Production of Aluminium AC8H/Al_2O_{3p} Coated Metal Matrix Composites by Stir Casting Route

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Abstract

Aluminium AC8H reinforced with Al_2O_{3p} coat has been produced by stir casting route. Al-AC8H/ Al_2O_{3p} composite is prepared with a varying volume fraction of reinforced from 2Vf% to 22.5Vf%. The stirring process was carried out at 500-700 rpm to achieve vortex in an aluminium melt using graphite impeller for 15 minutes. The microstructures and the phase present in composites were observed under optical and scanning electron microscope. It is concluded that the distribution of Al_2O_{3p} in Al matrix was random for a lower content of Al_2O_{3p}. The phases present in the composites were $MgAl_2O_4$, MgO, Al-Si and Si eutectic analyzed by XRD and EDS.

Keywords*: Al_2O_3 particles, Stir Casting, $MgAl_2O_4$*

1 Introduction

Aluminium matrix composites (AMCs) have been widely studied[1-4] due to their large variety of mechanical properties depending on the chemical composition of Al-matrix and the reinforced. Al-AC8H is an aluminium alloy with high Si content, good castability, corrosion resistance, machinability and weldability. Therefore, such alloy is usually used for automotive components, such as piston. This material is used as a matrix with Al_2O_{3p} coat reinforced to improve hardness, wear resistance and strength in current research. Cast aluminium matrix particle reinforced composites have higher specific strength, specific modulus and good wear resistance as compared to unreinforced alloys[5-7]. The mechanical properties that can be obtained with Aluminium Matrix Composites (AMCs) in combination with their relatively low cost process have made them attractive[8,9]. Casting route is preferred as it is less expensive and amenable to mass production. Among the entire liquid state production routes, stir casting is the simplest and cheapest one which is generally accepted as a particularly promising route[10-12]. It is also attractive because, in principle, it allows a conventional metal processing route to be used, and hence minimizes the final cost of the product and allows very large sized components to be fabricated[13]. The only problem associated with this process is the non-uniform distribution of the particles due to poor wettability and gravity regulated segregation. In order to achieve the optimum properties of the metal matrix composite, the distribution of the reinforcement material in the matrix alloy must be uniform, and the wettability or bonding between these substances should be optimised [14]. Therefore, in present work, we use coated particles reinforced to improve the wetting system and high mechanical stirring in order to enhance distribution of reinforced particles and superior mechanical properties of the composites produced.

2 Experimental procedure

2.1 *Coating of Al_2O_{3p} particles*

The γAl_2O_{3p} with particles size ranging from 50 to 180μm, was supplied by Merck, Germany. All

particles were previously pre-treated by electroless coating. Nitride Acid (HNO_3), aluminum and magnesium powders were used in the solutions to coat the surface of γAl_2O_{3p}. The metal oxide coating procedure is divided into the following steps:

1. Al_2O_3 particles were cleaned in alcohol for 30 min until translucent and dried at 100°C for 1 hour.
2. The dried powders were gently dispersed in an electroless bath containing aluminium and magnesium powders in HNO_3 solution with composition in **Table 1**.

Table 1: Chemical composition of the electroless metal oxide coating bath

Composition	Solution
HNO_3	40 ml
Aluminum Powder	0.5 gram
Magnesium Powder	0.1 gram

The electroless plating reaction is as follows:

$$HNO_3 \rightarrow H^+ + NO_3^- \quad (2.1)$$

$$Mg + Al + H^+ + NO_3^- \rightarrow Mg^{2+} + Al^{3+} + HNO_3 + 5e \quad (2.2)$$

Where NO_3^-, Mg^{2+} and Al^{3+} ion can be deposited onto Al_2O_3 particles surface. The pre-oxidation of Al_2O_{3p} was conducted in the furnace at 200°C for 1 hour and followed at 400°C for 2 hours (see Figure 1)

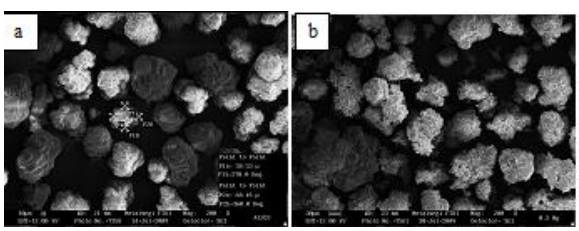

Figure 1: SEM micrograph of Al_2O_3 particles reinforced: a) before coating and b) after coating

