AJSTD Vol. 23 Issues 1&2 pp. 113-122 (2006)

EFFECT OF FLY ASH PARTICLE REINFORCEMENT ON MICROSTRUCTURE, POROSITY AND HARDNESS IN Al-(Si-Mg) CAST COMPOSITES

M.B. Harun*, S.R. Shamsudin, H. Yazid, Z. Selamat, M.S. Sattar and M. Jalil

Industrial Technology Division, Malaysian Institute for Nuclear Technology Research (MINT), 43000 Bangi, Selangor, Malaysia

Received 01 August 2005

ABSTRACT

The microstructure of cast Al-4Si-Mg reinforced with fly ash particles at various particle contents has been studied. The composites were fabricated by stir casting process and characterized by optical microscopy, image analyzer, scanning electron microscopy and hardness measurements. The results showed that particle contents affected to the presence of porosities and hardness of the composites. It was observed that increasing the fly ash content increase the porosity in the composites, with the matrix alloy reinforced with 15 wt.% of fly ash particles having the highest porosity and lowest hardness.

Keywords: Particle contents; Stir casting process; Hardness; Porosities

1. INTRODUCTION

The use of Al-Si alloys in the manufacture of automotive engine components, such as cylinder blocks, cylinder heads, pistons and piston rings, has increased. High purity Al-4Si alloys are mainly used in electrical appliances, automobiles, buildings, chemistry, mining, motorcycles, textile and cooking utensils. The principal characteristics of these alloys are their manufacturability, high wear resistance, low thermal expansion coefficient, good corrosion resistance, low density and improved elevated temperature properties [1, 2]. The incorporation of fly ash particles in aluminum alloy matrix leads to the production of low-cost aluminum composites with improved hardness, stiffness, and abrasion resistance. Aluminum-fly ash has high potential to be used in several components for automotive applications [3, 4, 5]. Fly ash is a particulate waste material formed as a result of coal combustion in power plants. The formation of fly ash is the result of thermal quenching of the mineral matter produced from coal combustion at temperatures in the range of 920^{o}C - 1200^{o}C as it leaves the flame zone. Due to quenching, spherical to rounded fly ash that resulted from the combustion have glassy exterior surface. It is then collected from flue gases using electrostatic, mechanical precipitators or bag houses. There are two types of the fly ash collected namely precipitator ash (solid particles) and chenosphere (hollow microsphere). The chenosphere contains gasses trapped during coal combustion, having low specific density of about 0.6 g cm^{-3} which floats on water, whereas precipitator fly ash has a density in the range of 2.0 g cm^{-3} - 2.5 g cm^{-3}. Major chemical constituents in fly ash are silica, alumina, iron oxide and calcium oxide.

* Corresponding author e-mail: mohd_harun@mint.gov.my

Problems faced during production of these cast composites are due to poor wettability of fly ash and its low weight. The fly ash that is introduced into liquid metal by stir casting often does not uniformly distributed in the alloy matrix [6]. Some porosities in the cast microstructure occur during processing of the composite by using melt-stirring technique and tend to cause a reduction in mechanical and fatigue properties of the composites [7]. In general, porosity arises from three causes: (a) gas entrapment during mixing [8, 13], (b) hydrogen evolution [9], and (c) shrinkage during solidification [9]. In this technique, there are three possible approaches to reduce porosity; (a) environment control, including application of vacuum and inert gas [10], (b) use of baffles and improved design of the stirrer [11], and (c) maintaining suitable levels of process variable such as stirrer speed, size and position [12]. The technical difficulties associated with attaining a uniform distribution of reinforcement, good wettability between substances, and a low porosity material were discussed by Hashim et al [14]. In this study, the porosity and reinforcement particle content in the Al-Si-Mg/fly ash composites were investigated using optical microscopy, image analyzer, scanning electron microscopy and hardness measurements.

