Characteristics of aluminium AC4B reinforced with nano-SiC composites through stir casting methods

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Abstract. Aluminium with a composition of 7-10% Silicon (Si), 2-4% Copper (Cu) is a matrix used by adding Nano SiC reinforcement particles with variations of 0.25; 0.3; 0.4% Vf with the purpose of making bullet-proof materials that have high toughness, impact resistance, and good strength. In this study, fabricated composites used a stir casting process to produce superior mechanical properties which was caused by the bond between an effective matrix and reinforcement particles. Besides, the addition of Nano SiC reinforcement particles, it was also followed by adding 5%wt Magnesium (Mg) as a wetting agent, 0.04%wt Strontium (Sr) as modifier agent, and 0.15%wt Al-5Ti-1B as a grain refining agent. Fabricated composite specimens will be carried out several tests to determine mechanical properties such as density, porosity, hardness, tensile strength, impact resistance, and ballistic test. The result of the test will be compared to the results of that without the addition of reinforcement particles. Then, using the Optical Microscope (OM), Scanning Electron Microscope (SEM), Dispersive X-Ray Spectroscopy (EDX), and X-Ray Diffraction (XRD) to analyse the dispersion of reinforcement particles in the selected alloy matrix.

1. Introduction
As one of the developing countries, Indonesia has now entered a new phase in the development of domestic infrastructure. Improving the quality of domestic infrastructure is considered as one of the foundations of a country’s progress. Almost all infrastructure in this country uses materials that have high strength, endurance and toughness to support all activities. Of the many materials used, there is currently an increasing use of composite materials to obtain superior properties, such as one of the studies that will be conducted is the use of composite materials. This composite material can be said as a substitute for iron or steel as the main source of infrastructure in this country. Steel has long been used for application because of the ease of the process and lower production costs and has a variety of mechanical properties that vary. However, steel has a high specific gravity so it is not suitable for application with light specific gravity requirements.

The material used as an alternative matrix is Aluminium which has a low density of 2.7 g/cm³ (1/3 mass of steel), corrosion resistant properties, cast ability properties, machinability properties, and a low processing temperature that facilitates the fabrication process in the Metal Matrix Composite. Because it has low strength, Aluminium is added with the elements of alloys to increase its strength in an efficient manner, for example is copper, manganese, silicone, zinc, magnesium, and etc. In addition, Aluminium has characteristics that are low density, melting point, resilience, and good ability to be moulded.
In this study, it was used a composite with an Aluminium matrix AC4B with micro Nano SiC reinforcement that have a superior properties compared to micro-sized particles because of their small size so that they have a high surface area to volume ratio. In addition of Nano SiC particles also have advantages when compared with the SiC particle, although the tendency for agglomeration is quite high. Beside that, there is addition elements of alloys such as Magnesium (Mg) to increase the wetting behaviour or the elasticity of SiC particles and matrices and to increase bonding strength, Strontium (Sr) as a modifier to change the microstructure of Aluminium to be more spherical so as to reduce stress concentration from material, TiB as a grain refiner that serves to nucletate grains so that a heterogeneous process will occur nucleation and make the solidification process run quickly. The addition of Nano SiC reinforcement will increase the mechanical strength of the composite material with the Orowan Strengthening mechanism[1]. Orowan mechanism is dislocation that also bow around a precipitate particle through. The effect of a precipitate on the movement of dislocations essentially depends on it’s strength and size. The two boundary cases in the interaction with dislocation are called by the Orowan mechanism. The formation of precipitation is dependent on temperature and time and usually occurs in discontinuous manner. While the alloy components first exhibit an irregular distribution, they accumulate at preferred positions in the lattice at elevated temperatures, leading to the formation of short-range separation and clustering.

Fabrication for the manufacture of composites is done by stir casting techniques, wherein the method is easy to do because it is economical and can produce defects in the porosity of the material and the uncomplicated process. With the stir casting method, the homogenous distribution of reinforcing particles is the final aim to be obtained in the composite final product to achieve maximum strength. Therefore, the rotation speed and length of the stirring process must be adjusted to achieve optimal conditions. This process is still widely used in aluminium casting because the process is simple, flexible, and applicative to produce in large quantities, so this method fit in with the current environment.

