the standard ASTM E18, and the Charpy impact test method with the ASTM E23 standard. Density testing was carried out to determine the effect of adding nano TiC on the composite density by using the Achimedes law and then the amount of porosity in the samples were calculated.

In addition, samples were characterized by metallographic testing, such as Optical Microscope (OM) to observe the shape of microstructure based on the ASTM E3-11 standard by using ETSA keller, SEM and EDS to identify phase and surface structure of AC4B/Nano TiC composites, OES to determine chemical composition, and XRD to determine the compounds formed.

3. Results and Discussion
3.1 Chemical Composition of AC4B/Nano TiC Composite

Table 2. OES Test Results of AC4B/Nano TiC Composite.

| Chemical Elements | No reinforce | 0.25% Nano TiC | 0.3% Nano TiC | 0.35% Nano TiC | 0.4% Nano TiC | 0.5% Nano TiC |
|------------------|--------------|----------------|---------------|---------------|---------------|---------------|
| Al                | 86.5         | 81.367         | 81.33         | 81.43         | 83.667        | 78.43         |
| Si                | 7.65         | 6.76           | 7.397         | 5.967         | 7             | 7.227         |
| Fe                | 1.01         | 5.373          | 4.53          | 4.353         | 3.513         | 4.65          |
| Cu                | 2.51         | 2.057          | 1.95          | 2.02          | 1.97          | 1.99          |
| Mn                | 0.233        | 0.214          | 0.277         | 0.195         | 0.178         | 0.183         |
| Mg                | 1.05         | >2.40          | >2.40         | 4.89          | >2.40         | 6.243         |
| Ti                | 0.036        | 0.06           | 0.109         | 0.081         | 0.092         | 0.09          |
| Sr                | <0.0001      | 0.011          | 0.018         | 0.008         | 0.011         | 0.009         |

Based on the results of the OES test, there are discrepancies in some percentage of the chemical elements possessed by composite of aluminium AC4B/Nano TiC, such as the content of magnesium (Mg) and iron (Fe) which experience the increase of amount percentage. Magnesium (Mg) has increased due to
the addition of magnesium levels which aims to increase the elasticity of aluminium to be able to wet the reinforce of Nano TiC. The Increase of the amount of magnesium content can affect the formation of several phases which can affect the mechanical properties of aluminium AC4B/Nano TiC composites, such as the formation of Mg2Si phase owing to the change of main solidification reaction from Al-Si-Cu to Al-Si-Mg.

This change of main solidification reaction can occur due to the change of main chemical elements composition from the composition of copper (Cu) which initially has a content ranging from 2-4% to magnesium which has a much larger content, which ranges from 4-7% after the manufacturing process. Meanwhile, the increase in the amount of iron (Fe) is due to the presence of iron (Fe) elements which also dissolve into the melting AC4B alloys derived from stirrer made of steel during the fabrication process of aluminium AC4B/Nano TiC composite. The iron (Fe) content that is too high can reduce the ultimate tensile strength (UTS) and tenacity of the AC4B/Nano TiC composite by forming an iron intermetallic phase [5].

3.2 Microstructure of AC4B/Nano TiC composite

![Figure 1](image-url)

**Figure 1.** Observation Results of Microstructure of Aluminium AC4B/Nano TiC Composite: (a) AC4B with no reinforce; (b) 0.25% Vf; (c) 0.3% Vf; (d) 0.35% Vf; (e) 0.4% Vf; (f) 0.5% Vf.

Based on figure 1., it can be seen that there are several phases identified, namely α-Al phase, Eutectic Si phase shaped acicular, Mg2Si phase, A13Cu phase, A13Ti phase and intermetallic phase of iron in the form of β-Al5FeSi shaped sharp and flat, A13Fe phase, and π-Al5FeMg3Si5 phase.
Figure 2. Results of SEM/EDS and fractography test for aluminium AC4B/Nano TiC composite of 0.3%

Table 3. Composition of Aluminium AC4B/Nano TiC Composite Elements.