2. EXPERIMENTAL PROCEDURE

Chemical composition of matrix alloy is determined by spark emission spectrometer. Its chemical composition is given in Table 1. The reinforcement particles used in this study are fly ash and silicon particles. Chemical composition of fly ash particles is determined by atomic absorption spectrometry as shown in Table 2. The fly ash powder was sieved at 20, 32 and 45 μm. The particle size for as-received (raw) fly ash, sieved fly ash and SiC particles are determined using Particle Size Analyzer. The composites with 5, 10 and 15 wt.% of fly ash particles were prepared by stir casting route involving of melting the alloy in a graphite crucible, creating vortex by mechanical stirring, addition of preheated fly ash particles during stirring and casting the composite melt in a cast iron mould. The set-up for the stir casting can be seen in Fig. 1. To prepare a 1580 g composite of 5 wt. % fly ash, 1500 g cleaned aluminum alloy was mixed with 80 g of fly ash particle. A similar method of the preparation was used for composite of 10 and 15 wt. % of fly ash particles. As for comparison, a composite of 10 wt. % SiC particles also was fabricated.

In composite preparation, the aluminum alloy are super heated above its melting temperature in a bottom pouring composite melting furnace. At the same time, stirring is initiated to homogenize the temperature. Cast iron coated with ceramic propeller is used for the stirring. The propeller is attached to a variable speed motor with a speed of 1200 rpm. The impeller is fabricated in accordance with the law of fluid flow and particle movement to create vortex motion. The depth of impeller is approximately one third of the height of the molten metal from the bottom of the crucible. At a melting temperature of 680^{o}C, the preheated fly ash particles (750^{o}C for 3 hours) will be introduced into the Al alloy melt at a rate of 9 g/min to 10 g/min and, along with Mg pieces weighing a total of 2% by weight of the melt in order to improve the wettability. After 15 minutes of stirring, the mixture of slurry was then poured into a heated permanent mould of 240^{o}C to produce a composite rod.

Optical microscopy was used to examine the general structure and particulate distribution in the matrix and composite. The density of the composites was obtained by the Archimedian principle of weighing small pieces cut from the composite rod first in air and then in water. Brinell hardness measurements were carried out on samples using AFFRI Hardness tester with 62.5 KgF (613.12 N) load and a ball 2.5 mm in diameter.

The microstructure was studied using optical microscope (ZEISS model) and scanning electron microscope (FEI Quanta 400). The hardness of composites and matrix alloy were measured after polishing to a 1 μm finish.

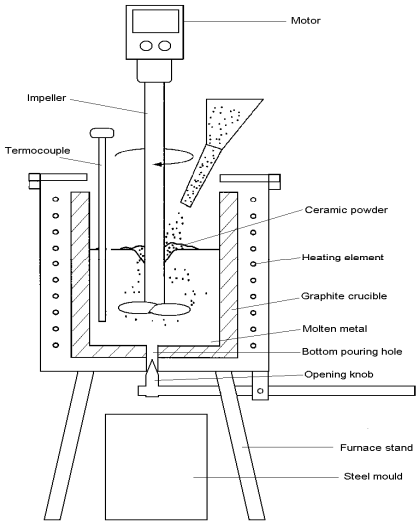

Fig. 1: *Set-up for the stir-casting technique*

Table 1: *The chemical composition of the matrix alloy used in this study*

Element	wt%
Si	4.56
Mg	0.37
Fe	0.39
Mn	0.02
Cu	0.12
Al	Balance

Table 2: *Chemical composition of fly ash particles*

Compound	wt.%
SiO_2	64.58
Al_2O_3	25.11
Fe_2O_3	6.24
K_2O	1.17
CaO	0.13
MgO	2.94
TiO2	1.77
Na_2O	0.79
P_2O_3	0.31
$S2O_3$	0.46

