Manufacturing of Mg-Al5TiB composite and addition of nano-sized Al2O3 through stir casting method with T6 heat treatment temperature variation

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Abstract. In order to find aluminium replacement for vehicle structure and also application of automotive components, the research on Mg-Al-Ti-B composite was performed in this work. The composite of magnesium is highly compatible to be applied in vehicle body structure because of its low weight and density. Therefore, the lightweight of vehicle body structure, great mechanical properties, and high efficiency can be obtained. Mg-Al-Ti-B serves as the matrix, and 0.20 vf% of nano-sized Al2O3 is applied as the reinforcement. The composite of Mg-Al-Ti-B was produced through a stir casting process. Hereafter, T6 heat treatment was employed with variation of aging temperature, such as 170 °C, 200 °C, 230 °C and 260 °C for 6 h, subsequent the previous 1 h, 420 °C for the treatment of solution. The T6 heat treatment impact on microstructure indicates a different morphology of primary Mg17Al12 in which spheroidization occurs, the precipitation of Al3Ti and TiB2 are also observed from the aging process. In this work, OM, EDS, XRD, OES, density and porosity measurements, as well as destructive tests were performed for the characterization. The T6 heat treated sample’s mechanical properties were rectified than the non-heat treated ones. The highest ultimate tensile strength was obtained with 65.31 MPa at 170 °C aging temperature. The optimum value of hardness, impact value and wear rate are observed at 200 °C aging temperature, the values are 92.4 HRH, 0.07 J/mm2 and 0.00254 mm3/m, respectively.

Keywords: Magnesium composite, nano-Al2O3, stir casting, T6 heat treatment, artificial aging

1. Introduction

Indonesia is one of the developing countries in the Southeast Asia Region, which is always trying to develop technology in various sectors, especially in the industrial sector. In this case, the industrial sector is believed to be one of the keys to national development. Development is one of the efforts to support the national industrial development, one of which is in the automotive sector. Technology in the automotive field is one of the fastest-growing technologies. In the automotive world, the thing that is constantly being developed is the efficiency of fuel in vehicles and reducing the pollution levels. The solution is to save costs and reduce fuel consumption by using a light vehicle mass [1].

One of the technological developments that carried out is the development of new materials that have low density but have good mechanical properties. This is done to improve the fuel efficiency, energy, cost saving and reduce the pollution levels, especially in automotive applications [2]. Material that is in accordance with these characteristics is magnesium, and it becomes one of the materials that has the
potential to be applied in the structural components in automotive panel beam instruments, components of steering, transfer cases, gears, bearing gearboxes, radiator support and connecting rods. Magnesium is a metal that has a low density [3].

In this study, the author made a composite with magnesium Al-Ti-B matrix with nano-sized alumina reinforced. In the previous study, it was explained that the volume fraction of 0.20 % is the optimum state of the nano-sized Al₂O₃ in magnesium composites. This process is done through a stir casting method. To increase the strength, especially on the surface of composite materials, the heat treatment process will also be carried out. The magnesium Al-Ti-B composite is expected to have a specific strength, stiffness, good wear resistance, and low density suitable for automotive application materials to improve their fuel and energy efficiency.

2. Materials and method

2.1. Materials
Before the experiment, the author was calculated the mass balance of composite to set up the materials. Pure magnesium 90 wt.% and Al-5Ti-B 10 wt% were applied as the matrix and nano-sized Al₂O₃ particle with 0.20 v1 % was used as the reinforcing. 335 g of pure magnesium and 15 g of Al-5Ti-B as matrix, 1.53 g of nano-sized Al₂O₃ as reinforce were prepared.

2.2. Fabrication of magnesium Al-Ti-B with nano-sized Al₂O₃ composite
Argon gas was flowed into the furnace to prevent the reaction between magnesium with oxygen. The air inside the furnace was absorbed by the vacuum furnace. Fabrication of the Mg-Al5TiB composite was carried out by stir casting. The composite of magnesium and Al-5Ti-B was heated at 675 °C. At this temperature, the composite reaches the molten state. Before the molten pouring, Al₂O₃ were also pre-treated by heating at 900 °C for 30 min. After pre-treatment, nano-sized Al₂O₃ were appended to the molten magnesium and Al-5Ti-B, followed by stirring. Before pouring the molten, the preparation of the casting die was firstly conducted by employing a mixture of thinner and zircon. Besides, the furnace was also coated by a mixture of thinner and zircon so that the furnace and the mould will have high thermal resistance to avoid cracking.

