Investigation on mechanical properties of AlZrCr- Al$_2$O$_3$ nanocomposites fabricated by stir casting

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Abstract. Aluminum alloy composite with nano Al$_2$O$_3$ reinforcement will be designed to have good mechanical properties that correspond to its application. The addition of nano Al$_2$O$_3$ in aluminum is to increase strength and stiffness. In this study aluminum matrix made as a master alloy with the addition of zirconium (Zr) and cerium (Ce) to form Al-Zr-Ce alloys which was reinforced with nanoscale alumina particles known as nano aluminum composite which has high strength and stiffness. Master alloy Al-Zr-Ce used as a matrix content of 0.12 wt% Zr and 0.13 wt% Ce, while Al$_2$O$_3$ nano particles (<100nm) used as reinforcement was various in the range of 0-3 Vf%. Aluminum composite was produced by stirring of molten metal with a rotational speed of 500 rpm at a temperature of 750°C in an inert argon gas environment then characterized both mechanical properties and microstructure analysis. The tensile strength increased with increasing Al$_2$O$_3$ nano particles up to 1 Vf%. Mechanical properties of composites were slightly increased and there was no significant change in elongation and hardness, perhaps due to the non-uniformity distribution or clustering formation of particles in the matrix.

1. Introduction

Researchers have conducted numerous studies to improve the mechanical properties of aluminum. Improved mechanical properties of aluminum can be done by adding alloying elements into aluminum or can be done by adding a reinforcing material in aluminum as Al$_2$O$_3$, SiC, CNT etc. Several studies have reported that the addition of rare earth metals to the aluminum can increase the tensile strength and electrical conductivity. If the aluminum containing impurity elements such as Fe and Si added rare earth metals such as Ce then some of these elements will be dissolved in the crystalline aluminum and others will form a metastable compound or the second phase as FeAl$_3$ or Ce$_2$Si$_2$ [1]. Pengfei Li et al [2] also reported that the addition of rare earth metals can alter the distribution of impurities in aluminum. Rare earth metals to form intermetallic bond with impurities and they will segregate at the grain boundaries. The content of rare earth metals on aluminum will refine the grain structure of aluminum so it will improve the mechanical properties. Similarly, the addition of Zr element in aluminum will also improve the mechanical properties. They reported that the addition of 15 % Zr in aluminum will
result in a solution of a second phase or a metastable compound Al$_2$Zr which has a very small grain size.

Commercially pure aluminum has been used as an electrical transmission lines for decades, because it is cheaper than copper. The use aluminum as a conductor tends to increase. Now most of the electrical transmission network has use commercially pure aluminum conductor. Nevertheless, the uses of aluminum as an electrical conductor still need to optimize the required properties as a conductor wire. This has prompted many researchers conduct research to improve the properties of the electrical conductivity, heat resistance and mechanical properties. Akhmad Zamroni[3] has do the addition of 0.1% - 0.3 wt% cerium zirconium in pure aluminum. The addition of rare earth metals cerium at 0.05 wt% and 0.72 wt% can increase electrical conductivity. The addition of zirconium increase durability and lower heat conductivity but cerium additions will increase the electrical conductivity. A similar study conducted by Zulkifli[4] by adding 1.5% - 4.0 wt% Zr and Gunawan[1] by adding Zr 0.04 wt% and 0.13 wt% lanthanum.

Other efforts are needed to improve the behavior of the aluminum conductor is to improve the mechanical properties. One effective way to improve the mechanical strength of the material is the use of nanoparticles as reinforcement in aluminum matrix. It has been well understood that the presence of a dispersion of fine particles (<100 nm) in a metal matrix greatly improves creep resistance, though with only a small volume fraction (<1%)[5]. Research the addition of nanoparticles in aluminum alloys has been performed several people, among others Takashi et al[6] who reported the effect of adding nanoparticles of titanium-zirconium in aluminum alloy. To increase knowledge in the field of strengthening of aluminum alloy, this study was done different things, namely the strengthening of aluminum alloy Al-Zr-Ce by nanosize particles with 0.2-3% olume fraction of Al$_2$O$_3$.