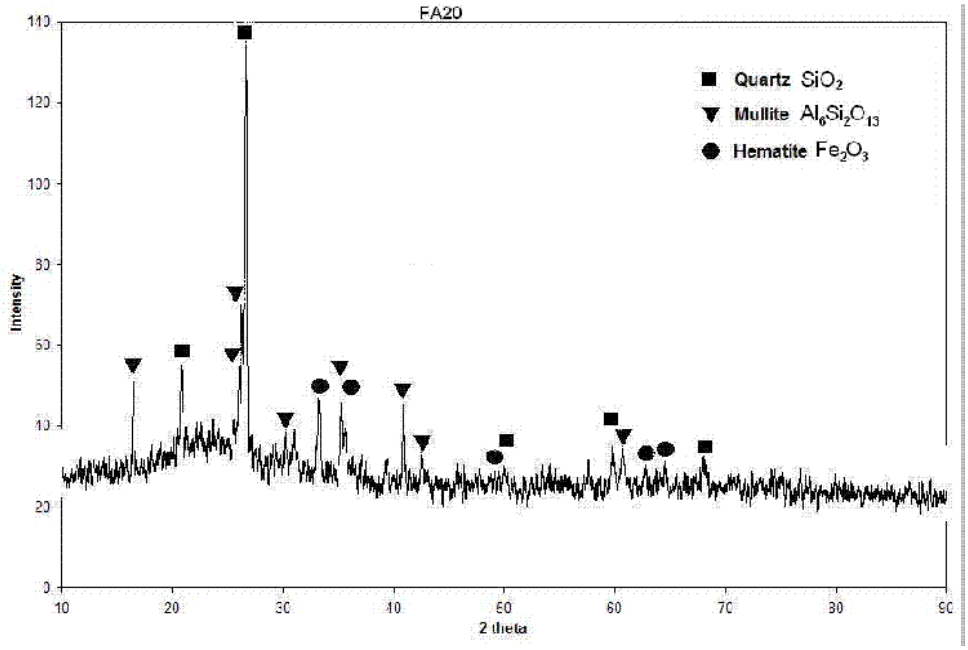

Fig. 2: *XRD pattern of fly ash particles*

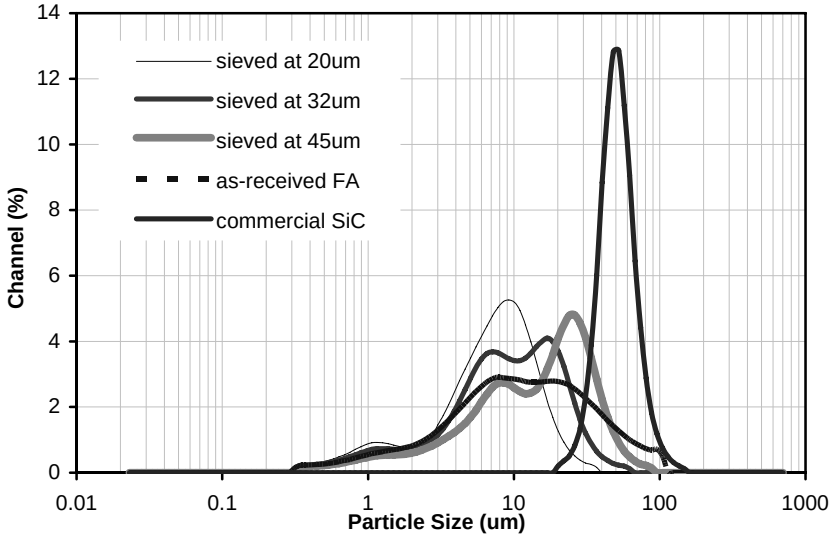

Fig. 3: *Particles size profile for fly ash and SiC particles*

3. RESULTS AND DISCUSSION

3.1 Microstructure

The X-ray patterns of the raw fly ash used in fabricating the composite is shown in Fig. 2. The quartz, mullite and hematite phases were present in the fly ash [15]. The profile of particle size

as measured by Particle Size Analyzer for both the fly ash and SiC particles is shown in Fig. 3. Fig. 4 shows the typical morphology of the fly ash and SiC particle that was used as a reinforcement phase.