2. Methods
The study was carried out by a 2-step process. Those were fabrication of composite samples with stir casting methods and testing of samples from chemical composition testing to mechanical properties. In the fabrication process, it began by melting Aluminium AC4B on the melting furnace tilting with a temperature of 850° C and pre-heating Nano SiC reinforcement particles for 1 hour at a muffle furnace at 950° C, after which the reinforcing particles are inserted into melting Aluminium. Then, the melting Aluminium was stirred using a stirrer for 40 seconds and followed by a degassing process using argon gas for 2 minutes. After that, elements of alloys were added, namely, 5wt% of Mg, 0.15wt% of TiB, and 0.04wt% of Sr on melting Aluminium which was then carried out by stirring with a stirrer followed by degassing again and followed by slaggging which floated on melting Aluminium composite with a mixture of Nano SiC reinforcement particles. Then, the melting Aluminium composite was poured into a mould that has been preheated at 400°C, followed by ice-water cooling process. AC4B/Nano SiC composite made with variations of 0.25; 0.3; 0.35; 0.4; and 0.5%Vf. The composition of pure Aluminium AC4B can be seen in table 1.

| Table 1. Al AC4B composition determined by Optical Electron Spectroscopy |
|-------------------|-----|-----|-----|-----|-----|-----|
| Element | Al  | Si  | Cu  | Mg  | Fe  | Mn  | Sr  |
| %       |     |     |     |     |     |     |     |
|         | 86.5| 7.65| 2.51| 1.05| 1.01| 0.233| <0.0001 |

Destructive and identification of elements and compounds test are carried out to determine the characteristics of the composite sample made. Tensile test is carried out to determine the tensile strength and elongation with the GoTech 27-7000 LA 10 according to ASTM E8 standard. Hardness test is carried out using the Rockwell B method according to ASTM E18-11 standard using a 1/16-inch diameter steel ball indenter. The impact test is carried out using the Charpy method with the GoTech Testing Machine according to the E23-01 standard. Density testing was carried out to determine the effect of adding Nano SiC to composite density using the Archimedes law and then calculate the amount
of porosity in the sample. Microstructural observations are carried out using an optical microscope according to ASTM E3-11 standard using ETSA HF.

Identification of phase, interface, and distribution of Nano SiC particles were carried out using SEM and EDS. OES testing is done to observe the elements contained in the composite. XRD is carried out to determine the compounds that can be formed in the AC4B/Nano SiC composite.

3. Results and Discussion

3.1 Chemical composition on Al AC4B/Nano SiC composites

Table 2. shows the comparison of the chemical composition of pure AC4B alloys with that with the addition of Nano SiC reinforcement particles in each variation, which is 0.25; 0.3; 0.35; 0.4; and 0.5%Vf. AC4B is an Aluminium alloy with the main alloy 7-10% Si and 2-4% Cu. If it is seen in table 2., the results of the test, there are less levels of Si and Cu in the composite material caused by the use of scrap AC4B composites as a matrix. The Mg element increased significantly in the composite because it was added to increase the wetness for 5%, that is increase the wettability between the AC4B matrix and reinforced Nano SiC for make the better interface [6]. While for the high Fe element it was caused by the stirrer rod of SKD 61 which melted during the stirring process. This can also be explained by the fading mechanism that occurs due to the too long holding time that is set when casting and the lack of stirring so that in the presence of deposition of grain refiner particles on crucible.

| Element | AC4B (Matrix) | AC4B / SiC | AC4B / 0.25% Nano SiC | AC4B / 0.3% Nano SiC | AC4B / 0.35% Nano SiC | AC4B / 0.4% Nano SiC | AC4B / 0.5% Nano SiC |
|---------|---------------|------------|------------------------|----------------------|----------------------|----------------------|----------------------|
| Al      | 86.5          | 82,300     | 57,473                 | 82,067               | 79,233               | 82,567               | 79,233               |
| Si      | 7.65          | 7,007      | 5,617                  | 6,617                | 7,013                | 6,900                | 7,013                |
| Fe      | 1.01          | 4,403      | 3,763                  | 4,013                | 3,600                | 4,677                | 3,600                |
| Cu      | 2.51          | 2,040      | 1,471                  | 2,107                | 2,037                | 2,037                | 1973                 |
| Mn      | 0.233         | 0.218      | 0.162                  | 0.310                | 0.225                | 0.202                | 0.202                |
| Mg      | 1.05          | > 2.4      | > 2.4                  | 4.517                | > 2.4                | > 2.4                | 6.500                |
| Sr.     | <0.0001       | 0.0097     | 0.005367               | 0.011033             | 0.00104              | 0.016167             |                      |