2. Experimen setup and Procedure
2.1. Starting materials
The material used were pure aluminum, Al-Zr5% and Al-Zr10% were supplied by Hunan Jinlianxing Special Material Co., Ltd. as a master Al-Zr5% and Al-Zr10%. These materials were manufactured as alloy of matrix composite in the form Al-Zr-Ce. The particles of Al$_2$O$_3$ was <80 nm as a reinforcement of composite which was supplied by US Research Nanomaterials, Inc. Houston-USA.

The chemical composition of composite master alloy Al-Zr-Ce conducted by Field Emission-Scanning Electron Microscope (FE-SEM) under Energy Dispersive Spectrometer (EDS) and the results of Al-Zr-Ce was shown in the table 1.

| Alloy material | Weight % |
|---------------|----------|
| Al-Zr-Ce      | 98.30    |
| Zr            | 0.12     |
| Ce            | 0.13     |
| Si            | 0.39     |
| Fe            | 0.88     |
| others        | 0.18     |

2.2. Production of Al-Zr-Ce/Al$_2$O$_3$$_{np}$ nano composites
Al-Zr-Ce/ Al$_2$O$_3$$_{np}$ nano composite was produced by stir casting method as is shown in figure 1. The reinforcement of Al$_2$O$_3$$_{np}$ was varied from 0 to 3 Vf%. The casting process start from feeding reinforcements into the matrix by inserting the particle into aluminum foil capsule before put it in the middle of stack small block of aluminum alloy in the furnace. Melts aluminum was degassed by inert argon gas and it were stirred with a rotational speed of 500 rpm for 2 to 3 minutes. When nanosize Al$_2$O$_3$ particles were added in the molten Al alloy, the viscosity of the molten Al alloy significantly increased. Thus, a higher melt temperature of 750°C was used to ensure the flow ability inside a mold. Then the melt metal matrix composite poured into a permanent tensile test mold (figure 2). After pouring is over, the melt was allowed to cool and solidify in the mold.
2.3. Mechanical Tests

Tensile specimens prepared three pieces from each composition as shown in Figure 3. The dimensions of tensile test specimens were gauge-length 32 mm and diameter 6 mm. The tensile properties of the nanocomposite test specimens were tested at universal tensile testing machine Carl Schenck RME100 in accordance with the ASTM E8 standard. The hardness of the samples was measured using macro hardness Brinnel by applying load of 31.25kg with 2.5mm diameter of indenter and the load was applied for 20 seconds. In order to eliminate possible nonhomogenized material effect, a minimum of five hardness readings were taken for each specimen at different locations of the test samples. The specimens for hardness were prepared with dimension of sample is 50 mm length and 10mm thickness. First the surface was ground with emery paper to remove the oxide layer and cleaned. On the other hand, microstructural analysis was prepared by metallography procedure, start from grinding the specimens of 10x10x10 mm through 80, 120, 240, 500, 700, 1000, 1200 grit SiC papers followed by polishing with 6, 3, 1, ¼μm diamond pasta and etch- ing with Keller’s Reagent. The microstructures were obtained by viewing the samples at different magnification levels on optical microscope and FE-SEM.

3. Results and discussion

3.1 Microstructur of Al-Zr-Ce /Al₂O₃ nanoparticle composite

Aluminum alloy Al-Zr-Ce as a matrix of composite has been examined using an optical microscope and FE-SEM images as shown in figure 4. Microstructure of Al-Zr-Ce shows a fine grain size of aluminum alloy. Zirconium was dissolved in pure aluminum matrix whereas cerium went to the grain boundaries together with other impurities such as Fe and Si. The fine grain size is similar to what was reported by AA Rao[8] that adding 0.2 wt% Zr to 99.7% pure Al which causes a fine grain size.

Figure 5 shows microstructures of the aluminum matrix composite with 0.2 Vf% nanoparticle Al₂O₃ content. After addition nanoparticles to molten matrix, the grain refinery has occurred on the matrix even though only occurred in several areas. The occurrence of uneven grain refinement is expected as a result of non-homogeneous distribution of cerium. Distribution of cerium in the composite can be seen in Figure 6 where the elements cerium is marked by a bright white color phase.
Microstructure of Al-Zr-Ce unreinforced has small/fine grain and same precipitate in grain boundary. It is assumed that there is a primary Al in the material which was investigated at low magnification but at higher magnification under EDS, all of phase formed was identified contain of Al, Si, Zr, Ce and Fe in the table 2. The Chinese script was also seen the matrix.