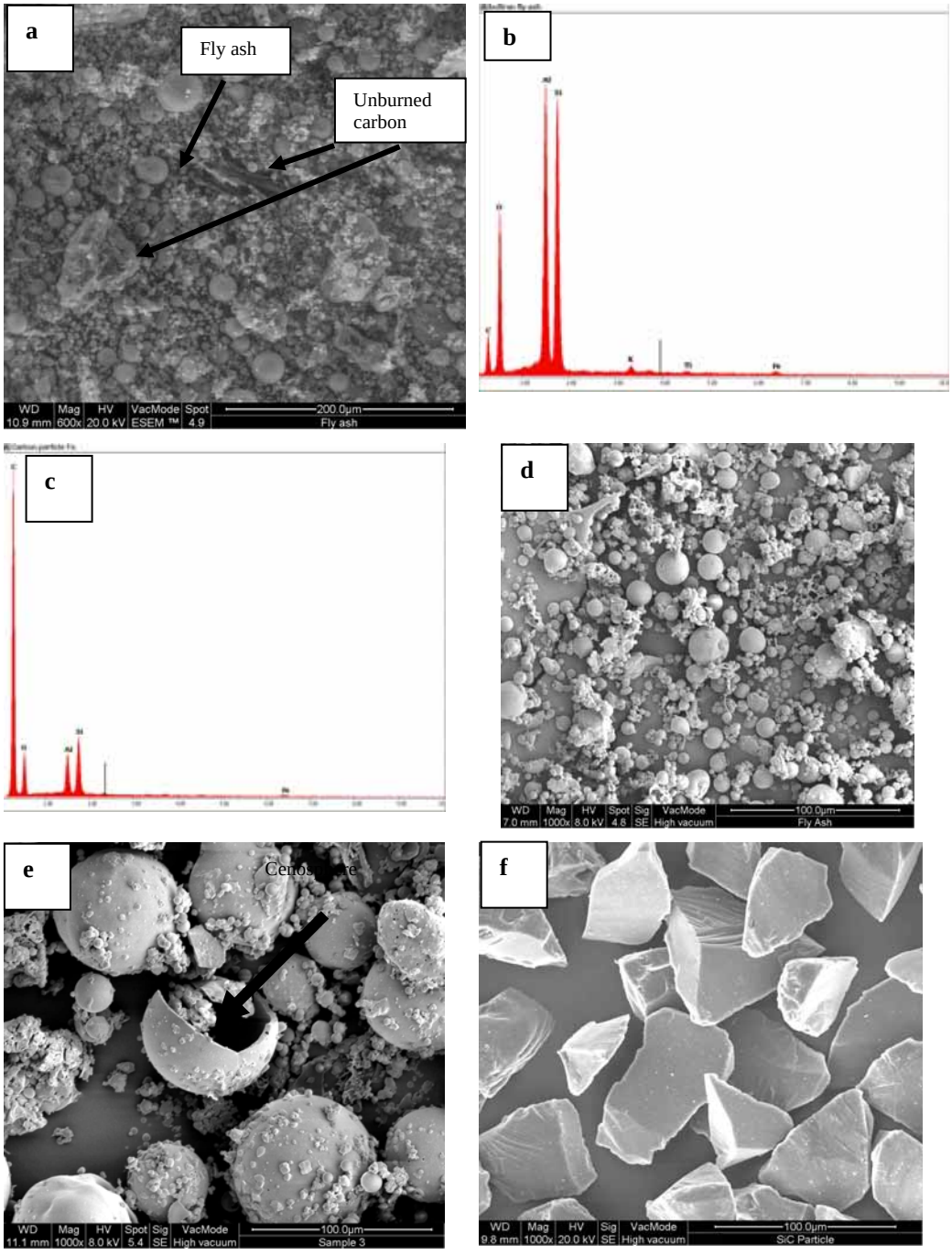

Fig. 4: *SEM Mikrograph on (a) as-received, (b) EDX analysis on fly ash particle (c) EDX analysis on unburned carbon (d) sieved at 20 µm (e) chenosphere fly ash and (f) SiC particles*