3.2 Microstructure of Al AC4B/Nano SiC Composites

Al_{5}FeSi phase is shown in Figure 1, which has a flat and sharp shape which is hard and brittle, where the phase can be formed due to the presence of a high Fe element in the composite. This phase is clearly seen in the composition of 0.3% of Nano SiC shown in figure 1 (c). The eutectic phase is also slightly seen where the phase will increase the hardness of the composite which is the residual of the Si element which does not form a phase with other elements[7]. Mg_{2}Si phase is also seen where Mg_{2}Si binary has a Chinese script form, which looks almost in all parts of the microstructure in each variation. The π-Al_{5}FeMg_{3}Si_{5} phase also present in microstructure wherein the phase is brittle[8]. The phase formed can be seen more clearly in figure 2.

Al_{5}FeSi phase can be formed through a rapid cooling process with the reaction of L + Al_{5}Fe_{2}Si → Al+ Al_{5}FeSi in which the phase has lower mechanical properties than Al_{5}Fe_{2}Si[2]. The π-Al_{5}FeMg_{3}Si_{5} phase can be formed due to the presence of Fe and also Mg elements with a content below 6%. The phase is formed by reaction L → Al+Si+MgSi+ Al_{5}FeMg_{3}Si_{5}[3,11].

The morphology of these phase which tend to have a sharp angles like the needle-like that can provide many points of high loading concentrations, so that final product of composite will be brittle and the mechanical strength will be decrease[10].
Figure 1. Microstructure of (a) Al AC4B as cast magnification of 500x and microstructure of composite Al AC4B / Si₃N₄ at volume fraction (b) 1%, (c) 3%, (d) 5%, (e) 7%, and (f) 10%.
Figure 2. Microstructure of Al AC4B with addition of 0.3% volume fraction Nano SiC

3.3 SEM & EDS

Figure 3. SEM and EDS observation
Table 3. EDS testing result

| Points | B  | C  | O  | Mg | Al | Si | Ti | Fe | Cu | Sr. | Prediction Phases Formed |
|--------|----|----|----|----|----|----|----|----|----|----|----------------------------|
| 1      | 1.31 | 45.95 | 28.5 | 7.5 | 23.22 | 0.04 | 0.22 |    |     |    | Mg$_2$Si (Primary)        |
| 2      | 46.83 | 5.55 | 42.33 | 4.73 | 9.11 | 0.56 |    |    |    |    | $\beta$-Al$_3$FeSi       |
| 3      | 1.41 | 46.52 | 1.29 | 44.72 | 4.93 |    | 1.13 |    |    |    | Al$_2$Cu                  |
| 5      | 10.88 | 54.58 | 34.08 | 0.06 |    |    |    | 0.4 |    |    | $\alpha$-Al               |
| 6      | 1.04 | 44.96 | 27.37 | 11.26 | 14.71 | 0.29 | 0.36 |    |    |    | Al + Mg$_2$Si (Binary)    |
| 8      | 1.59 | 45.92 | 8.23 | 34.67 | 5.29 | 0.23 | 0.08 |    |    |    | $\pi$-Al$_4$FeMg$_3$Si$_5$ |
| 11     | 0.78 | 46.29 | 4.95 | 46.54 | 1.44 |    |    |    |    |    | Al + Mg$_2$Si (Binary)    |

SEM and EDS test were carried out at 7 different points. Besides, it was also observed on fracture formed on AC4B composites with the addition of Nano SiC reinforcement particles. In the results of SEM, it can be seen that the phases formed are the same as observing microstructural results. The dominant phase formed in the SEM results is Mg$_2$Si in the binary phase formed Chinese script which will increase material mechanical strength. Area number 8 shows the presence of elements Al, Fe, Mg, and Si which indicate the presence of $\pi$-Al$_4$FeMg$_3$Si$_5$ phase that has a grey colour. Al$_2$Cu phase is also detected in the EDS test results shown in area number 3, that contain the copper which will increase mechanical strength. $\beta$-Al$_3$FeSi that formed needle-like, which mean that phase have a high concentration and will decrease the mechanical strength[11].