| Spot | C  | Mg  | Al  | Si  | Ti  | Fe  | Cu  | Phase           |
|------|----|-----|-----|-----|-----|-----|-----|-----------------|
| 1    | 1.15 | 31.54 | 3.33 | 18.64 | 0.02 | 0.16 | 0.12 | Mg$_2$Si Primary |
| 2    | 0.89 | 0.89 | 38.88 | 2.81 | 12.65 | 1.51 |     | A$_1$Ti         |
| 3    | 0.39 | 4.56 | 46.04 | 2.24 | 0.06 | 0.13 |     | A$_1$Cu         |
| 4    | 1.86 | 0.95 | 23.86 | 0.34 | 0.03 | 0.59 | 39.14 | A$_1$Cu         |
| 5    | 0.41 | 0.56 | 50.89 | 0.45 | 0.09 | 0.01 | 0.06 | α-Al            |
| 6    | 1.2  | 0.59 | 49.8  | 0.53 | 0.02 | 0.18 | 0.69 | β-Al$_2$FeSi    |
| 7    | 1.85 | 21.36 | 16.99 | 14.06 | 0.06 |     | 0.38 | Mg$_2$Si Primary |

Figure 3. Results of fractography test for aluminium AC4B/Nano TiC composite of 0.3%

Besides, to ensure the phase correctness formed based on observations of microstructures as well as conformity between the phases formed and the elements contained within, several tests are carried out, such as SEM/EDS and XRD test which can be seen in figure 2. and figure 4.
Figure 4. Results of XRD test of aluminium AC4B/Nano TiC composite of 0.3%

Based on the figure 4, it can be seen that there are several compounds that have been identified through XRD testing, including aluminium, Mg$_2$Si, Al$_2$Cu, Al$_3$Fe, Al$_3$Ti, $\beta$-Al$_5$FeSi, and $\pi$-Al$_9$FeMg$_3$Si$_5$. The identified Mg$_2$Si compound is a compound formed from the main solidification of Al-Si-Mg. Meanwhile, the Al$_3$Ti phase can be formed due to the addition of nano TiC amplifiers and TiB grain refining agents, where the Al$_3$Ti phase formed can be a nucleating agent for $\alpha$-Al so that the $\alpha$-Al phase which initially has a dendrite form will change to an equaixed form. Meanwhile, the Al$_3$Fe, $\beta$-Al$_5$FeSi, and $\pi$-Al$_9$FeMg$_3$Si$_5$ phases are formed due to the high content of magnesium and iron in the AC4B / Nano TiC aluminum composite.

Mg$_2$Si phase can be formed due to the high content of magnesium in which it is equal to 5.23%wt. This increase occurs due to the addition of magnesium levels of 5%wt. in which it aims to increase the elasticity of aluminium to be able to wet the reinforce particle. The increase of magnesium content which reaches 5.23% wt. cause a magnesium content being greater than the copper content in which it is only 1.997%wt. Thus, it causes a change of main solidification reaction from Al-Si-Cu to Al-Si-Mg. Therefore, Mg$_2$Si phase will be more dominant than Al$_2$Cu phase.

However, based on microstructural observations, it can be seen that in the AC4B/Nano TiC composite microstructure included an Al$_2$Cu phase although the main solidification reaction experiences a change from the Al-Si-Cu solidification reaction to the Al-Si-Mg solidification reaction. This Al$_2$Cu phase can be formed when done by a rapid cooling process was done in which it caused the solidification line would touch the eutectic line so that a decrease in temperature during solidification will allow the formation of Al$_2$Cu phase even though it is only in small amounts.

Iron intermetallic phase in the form of $\beta$-Al$_5$FeSi phase, Al$_3$Fe phase, and $\pi$-Al$_9$FeMg$_3$Si$_5$ phase can be formed because it was influenced by two factors, namely the high content of iron and magnesium which is equal to 4.48%wt. and 5.23%wt. with a low solidification rate. $\beta$-Al$_5$FeSi phase can be formed by reaction of L+Al$_3$Fe $\rightarrow$ Al+Al$_5$Fe$_2$Si+L $\rightarrow$ Al+Al$_3$FeSi. Based on these reactions, it can be seen that the reaction between Liquid and the intermetallic phase of Al$_3$Fe with a high solidification rate will form the Al$_3$Fe$_2$Si phase. However, when the solidification rate has not stopped, the Al$_3$Fe$_2$Si phase will react with Liquid to form a $\beta$-Al$_5$FeSi phase [6].
The formation of $\beta$-Al$_5$FeSi phase is influenced by the content of the elements iron and magnesium, where the iron content is above 2.4% wt. and magnesium content below 6% wt. with a low solidification rate can form $\beta$-Al$_5$FeSi phase which has a needle-like shape so that this phase has a relatively high strength.

![Figure 5.](image)

**Figure 5.** Comparison of Al$_3$Fe microstructure forms: (a) Needle-shaped Al$_3$Fe forms; (b) Irregular-shaped Al$_3$Fe forms.