Figure 4(a) shows a typical SEM micrograph of the as-received fly ash powders that showed highly agglomerated particles. In coal combustion, the coal containing up to 95 wt.% the inorganic matter of clays, pyrite, quartz and calcite is converted to ash. The microscopic examination reveals that the fly ash samples consist predominantly of glassy spheres due to quenching of the mineral matter when it leaves the flame zone of coal combustion furnace. There are wide ranges of size of particles with spherical, spheroidal and faceted geometry. The spherical and spheroidal particles are known as fly ash precipitator whereas the faceted particles are known as unburned carbon. Most of the unburned carbon is in the form of distinct, fused particles with an extensive macro porous structure, many appearing in the form of fragments as seen in Fig. 4a. The spectrums of EDX analysis taken at fly ash and unburned carbon particles are shown in Figs. 4b and 4c, respectively. The morphology of the fly ash particles is seen to depend on size. The distribution of agglomerated particles decreased when the powder was sieved at 20 μm (Fig. 4d). A smaller fly ash particle exhibits more spherical in shape than the larger ones. Some of the gases involved during combustion are trapped in the fly ash particles, producing cenospheres which float on water. The morphology of the cenospheres is shown in Fig. 4e. Morphology of the SiC particle (Fig. 4f) is observed to be angular in shape.