The occurrence about fracture of this mixture as shown at figure 3, commonly found in composites, where the soft properties of the matrix and the brittle by reinforcement are combined into one material and this can increase the toughness of material. Order than that, there is porosity and shrinkage to the fracture surface, where the shaft and shrinkage occur due to poor casting process[9]. In the result of EDS test in the fractographic also found an inclusion in the form of Fe, because of the aluminium used for casting is in the form of scrap, so that there is a fairly high iron inclusion.

3.4 XRD

![Figure 4. Results of XRD test on Al AC4B/Nano SiC composites of 0.3 % volume fraction](image-url)
XRD testing on aluminium AC4B composite with Nano SiC particles was carried out on samples with composition of Nano SiC particle 0.3% Vf, where the samples has the highest impact resistance value of 0.0959 Joules/mm². To process the data generated after testing, used the software X’Pert HighScore Plus oleh PANalytical B.V..

The results of XRD test in Figure 4 show the existence M₂Si phase which is quite high and hard is also present in the composite. In addition, there is a spinel MgAl₂O₄ phase which will increase contact between the AC4B matrix and Nano SiC, so that the gas layer on the surface of reinforced Nano SiC can be minimized where this can prevent the clumping of SiC[4]. The presence of Al₃Fe and Al₅FeSi phases is due to the presence of Fe content in the composite. π-Al₉FeMg₃Si₅ phase was formed due to the presence of Fe and Mg levels below 6%. MgO phase can be formed because of the magnesium reaction with oxygen in the environment[5].

3.5 Mechanical Properties of Al AC4B/Nano SiC Composites

The highest tensile strength was obtained by adding 0.3% volume of Nano SiC fraction with a tensile strength of 94.67 MPa as seen in figure 5 (b). Composite elongation is initially increase, and then it has decreased due to the strengthening of the grain refinement phenomenon, where Nano SiC can act as a grain refiner[1]. The more Nano SiC is added, the higher the elongation, but after 0.3%, it decreased due to the presence of Nano SiC agglomeration as shown in figure 5 (a). The increase of tensile strength in the addition of 0.3% of Nano SiC was caused by the orowann strengthening mechanism owing to the gathering of the reinforcement to form a loop between the Aluminium matrix AC4B and of Nano SiC which causes an increase of composite dislocation density[14]. Fluctuating tensile strength values are caused by the porosity of hydrogen gas formed on the composite, where hydrogen gas solubility in melting Aluminium will increase at high temperatures. While the decreasing elongation can be caused by the presence of the Mg₂Si phase which is brittle and also the porosity formed.

The addition of volume fraction of Nano SiC caused the hardness of the composite increase, with the highest hardness occurring in the addition of 0.3% of Nano SiC is 52.57 HRB while hardness at 0.25% of Nano SiC volume is 51.9 HRB as shown in figure 5 (c). The increase of hardness is caused by orowann strengthening mechanisms and also the formation of Mg₅Si and Al₅Cu phases which are hard[12]. Other that that, Nano SiC can act as a grain refiner, so the higher the addition of Nano SiC, the material will increase the mechanical strength[15], but as can see in the figure 5 (c) if the addition of Nano SiC exceed the optimum point the mechanical strength will decrease because of clustering by reinforced Nano SiC. Addition of Nano SiC particles caused composite toughness decrease which is indicated by the decreasing impact price according to figure 5 (d). This shows that the energy that can be absorbed by the composite decreases. The reduced impact price can be caused by the presence of brittle Al₉FeMg₃Si₅ phase.

Composite density should increase with the addition of Nano SiC owing to Nano SiC density higher than AC4B according to theoretical density calculations. However, the composite density decreases as shown in figure 5 (e). The decrease of the density value is related to the higher amount of porosity in the addition of Nano SiC in figure 5 (f) which will affect the composite density. This high porosity affects the fluctuations in the value of hardness, tensile strength, and also the toughness of the composite, which is caused by the trapping of enough hydrogen gas[13].
Conclusion
Nano SiC particles can increase the hardness and tensile strength of Aluminium through an orowan strengthening mechanism in which all the optimal points of mechanical strength are in the variation of 0.3%Vf reinforcement particles. Composite hardness is also influenced by the formation of Mg$_2$Si dan Al$_2$Cu phases. The presence of composite mechanical properties fluctuations because of the formation of porosity and also Nano SiC agglomeration. The agglomeration is caused by too much reinforcement of the incoming Nano SiC particles and also the uneven stirring. Besides that, there are things could be develop based on this research are, optimizing the parameter processing such as the temperature melting and pouring.
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