2.2 *Casting of composites*

The Al alloy-AC8H ingots with composition in **Table. 2** were melted in a furnace at 700-720°C. *Gases Buble Filtration* (GBF) was carried out using graphite impeller and flushed with UHP argon for 10 minutes. This process is to prevent gas dissolved in the molten metal. It is expected the cast will be free of defects, such as gas holes, porosity and pin holes. Then the molten metal was poured into a small crucible with 2kg capacity and placed in the holding furnace before it was mechanically stirred with a carbon impeller. A controlled argon atmosphere was maintained inside the furnace throughout the experiment to prevent an oxidation melt. At the same time, the Al_2O_{3p} was preheated in a furnace at 400°C for approximately 30 minutes to remove surface impurities. The Al_2O_{3p} coated with 2; 9; 12.5; 18 and 22.5Vf% was poured slowly and continuously into the molten metal *via* a vortex introduced by mechanical stirring and the melt was continuously stirred at 500-700rpm for 15 minutes (**see Figure 2**). The composites then were poured into a pre heat tensile test mold at 680 - 700°C.

Table 2: Chemical composition of Al AC8H (*as-received*)

Element	%Wt
Cu	3.216
Si	11.316
Mg	1.073
Zn	Zn
Fe	0.134
Mn	0.009
Ti	0.219
Ni	0.003
Al	Balance

(a) **(b)**

Figure 2: Stir casting process of Al-AC8H/ Al_2O_{3p} MMC. a) GBF process, b) stir cast of MMC

2.3 *Microstructural analysis*

Microstructures of composites produced by stir casting were analysed both optical and scanning electron microscope (SEM) coupled with energy dispersive spectroscopy (EDS: Link Analytical) to analyse the phase present in composites and

additional phase was identified using X-ray diffractometry, Philips. The fracture surface of tensile test specimens was also analysed by SEM

3 Results and discussion

3.1 *Morphology of Al_2O_{3p} coat*

The morphology of Al_2O_{3p} coat as shown in Figure 1 formed a rounded agglomerate shape. The metal oxide layer on the Al_2O_{3p} surface layer was not homogenous. Such finding was observed with higher magnification in Figure 3a where the surface layer on Al_2O_{3p} was not flat but a relief formation and roughness. The surface layer was analysed by EDS content of Mg, Al and O. It was further analysed by XRD and confirmed as a spinel ($MgAl_2O_4$). This phase is potential to increase the wettability in the Al/ Al_2O_{3p} MMC system[15].

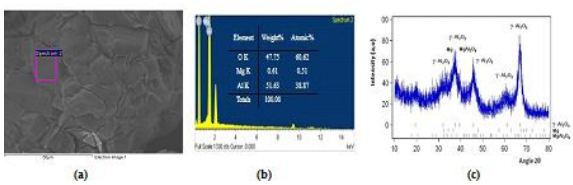

Figure 3: a) The surface layer of Al_2O_{3p} coated , b) surface layer content of Mg, Al and O analysed by EDS and c) XRD confirm was $MgAl_2O$ (spinel)

3.4 *Microstructural analysis*

3.4.1 Microstructure of Al alloy-AC8H/Al_2O_{3p} MMCs

The microstructure of Al-AC8H/ Al_2O_{3p} is show in Figure 4. The Al_2O_{3P} was a random distribution in the Al matrix particularly for its lower volume fraction. There are a number of clusters distributed in the Al matrix for composites with higher volume fraction such as 18% and 22.5% (Figures 4d, 4e). The microstructure of Al-AC8H (as-cast) is shown in Figure 5a. The phase present in the matrix was inter dendritic α-Al and Si eutectic in the form of a needle like and chinese script of intermetallic of Mg_2Si which was analysed by XRD. Si eutectic and dendrite α-Al in the composites were finer than the unreinforced, therefore these finer phases in composites also contributed to the increase in hardness and wear resistance. The cluster distribution of Al_2O_{3p} was found in the Al matrix with higher volume fraction which explains why mechanical properties of these composites decreased. This is also associated with high stress concentration owned by the cluster structure. G.B. Veeresh Kumar et al[16] reported that Al_2O_3 homogenous distribution was achieved at 6Vf% and mechanical properties, such as hardness and tensile strength, continously increased with the increase of Al_2O_{3p} up to 6Vf%.