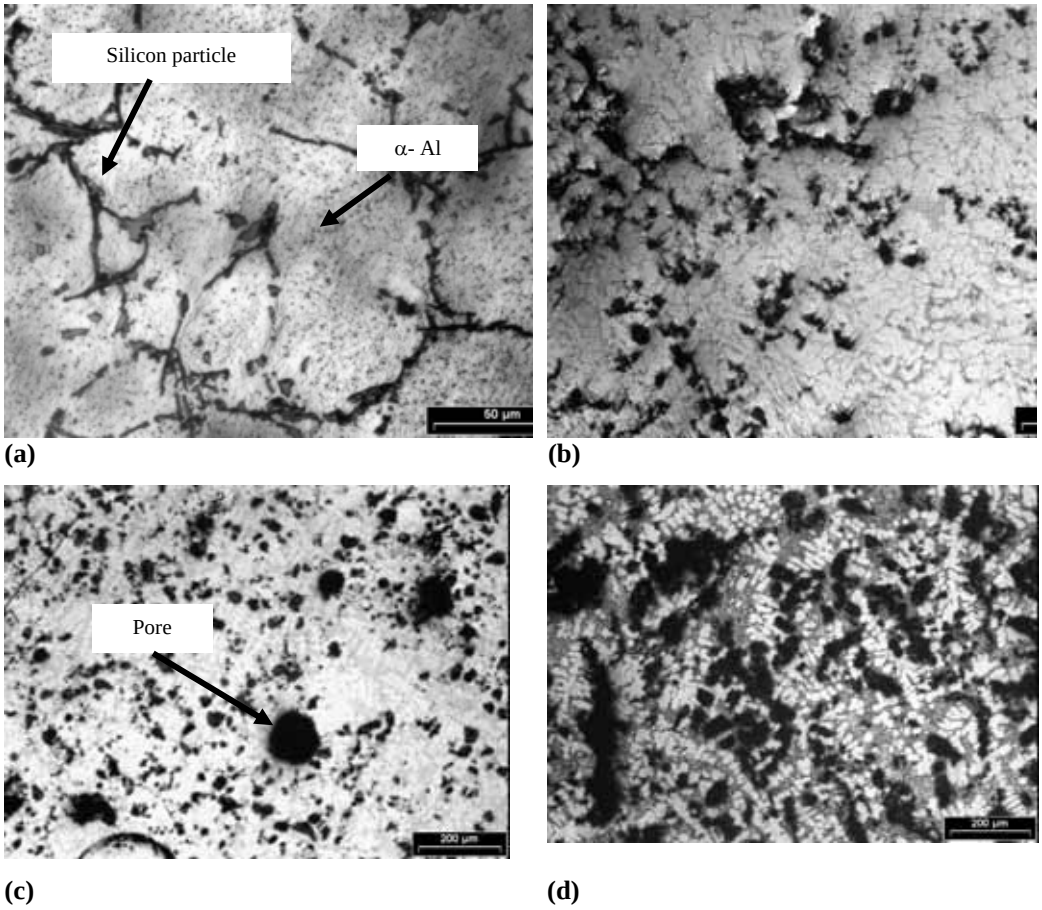

Fig. 5: *Optical microscope micrograph on (a) Matrix Al-4wt %Si alloy, (b) Al-4wt %Si/5 wt.% FA composite (c) Al-4 wt % Si/10wt.% FA composite and (d) Al-4 wt. % Si/15 wt.% FA composite*

Microstructure of matrix alloy contains primary aluminum phase with acicular silicon particles presented along the aluminum grains due solid-liquid interface growing during solidification in the mould (Fig. 5a). Microstructures of the composites containing the fly ash (FA) are shown in Figs. 5b to 5d. Microstructures of the composites containing SiC particles are shown in Figs. 6 and 7. An important feature of the aluminum alloy composite containing fly ash particles is their distribution and interface between reinforcement and matrix. The homogeneous particle was observed in the 10% fly ash and 15%SiC particles reinforcement composite. Some agglomeration is observed in the 15% fly ash particle reinforcement. The particles are nonwettable [16 - 18] by the metallic melt and this requires an external force to overcome the surface energy barriers. It has been shown that alloy chemistry, temperature of particle addition, stirring rate etc. are some of the parameters controlling wetting of the reinforcements by the melt [18]. The force to overcome the surface energy barriers can be provided by stirring the melt with mechanical impeller. The stirring also helps in homogeneous distribution of the reinforcements in the melt [13, 19]. Microstructures of in composite castings depend on the dendritic solidification of composite slurry in the mould [13, 20]. During solidification, particle is rejected by growing crystal and pushed ahead of the advancing interface and then segregated to interdendritic regions or pushed ahead of the growing dendrites resulting in macro segregation along dendrite boundaries or dendrite termination depend on the growth velocity, temperature gradient etc. The presence of porosity cannot be avoided in the composite (Figs. 5b to 5d). The porosity of a composite results primarily from air bubbles entering the slurry either independently or as an air envelope to the reinforcement particles [21]. Entrapment of hydrogen gas in a pocket of interdendritic liquid also can attribute to the formation of porosity due to the decrease in the solubility accompanying solidification.

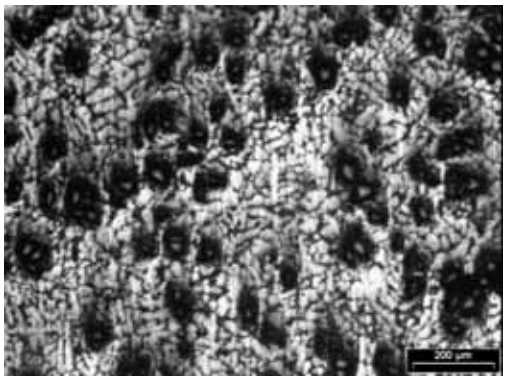

Fig. 6a: *Optical microscope micrograph on Al-4 wt.% Si alloy reinforced with 10% SiC particles*