After that, as produced cast was passed pass through the T6 heat treatment procedure, start with the solution treatment for 1 h at 420 °C then subsequently performed the water quenching at room temperature [4]. Then, the 6 h artificial aging was conducted at the variation aging temperature of 170 °C, 200 °C, 230 °C, 260 °C, followed by natural aging at room temperature. This is the final step of the composite production [5].

2.3. Characterization
The preparation of all post heat-treated products was performed to undergo the destructive testing and microstructural analysis. Optical microscope was applied to identify the microstructure. The samples were ground and polished by TiO₂. Scanning electron microscope (SEM) was carried out to know the phases in microstructure. X-ray diffraction (XRD) was performed to obtain the different phases in microstructure. The observation of chemical properties was conducted by applying the optical emission spectroscopy. The investigations of mechanical properties were performed with the tensile test based on JIS Z2241; impact test based on Charpy method with ASTM E23 standard, hardness test (Rockwell H) based on ASTM E18 and wear rate test with pin on disc (Ogoshi) method based on ASTMM G99. Each sample’s density was also examined with Archimedes Theory. All mechanical tests were carried out at room temperature.
3. Results and discussion

3.1. Chemical composition

The chemical composition test by Optical Emission Spectroscopy method of the Al-Ti-B magnesium composite sample with the nano-sized Al$_2$O$_3$ reinforcement through T6 heat treatment was carried out in order to find out what elements present in the sample so that it can be predicted which phase is formed.

Chemical composition analysis is performed to compare the theoretical chemical composition of magnesium Al-Ti-B composites/nano Al$_2$O$_3$ with the actual chemical composition of magnesium Al-Ti-B composite/nano Al$_2$O$_3$. During the casting process, according to the mass balance, the composition of magnesium was added by 90 % is 315 g, while the composition of Al-Ti-B added by 10 % is 35 g. Then, the composition of nano Al$_2$O$_3$ reinforcement was added as much as 0.2 % which is equal to 1.49 g. However, according to the results from the chemical composition test, the composition of Mg is 95.2 %, which means the composition of magnesium is increased (table 1).

Based on the theoretical composition of aluminum, it should be as much as 9 %, but Al composition was decreased to only 3.98 %. This is caused by the occurrence of dross which is a reaction between aluminum and oxygen. In addition, there was a decrease in Ti levels, in the actual condition of the Ti composition which was only 0.0798 %. This happens because the Ti added is a TiB compound, not a pure Ti element, so there are only a few compositions. Furthermore, as much as 0.0494 % boron can produce TiB$_2$ phase as a nucleating agent [6].

3.2. SEM and EDS

SEM and EDS tests were carried out on the samples to examine the elements and their composition in Al-Ti-B nano-sized Al$_2$O$_3$ magnesium composite samples which were given T6 heat treatment and aging at 200 °C (figure 1 and table 2). In addition, this test is carried out to predict the phases and compounds contained in the composite material.

| Table 1. Chemical composition of Mg-as cast and composite material. |
|-------------------------|-----------------|----------------|----------------|-----------------|-----------------|-----------------|-----------------|-----------------|
|                         | Mg  | Al   | Si   | Ti    | B    | Zn   | Mn   | Fe   |
| Mg Al-Ti-B/nano-       | ~95.2 | 3.98 | 0.406 | 0.0798 | 0.0494 | 0.0310 | 0.0116 | 0.0087 |
| sized Al$_2$O$_3$       |     |      |      |       |      |      |      |      |
| composite              |     |      |      |       |      |      |      |      |
| Cr                      | 0.0081 | < 0.00010 | 0.0016 | 0.0348 | 0.00017 | 0.0025 | 0.00047 | < 0.00040 |
| Cu                      |      |      |      |       |      |      |      |      |
| Ca                      |      |      |      |       |      |      |      |      |
| Sn                      |      |      |      |       |      |      |      |      |
| Sr                      |      |      |      |       |      |      |      |      |
| Na                      |      |      |      |       |      |      |      |      |
| Ag                      |      |      |      |       |      |      |      |      |
| Ni                      |      |      |      |       |      |      |      |      |