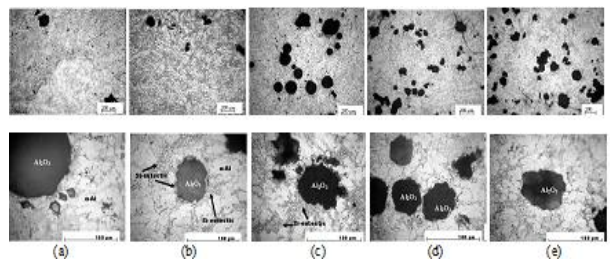

Figure 4: Optical Micrograph of Al-AC8H/Al_2O_{3p} MMCs showing distribution of Al_2O_{3p} in the Al matrix for different volume fractions: (a) 2% (b) 9% (c) 12.5 % (d) 18% and (e) 22.5%

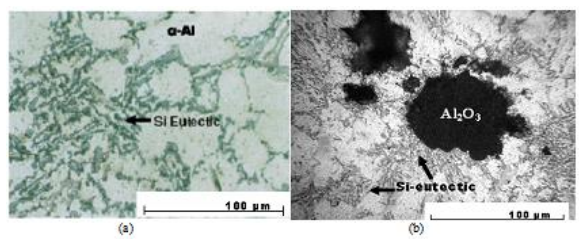

Figure 5: a) Microstructure of Al-AC8H showing interdendritic connected in Al matrix while Si eutectic in the form of needle like accumulated in the interdendritic boundary. b) Microstructure of Al-AC8H/12.5Vf% Al_2O_{3p} where Si eutectic accumulated at the surface of Al_2O_{3p}.

3.4.2 The interface region

The interface region between Al matrix and Al_2O_{3p} was observed under a Scanning Electron Microscope link to EDS in Figure 6. There was a metal oxide layer formed on the surface of Al_2O_{3p} which contained Mg, Al, O, C and Si elements (Figure 6b). A high peak intensity went out for Al-Si which was assumed as interdendritic α-Al and silicon eutectic phases contained in the aluminium matrix. Carbon comes perhaps from a graphite impeller reacted with aluminium matrix during process and silicon eutectic from aluminium accumulated on the surface of Al_2O_{3p} during solidification which was proven by the microstructure of composites as seen in Figure 4 & 5b. The metal oxide layer, such as MgO and

$MgAl_2O_4$, was actually formed during electroless coating on Al_2O_{3p} particles and continued to react with Al during the process to form $MgAl_2O_4$ at the interface. The spinel phase will reduce the surface tension of Al melt so Al_2O_{3p} will be wetted during the stir casting process. Indeed the wetting between Al matrix and Al_2O_{3p} coated was good enough but there was a micro void found at the interface between Al and 22.5Vf% of Al_2O_{3p} as seen in Figure 6a. It is evident that mechanical properties of this composite decreased due to micro void formed at the interface in addition to cluster distribution in the matrix (see Figure 8).

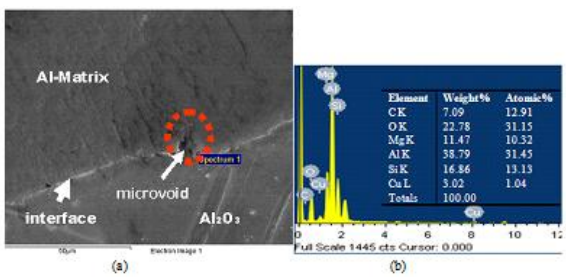

Figure 6: Interface region between Al and 22.5Vf% of Al_2O_{3p} observed by SEM. a) microvoid at the interface and b) phase at the interface detected by EDS

The phases present in Al-AC8H/ Al_2O_3 MMCs were indicated as MgO and $MgAl_2O_4$ formed at the interface which were analysed by SEM link to EDS and confirmed by XRD (Figure 7). This feature contributed to improve the wetting system between Al and Al_2O_3p for a lower content of Al_2O_3p. A high peak intensity went out for Al-Si which was assumed as interdendritic α-Al and silicon eutectic phases contained in the aluminium matrix.

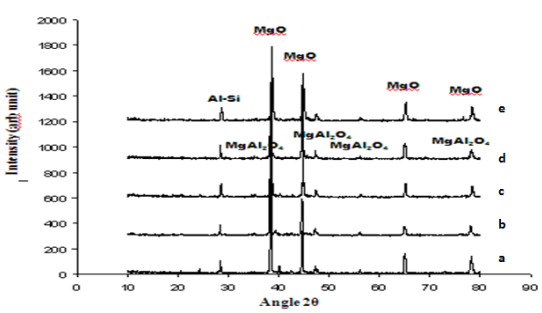

Figure 7: XRD patterns of Al AC8H/Al_2O_{3p} MMCs produced by stir casting : a) 2 Vf% Al_2O_{3p}, b) 9 Vf% Al_2O_{3p}, c) 12.5 Vf% Al_2O_{3p}, d) 18 Vf% Al_2O_{3p}, e) 22.5Vf% Al_2O_{3p}.