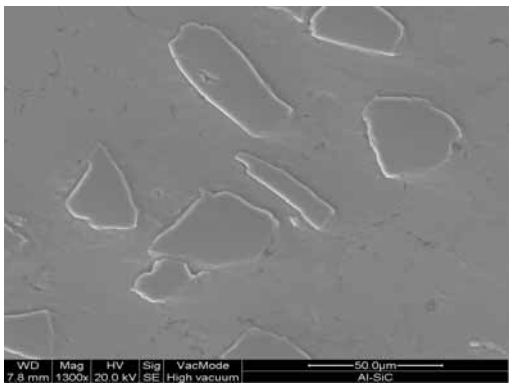

Fig. 6b: *Electron micrograph on Al-4 wt.% Si alloy reinforced with 10% SiC particles*

3.2 Density and Porosity

Porosity assessment is made from density measurement using Archimedes' principle. Fig. 7 shows the presence of particle-porosity clusters. To determine overall porosity content, density measurements were conducted on unreinforced alloy and composites reinforced with 5,10 and 15 wt.% fly ash particles and one with 10% SiC particle. The theoretical density of the composites and alloy matrix specimens was then calculated according to the rule of mixtures. The effect of the reinforcement volume fraction on observed porosity of the composites are summarized in Table 3. It was observed that porosity content increased with increasing fly ash contents. In contrast, Samuel [22] found that the presence of a larger volume fraction of SiC

particles (average particle size of 47 μm) physically restricts the growth of porosity and thus reduce the overall porosity content. Reinforcement particles have a tendency to associate themselves with porosity and give rise to particle-porosity clusters. However, porosity content in the composite reinforced with 10% SiC was much lower compared with all composites reinforced with the fly ash particles. The difference in test results compared with the Samuel [22] work can be attributed to the following reasons: (i) lower reinforcement particles size can cause increase in nucleation sites for porosity at the matrix/fly ash particles; in other words, the contact surface area was increased (ii) broken of cenosphere fly ash particles promote gas porosity and (iii) improper feeding due to fluidity of liquid metal is insufficient to fill the gaps between adjacent particles.

Table 3: *Density and porosity values of the matrix alloy and as-cast composites*

Sample	Weight Fraction of Al Alloy, V^c_1	Weight Fraction of Reinforcement, V^c_2	Experimental Density of Casting P^c_e	Calculated Density of Casting, P^c_k	Porosity in casting, $Pr = 1-P^c_e/P^c_k$ (%)
Fly ash Powder	1.00	0.00	2.316	2.316	0.000
SiC Powder	0.00	1.00	3.383	3.383	0.000
Al alloy	1.00	0.00	2.596	2.596	0.000
Al-5FA	0.95	0.05	2.440	2.582	5.501
Al-10FA	0.90	0.10	2.271	2.568	11.543
Al-15FA	0.85	0.15	2.163	2.554	15.287
Al-10SiC	0.90	0.10	2.496	2.674	6.691

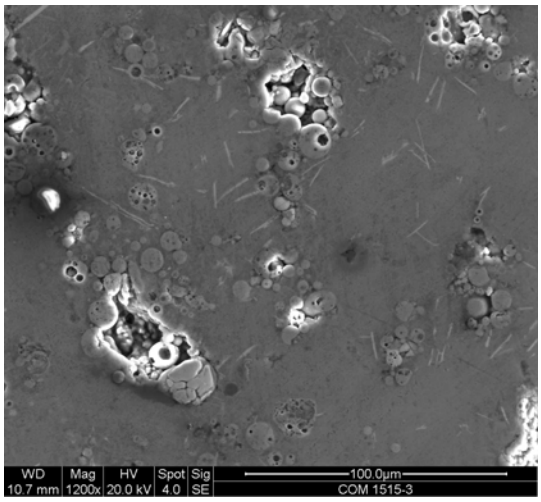

Fig. 7: *Scanning electron micrograph on Al-4% Si alloy reinforced with 10% fly ash particles, shows porosities at the particle clusters*