Table 2. Chemical composition of Al-Zr-Ce /Al2O3 composite by EDS in figure 4

| Spectrums | Al       | Zr       | Ce | Fe     | Si     |
|-----------|----------|----------|----|--------|--------|
| 1         | 99.87    | 0.13     | -  | -      | -      |
| 2         | 71.00    | -        | 20.23 | -   | 8.77   |
| 3         | 95.09    | -        | -  | 4.91   | -      |
| 4         | 93.64    | -        | 0.31 | 6.05 | -      |

Figure 4 Microstructures of the as-cast Al-Zr-Ce alloy without reinforcement, (a) optical microscopy image (magnification with an uniform fine grain size and (b) FE-SEM image, EDS analysis Zr in the grain and Ce (bright white color) and impurities Fe and Si at grain boundary, the microstructure of matrix observed as Chinese script.

Figure 5. A typical microstructure of Al-Zr-Ce composite, (a) finer grain size of the metal matrix Al-Zr-Ce in local area then whole area and (b) microstructure of composite with nanoparticle Al2O3 agglomerations

Another observation from the FE-SEM test result in figure 6 showed clearly that there is a black dot on the microstructure of composite which identified as an agglomeration of Al2O3. Analysis Al2O3 particles form clusters with a diameter ± 5μm. Effect of clustering particle will cause Orowan's strengthening mechanism in composites cannot work well because the particles do not inhibit intergranular dislocations due to a larger particle size of 100nm[9]. Some porous was observed at interface which was indicated as dewetting.
3.2 Mechanical properties of Al-Zr-Ce /Al₂O₃np composite

Al-Zr-Ce alloy nano composites with the addition of reinforcing particles 0 to 3.0 Vf% Al₂O₃ have evaluated in the mechanical behavior. The mechanical properties of the composite Al-Zr-Ce /Al₂O₃np with different tensile strength, elongation and hardness composites are presented in Figure 7. The tensile strength increases with an increase in the amount of Al₂O₃ nanoparticles up to 1 % and then decreased when volume fraction of Al₂O₃ nanoparticles more than 1 % Vf. The slight increase in tensile strength is expected due to the effect of smoothing the grain or the effect of Al₂O₃ nanoparticles. Contrary, the tensile strength decreases when the nanoparticles Al₂O₃ more than 1 % Vf due to the effects of the more particle agglomeration. This result is decline from the study reported by Pradeep Rohatgi [10] that nanostructure aluminum can be 10 times stronger than conventional aluminum alloys. Failure of an increase in tensile strength up to 10 times as a result of porosity and nanoscale alumina particles agglomerations. This happens because the smaller the particle, the more easy-going particle clustering and distribution methods of nanoparticles with a mechanical stirring system with 3 minutes stirring time is not optimal to distribute the particles evenly. And also, argon gas was not used to protect the atmospheric air during the stirring process that allows the formation of porosity in the composite.

Figure 6 Microstructure image by SEM of Al-Zr-Ce /1%Al₂O₃ composite with Vf 1% nanoscale alumina, the cluster Al₂O₃ particles is clearly appear in magnification 20,000 times.

Figure 7 Mechanical properties of Al-Zr-Ce /Al₂O₃ np composites, (a) tensile strength, (b) elongation, (c) hardness
The addition of the aluminum alloy Al$_2$O$_3$np Al-Zr-Ce for elongation and hardness do not change much. Increasing the elongation at Vf 1 and 1.5% in Figure 7 (b) is assumed to be the effect of grain size on the homogeneous matrix in which there was not found a finer grain size than the grain size for the whole area.

4. Conclusions

Hardness and tensile strength of aluminum alloy (Al-Zr-Ce ) that reinforced with various percentage of nanoparticles Al$_2$O$_3$ (0-3 Vf%) was examined and compared with matrix alloy. Results of the analysis indicate that by increasing number of nanoparticles, tensile strength of the composites was slightly increased by adding of Al$_2$O$_3$np up to 1 Vf% whereas ductility and hardness are relatively no change by adding of Al$_2$O$_3$np. Observation of microstructure by FE-SEM, and optical microscope also be identified that distribution of nanoparticle on the aluminum matrix are very poor and the grain size of aluminum matrix of composite have changed to be fine grain in localized area of the metal matrix.

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