3.4.3 Tensile strength

The results of the tensile tests are shown in Figure 8. It is observed that the ultimate tensile strength (σ_{UTS}) was 220 MPa for composites with 12.5Vf% of Al_2O_{3p} which increased 5% than the unreinforced. The tensile strength decreased around 7-25% with the increase of the volume fraction of Al_2O_{3p}. Moreover, a micro void at the interface region of Al alloy/22.5Vf Al_2O_{3p} was found. This void contributed to decrease of tensile strength of these composites. It is believed that the micro void occurred during mechanical stirring and shrinkage at solidification phase. G. Straffelini et.al[17] also reported that Al-5Si with 2-2.5% hydrogen porosity caused a decrease in tensile strength (UTS) around 33.33% i.e, from 225 MPa to 150 MPa. On the other hand, Lyod[18] revealed the role of volume fraction of reinforcement which strongly contributed to the elasticity modulus. The higher the volume fraction, the higher the tensile and yield strengths, but the lower the ductility and fracture toughness. Arsenault[19], with his argument said that increasing the volume fraction of reinforcement was in line with increasing the dislocation density as well as reduced grain size or subgrain in the matrix. Therefore, hardness and strength increased. However, changing mechanical properties was not significant with the existing particles' reinforcement[20].

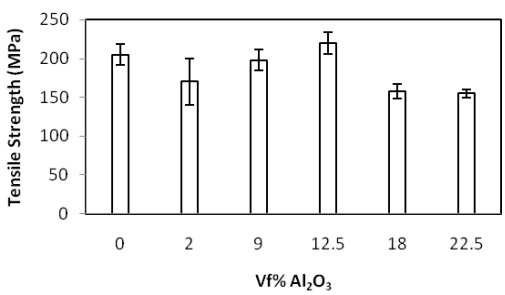

Figure 8: Tensile strength (UTS) Vs Vf% of Al_2O_{3p} in Al AC8H/Al_2O_{3p} MMC system.

3.4.4 Fractography

The fracture surface of Al/Al_2O_{3p} MMC was taken from the tensile test sample shown in Figure 9. The fracture of the composites was caused by porosities present in the matrix as well as poor wetting between matrix and reinforcement (Figure 9a). The non-homogeneous distribution, such as agglomerates and particles cluster, were found at a high volume fraction of Al_2O_{3p} which affected the premature

fracture, therefore the tensile strength decreased in addition to disbonding (Figure 9b & 9e). The cleavage fracture surface and decohesion of Al_2O_{3p} were also found in the composites particularly for 2Vf% of $Al_2O_{3p.}$ Composites with 9Vf% and 12.5 Vf% of Al_2O_{3p} were more ductile due to dimple fracture. The fracture surface at 18 Vf% of Al_2O_{3p} was a combination between the cleavage and the dimple. Large porosity due to poor wetting and cluster distribution of reinforcement was found at 22.5 Vf% of Al_2O_{3p} which caused the worst mechanical properties of the composites compared to a lower volume fraction of Al_2O_{3p} as seen in Figure 9e.

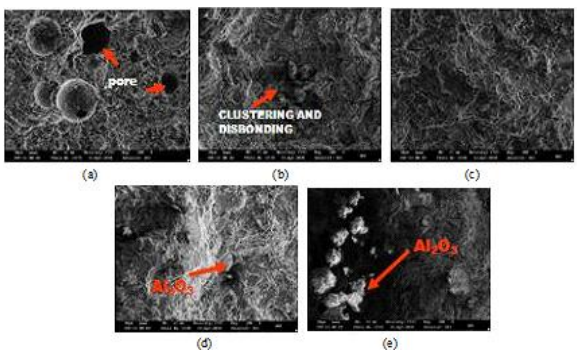

Figure 9: Fracture surface of Al AC8H/ Al_2O_{3p} MMCs : a) 2 Vf% Al_2O_{3p}, b) 9 Vf% Al_2O_{3p}, c) 12.5 Vf% Al_2O_{3p} , d) 18 Vf% Al_2O_{3p} , e) 22.5Vf% Al_2O_{3p}