In addition, the intermetallic phase of Al$_3$Fe generally has a needle-shaped microstructure as shown in figure 5 (a). However, in this study the microstructure shape of the Al$_3$Fe intermetallic phase is irregular-shaped as shown in Figure 5 (b). The microstructural transformation of the Al$_3$Fe intermetallic phase is due to the high magnesium content in aluminium AC4B/Nano TiC composite, where the magnesium content is above 1.5% wt. able to change the microstructure of Al$_3$Fe from needle-shaped to irregular-shaped.

Meanwhile, Al$_3$Ti phase can be formed due to the addition of TiB grain refiner agent. The Al$_3$Ti phase formed will act as a nucleation agent for $\alpha$-Al, so $\alpha$-Al phase which initially has the form of dendrites will turn into an equiaxed form that can increase the mechanical strength of aluminium AC4B/Nano TiC composite.

### 3.3 Results of mechanical test of AC4B/Nano TiC composite

![Graph](image)
Based on the results of the mechanical test, it can be seen that the addition of the Nano TiC reinforce can increase the mechanical strength of aluminium AC4B/Nano TiC composite by inhibiting dislocation movement by distributing the stress evenly in the composite matrix through three mechanisms, namely orowan strengthening mechanism, grain boundary strengthening mechanism through refining grain of α-Al phase and the precipitation hardening mechanism through the formation of saturated solids θ (Al2Cu)[7]. This is shown in the results of the tensile test in figure 6 (a) and the hardness test results in figure 6 (c), in which the increase of number of nano TiC reinforce reaches 0.3%Vf. was able to produce the ultimate tensile strength (UTS) and the highest hardness value, which is 132.31 MPa and 55.18 HRB. The grain boundary strengthening mechanism occurred when the nano TiC reinforce acted as a nucleating agent that will change the structure of the dendrite of α-Al phase being more subtle (Equiaxed). Dendrite structure of α-Al phase which is refine can increase the resistance to crack propagation of aluminium AC4B/Nano TiC composite. Meanwhile, the precipitation hardening mechanism occurred when the nano TiC reinforce increases the formation through saturated solids θ (Al2Cu) phase, in which saturated solids (Al2Cu phase) formed will result in lattice and internal stress distortion which can inhibit dislocation movement [8].

In figure 6 (c), the hardness value of aluminium AC4B/Nano TiC composite from each variation of the volume fraction of nano TiC reinforce is higher than the hardness of AC4B without the reinforce. A higher hardness value is obtained because of the addition of reinforce particles, where the addition of reinforcing particles evenly into the aluminium matrix will distribute the stress evenly so that the dislocation movement is not focused in just one area.

In this study, an increase in the hardness value was obtained due to the formation of the θ (Al2Cu) phase and the Mg2Si phase. The addition of nano TiC reinforce is able to initiate the formation of phase θ (Al2Cu) which is a saturated solid (Supersaturated Solid Solution) formed through the Precipitation Hardening mechanism. The saturated solid (Al2Cu Phase) formed in lattice distortion so it can increase the hardness value of aluminium AC4B/Nano TiC composite. In addition, the formation of the Mg2Si
phase can also increase the hardness value of the aluminium composite AC4B / Nano TiC, where the Mg7Si phase is formed due to the addition of magnesium and a modifying agent in the form of strontium. Strontium will react with aluminium to Al2Sr, then Al2Sr will act as a nucleating agent for primary Mg2Si so that the Mg2Si phase formation will increase the hardness value of aluminium AC4B / Nano TiC composite in line with the addition of strontium. In other words, as the number of Mg2Si phases is formed, the hardness value of the AC4B / Nano TiC aluminium composite will tend to increase.

However, in the variables 0.35%, 0.4%, and 0.5%Vf, there is a decrease in mechanical strength as the percentage of the increase of Nano TiC reinforce. This was caused by the uneven dispersion of nano TiC reinforce particles and the greater number of porosities in line with the increase of percentage of the volume fraction of nano TiC reinforce which can be seen in figure 6 (f).

In addition, in figure 6 (b), it can be seen that the greater the percentage of the volume fraction of the nano TiC reinforce can reduce the percentage of elongation of aluminium AC4B / Nano TiC composites, which is due to the increased agitation of aluminium AC4B / Nano TiC composites. The increase in aluminium AC4B composite agitation was caused by the formation of the Mg2Si phase, the intermetallic phase of β-Al3FeSi and intermetallic phase of π-Al3FeMg2Si3, where these phases have brittle properties so these phases are able to reduce percentage of elongation of aluminium AC4B / Nano TiC composites [9].