Figure 1. The SEM result of Mg Al-5Ti-B/ nano Al$_2$O$_3$ 0.20%vf.
Table 2. The EDS result of Mg Al-5Ti-B/ nano Al$_2$O$_3$ 0.20%vf.

| No | Mg  | Al  | Ti  | B   | O   | Si  | Phase formed                                      |
|----|-----|-----|-----|-----|-----|-----|--------------------------------------------------|
| 013| 4.88| 31.34| 62.06|     |     | 1.71| Mg$_{17}$Al$_{12}$, Al$_3$Ti                      |
| 014| 81.04| 5.74 | 0.07 | 11.50| 1.65|      | MgO, Al$_2$O$_3$, Al$_3$Ti                        |
| 015| 33.81| 11.74| 0.06 | 0.91 | 53.49|      | MgO, Mg$_{17}$Al$_{12}$, Al$_3$Ti, Al$_2$O$_3$, TiB$_2$ |
| 023| 63.94| 6.74 | 20.47|     | 7.89| 0.96| Mg$_{17}$Al$_{12}$, Al$_2$O$_3$, Al$_3$Ti          |
| 025| 89.68| 1.90 |     | 1.24| 7.16| 0.02| Mg$_{17}$Al$_{12}$, MgO, Al$_2$O$_3$              |

Figure 2. The XRD result of Mg-Al-5Ti-B/ nano Al$_2$O$_3$ 0.20%vf.

Based on the SEM-EDS results, point number 013 shows the rounded Mg$_{17}$Al$_{12}$ phase due to T6 heat treatment. Then, the intermetallic phase of Al$_3$Ti is formed. Furthermore, at point number 014, there is an oxide compound produced by the reaction between magnesium with nano-sized Al$_2$O$_3$ and as a reaction of melting magnesium with oxygen gas to form MgO. At point number 015 indicates the presence of elements Mg, Al, Ti, B and O. The phases formed at this point are the MgO oxide compounds formed, while the precipitate formed is Mg$_{17}$Al$_{12}$. This is consistent with the solidification reaction of the Mg-Al binary diagram at 436 °C [7]. Then, the formation of TiB$_2$ phase. TiB$_2$ particles are also present in every fine equiaxed dendrite based on SEM results [8]. Based on the results of plotting from each point, the distribution of Mg$_{17}$Al$_{12}$ precipitate phase and Al$_3$Ti intermetallic phase is evenly distributed.

3.3. XRD

X-ray Diffraction test was performed to find out what compounds are formed in the composite sample. The identification of all phases with optical microscope and scanning electron microscope can be seen in figure 2. Based on the graph, it appears that the most detected compounds are Mg$_{17}$Al$_{12}$ phase and periclase or MgO. The MgO compound formed is in accordance with equation $3\text{Mg} + \text{Al}_2\text{O}_3 \rightarrow 3\text{MgO} + 2\text{Al}$ where a stable compound is formed when reacting with magnesium [9].
Theoretically, TiB$_2$ particles act as heterogeneous nucleation of the $\alpha$-Mg substrate. TiB$_2$ particles are also present in every fine equiaxed dendrite based on SEM results. Other oxide compounds observed from the XRD test results consist of two types, namely TiO$_2$ and Mg$_2$TiO$_4$.

3.4. Microstructure
Based on figure 3, it can be seen that microstructure (a) composite without heat treatment, and composite samples given heat treatment T6 with variations in temperature aging are (b) 170 °C (HT 170), (c) 200 °C (HT 200), (d) 230 °C (HT 230), (e) 260 °C (HT 260) at a magnification of 500x.