4 Conclusions

1. Metal oxide layer formed on Al_2O_{3p} surface was MgO and $MgAl_2O_{4.}$ It was an effective method to improve the wetting system between Al alloy AC8H and Al_2O_{3p} in composites system particularly for lower content of Al_2O_{3p} up to 12.5Vf%.
2. Composites produced by stir casting owned random distribution for lower volume fraction of Al_2O_{3p} up to 12.5 Vt% but at higher volume fraction i.e, 18% and 22.5%Vf tend to cluster distribution. Micro void was observed at the interface region particularly for a higher volume fraction of Al_2O_{3p} i,e. 22.5Vf%.
3. The phases present in composites were MgO, $MgAl_2O_4$ and AlSi analysed by XRD for all Vf% of Al_2O_{3p}.

Acknowledgments

Authors would like to thank to Directorate General of Higher Education, Ministry of National Education of Republic of Indonesia for financial support to carry out this research under Competence Grant Project Batch II (Hibah Kompetensi) for 2010-2011 fiscal.

References

[1] Aluminium Matrix Composites, Processing Properties, Chapter 2. *cimewww.epfl.ch/people/cayron/Fichiers/thesebook-chap2*, access on 22 September 2010, pp. 5-20

[2] Surappa M.K., (2003). *Aluminium matrix composite : Challenges and opportunities*., Sadhana Vol. 28, Part 1 & 2, pp.319-334.

[3] Gibson, Ronald F., *Principles of Composite Material Mechanics*, McGraw-Hill. Inc. New York (1994), p 27 – 29.

[4] Kainer, Karl U., (2006). *Basic of Metal Matrix Composites*. Metal Matrix Composite Costome-mate Material for Automotive and Aerospace Engineering. WILEY-VCH Verlag Gmbh & Co. KgaA. pp 5

[5] Akbulut M., Darman, F., Yilmaz., Wear 215 (1998) 170.

[6] Skolianos S., Kattamis T.Z., Mater. Sci. Eng. A163 (1993) 107.

[7] Clyne T.W., Withers P.J., An introduction to Metal Matrix Composites. Cambridge: Cambridge University Press, 1993.

[8] Miracle D.B., Metal matrix composites – From science to technological significance. Compos Sci Technol 2005;65:2526-2540.

[9] Surappa M.K., Prasad S.C., Wear 77 (1982) 295.

[10] Koczak M.J., Prem Kumar M.K., JOM 45 (1993) 44.

[11] Maity P.C., Chakraborty P.N., Panigrahi S.C., Scripta Metall. Mater. 28 (1993) 549.

[12] Kumar S., and Theerthan J. A., *Production and Characterisation of Aluminium Fly Ash Composites Using Stir Casting Method* Thesis Bhacelor of Technology, Department of Metallurgical & Materials Engineering, National Institute of Technology, Rourkela, 2008

[13] Surappa M.K., *J. Mater. Proc. Tech.* **63** (1997), pp. 325–333.

[14] Manoj S., Deepak D.D., Lakhvir S., Vikas C., (2009), Development of Aluminium Based Silicon Carbide Particulate Metal Matrix Composite, *Journal of Minerals & Materials Characterization & Engineering*, Vol. 8, No.6, pp 455-467

[15] Ray S., (1996). *Casting of Composite Components*. Proceeding of the 1995 Conference on Inorganic Matrix Composites. Bangalore, India. 69 – 89

[16] Veeresh Khumar G.B., Rao C.S.P., Selvaraj N., Bhagyashekar M.S., (2010). *Studies on Al6061-SiC and Al7075-Al_2O_3 Metal Matrix Composite*, Journal of Minerals and Materials Characterization and Engineering, Vol.9 No.1, pp 43-55.

[17] Straffelini G., Bonollo F., Molinari A. and Tiziani A., (1997)., *Influence of Matrix Hardness on The Sliding Behavior of 20 vol% Al_2O_3-Particulate Reinforced 6061 Al Metal Matrix Composite*., Wear, Vol. 211, pp.192-197.

[18] Lyod D.J., Int. Mater. Reviews 39 (1994) 1.

[19] Arsenault R.J., (1993). In "Advance Composites'93", Edited by T. Chandra and A.K. Dhingra, pp.19.

[20] Park B.G., Crosky A. G., and Hellier A. K., (2001). *Material Characterisation and Mechanical Properties of Al_2O_{3p}-Al Metal Matrix Composites*, Journal Of Material Science 36, p.2417-2426.