In addition, the increase in the elasticity of aluminium AC4B / Nano TiC composite caused the aluminium AC4B / Nano TiC composite to be more difficult to absorb energy from impact loads, where it can be seen in figure 6 (d) which shows the increasing number of nano TiC reinforce, the impact prices from aluminium AC4B / Nano TiC composites tending to decrease.

In figure 6 (e), it can be seen that the actual density values tend to be lower than the theoretical density in each variation of the nano TiC reinforce. This is influenced by the presence of Mg2Si and porosity found in aluminium AC4B / Nano TiC composites, where the Mg2Si can be formed in aluminium AC4B / Nano TiC composites due to the addition of magnesium during the fabrication process of aluminium AC4B / Nano TiC composites by 5%wt. which is used to increase the wettability of aluminium to be able to form a good interface with reinforce nano TiC. Therefore, more Mg2Si phases are formed which can affect the density of aluminium AC4B / Nano TiC composites.

For the presence of porosity in aluminium AC4B / Nano TiC composites, the percentage of the increase of porosity in aluminium AC4B / Nano TiC composites, the density value of aluminium AC4B / Nano TiC composites will tend to be more decrease. This is because porosity is gas/air trapped inside a solid and the material contained in the shaft has a lower density than material that is free from the shaft so that the material tends to be lighter than the material that is free from the shaft [10].

Based on the porosity test results in figure 6 (f), it can be seen that the decrease in the mechanical strength of aluminium AC4B / Nano TiC composite is due to the presence of increasingly large porosity [11]. In this study, porosity can be formed due to the non-optimal stirring process, the formation of shrinkage during the solidification process, and hydrogen evolution [12]. The evolution of hydrogen gas can occur due to the high pouring temperature, where the safe temperature limit that can be used to prevent the formation of hydrogen gas should be 675-700°C. However, in this study the pouring temperature was around 800-850°C. This is needed to prevent premature cooling during the stirring process of the reinforcement and pouring process into the mold. The effect of the presence of hydrogen gas in the melted AC4B alloy itself is able to reduce the tensile strength of the aluminium AC4B / Nano TiC composite because the material tends to be brittle [12].

4. Conclusion
1) Aluminium AC4B / Nano TiC composite with variations of the addition of volume fraction of Nano TiC reinforce has better mechanical properties than AC4B alloy without nano TiC reinforce particle. In addition, the addition of a nano TiC reinforce can increase the mechanical strength of aluminium composites through three mechanisms, namely the orowan strengthening mechanism, the Grain Boundary Strengthening mechanism, and the Precipitation Hardening mechanism.
2) Variable aluminium AC4B/Nano TiC composite of 0.3%Vf is an optimal variable because it has the highest mechanical strength among other variables, such as Ultimate Tensile Strength (UTS) amounted to 132.31 MPa, hardness of 55.18 HRB, and impact strength of 0.05 J/mm².

3) Microstructure observation of aluminium AC4B/Nano TiC composite shows that there are several phases formed, including phase of MgSi Primary, Mg2Si Biner, Mg3Si Terner, Al2Cu, Al3Ti, Al3Fe, β-Al5FeSi, and π-Al5FeMg3Si3.

4) The addition of magnesium (Mg) reaches 5% wt. cause the magnesium content (Mg) is higher than the copper content (Cu) which is only 1.997% wt. thus causing a change in the main solidification reaction from Al-Si-Cu to Al-Si-Mg.

5) The change of the main solidification reaction from Al-Si-Cu to Al-Si-Mg causes the MgSi phase to form easily. In addition, the addition of magnesium (Mg) which reaches 5% wt. also influences the formation of the intermetallic phase β-Al5FeSi and the intermetallic phase π-Al5FeMg3Si3 which is brittle and changes the microstructure of the Al3Fe phase from needle-shaped to irregular-shape.

6) The addition of TiB grain refining agent can form the Al3Ti phase which can be a nucleating agent for α-Al so that the α-Al phase which initially has a dendritic shape will change to an equiaxed form.

7) The decrease of mechanical strength and percentage of elongation in aluminium AC4B/Nano TiC composite occurred due to a greater percentage of porosity, where the porosity can be formed due to the process of melting AC4B alloys that are too fast and the formation of shrinkage during the solidification process.

8) The decrease of mechanical strength and percentage of elongation in aluminium AC4B/Nano TiC composite occurred also due to the formation of Mg2Si phase, β-Al5FeSi, and π-Al5FeMg3Si3 which has brittle properties.

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