Figure 3. Microstructure images of (a) non-HT, (b) Mg-Al$_5$TiB/Nano Al$_2$O$_3$ heat treated at 170 °C, (c) 200 °C, (d) 230 °C, and (e) 260 °C at a magnification of 500x.
Based on the results of the microstructure, it can be seen based on the colors of the arrow, where the green colored arrow shows the matrix $\alpha$-Mg. The $\alpha$-Mg matrix is shown as the basis of the phases mentioned earlier, where $\alpha$-Mg is brownish white. Blue arrows indicate the phase $\beta$-Mg$_{17}$Al$_{12}$, orange arrows indicate the TiB$_2$ phase, while yellow arrows indicate the Al$_3$Ti phase. Meanwhile, the formation of Al$_3$Ti at 660 °C. Whereas, TiB$_2$ can be formed at temperatures around 700 °C [9]. Then, there is a black circle which indicates porosity in the microstructure of the composite sample. The transformation of the needle-like structures occurs in this composite. It transforms into spherical. At 200 °C and 230 °C, columnar grain in the samples starts to grow. The higher aging temperature causes the more spherical structure and columnar grain becomes finer. The increasing aging temperature is directly proportional with grain growth. When aging temperature increases, it triggers the columnar grains to become bigger and also the dendrites tend to be more spherical.

3.5. Tensile and elongation

Based on figure 4, the ultimate tensile strength (UTS) value of the composite can be seen on the graph. At 170 °C aging temperature, Mg-Al5TiB composite achieved the highest UTS number. This highest number was occurred at the peak age, which achieved at the 170 °C aging temperature. When the temperature increases until it passed the optimum temperature, the microstructure grows bigger. The Mg$_{17}$Al$_{12}$, Al$_3$Ti and TiB$_2$ phases tend to be incoherent when over aging occurs. The phases reach the semi coherent state at the peak age. Mechanical properties decrease during this phenomenon. The process of T6 heat treatment caused the homogenization of the material structure, which made the value of UTS and elongation percentage became higher. The value of elongation at 230 °C was decreased, this is caused by porosity on that sample.

3.6. Hardness, wear rate and impact

The increasing hardness value of Magnesium Al-5TiB happened when the aging temperature increases, but is decreasing after a particular temperature when over aging occurrence takes place. At 200 °C aging temperature, the magnesium composite achieved the highest hardness value. However, sample with 230 °C, 260 °C aging temperature only displays a slight purpose in the reduction of hardness value. When the temperature aging increases to peak age, precipitates will form coarsening which will reduce the strength and hardness of the composite material [10]. Figure 5 impact value data also show that the composite with 200 °C achieved the highest impact value. The trend of graph is quite similar to the hardness test results. Figure 6 shows the wear rate data, based on this graph the high value of hardness in material will have a low wear rate value. This is due to the ability to avoid material loss. If the value of hardness in the material increases, then the rate of wear will decrease. Meanwhile, if the value of hardness of the material decreases, then the rate of wear will increase [11].

Figure 4. (a) Tensile and (b) Elongation.
3.7. Density and porosity

According to the density measurement of magnesium composite, the value of density is 1.73 gr/cm³. This value is higher than the pure magnesium (1.7 gr/cm³) based on the actual density test. This is due to Al-Ti-B (2.75 gr/cm³) and Al₂O₃ (3.95 gr/cm³) that have a higher density. Thus, it affects the density of magnesium composite. The actual density of the composite was decreased if compared to its theoretical density. The decreasing of density value is due to the existence of excess hydrogen in the casting process which enter the molten magnesium during the stirring process. The existence of porosity in the system reduces the tensile strength significantly.

![Figure 5.](image)

(a) Hardness, (b) Wear rate, and (c) Impact.

![Figure 6.](image)

(a) Density, and (c) Porosity of composite material.
4. Conclusion
Magnesium Al-Ti-B composite with nano-sized Al2O3 with T6 heat treatment achieved the higher values than composite without T6 heat treatment. It can be regarded as a selection for varied uses, especially in the automotive application. This is due to the greater mechanical properties which achieved at the optimum aging temperature of 170 °C in ultimate tensile strength. It reaches 65.31 MPa. Then, at 200 °C is obtained in hardness, toughness and wear rate. It reaches 92.4 HRH in hardness value, 0.07 J/mm² in impact value, and 0.00254 mm²/m in wear rate. The T6 heat treatment generates the homogenization structure of material. Also, this treatment triggers the grain and phase transformation which gives the high tensile strength and percentages of elongation. Due to the over aging phenomenon, the material properties decreased, which occurred at a higher aging temperature (230 °C and above). The mechanical properties enhancement is supported by the addition of grain refiner which known as TiB. The result shows the presence of Mg17Al12, Al3Ti and TiB2 as precipitates which affected the mechanical properties of materials.

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