3.3 Hardness

AFFRI Hardness tester with 62.5KgF (613.12 N) load was used to obtain Brinell Hardness values (HB 10) for the cast composites. The ball size used is 2.5 mm in diameter. The variations of hardness of the composites are shown in Table 4. The hardness of the composite reduced with the volume fraction of fly ash particulates in the alloy matrix due to the increasing of porosity in the composite. This results seem to be in contrast with a work done by other investigator [23], where the introducing SiC into Al matrix resulted in increase in wear and hardness values.

Table 4

Sample	Volume Fraction of Al Alloy , V^c_1	Volume Fraction of Reinforcement, V^c_2	Hardness, HB10
Fly ash powder	0.00	1.00	-
Al Alloy	1.00	0.00	81.23
Al-5FA	0.95	0.05	64.43
Al-10FA	0.90	0.10	45.33
Al-10FA*	0.90	0.10	47.31
Al-15FA	0.85	0.15	33.83
Al-10SiC	0.90	0.10	51.10

Note: *different in mould temperature during pouring.

4. CONCLUSIONS

Conclusions based on the microstructure investigation, density, porosity and hardness measurements are as the following:

i. A stir casting with bottom pouring was developed for manufacturing the metal based composites by adding fly ash and SiC particles into molten aluminum alloy.

ii. Metal Matrix Composite consisting of 10 wt.% fly ash and SiC particles produced homogeneous particle distribution.

iii. Increasing the fly ash content increase the porosity in the composites, with the matrix alloy was reinforced with 15 wt.% of fly ash particles having the highest porosity and lowest hardness.

iv. Hardness of the aluminum alloy improved significantly by addition of SiC particles into it.

ACKNOWLEDGEMENTS

This research project was funded by Ministry of Science, Technology and Innovation (MOSTI) Malaysia under IRPA:03-01-03-0009-EA001. We would like to thanks to Dr Abd. Nassir Ibrahim, Director of Industrial Division, Dr Azali Muhamad, Head of Materials Technology Group, MINT for their advice and valuable discussion.

REFERENCES

1. Bindumadhavan, P.N., Chia, T.K., Chandrasekaran, M., Wah, H.K., Lam, L.N., and Prabhakar, O. (2001), Mater. Sci. Eng. A315, pp. 217-226.
2. Mantaux, O., Lacoste, E., and Danis, M. (2002), Comp. Sci. Tech., vol. 62, pp. 1801-1809.
3. Rohatgi, P.K. and Guo, R.Q. (1997), Fuel and Energy Abstracts, vol. 38, Issue 5, September 1997, p. 319.
4. Rohatgi, P.K., Guo, R.Q., Iksan, H., Borchelt, E.J., and Asthana, R. (1998), Mater. Sci. Eng., A244, pp. 22-30.
5. Rohatgi, P.K., Guo, R.Q., Keshavaran, B.N., Golden, D.M. (1995), Trans. Am. Foundrymens's Soc., vol. 103, pp. 575-579.
6. Rohatgi, P.K. (1994), J. Met., vol. 46, pp. 55-59.
7. Caceres, C.H and Selling, C.H. (1996), Mater. Sci. Eng., A220, pp. 109-116.
8. Miwa, K. and Ohashi, T. (1990), Preparation of Fine Particle Rin forcement Al, Alloy Composites by Compocasting Process, in Proc. of 5th Japan-US Conf. on Comp. Mater. Tama City, Tokyo, pp. 355-362.
9. Lajoye, L. and Suery, M. (1987), Proc. Conf on Solidification Processing, Sheffield, pp. 473-476.
10. Girot, F.A., Albingre, L., Quenisset, J.M., and Naslain, R. (1987), J. Met., vol. 39, pp. 18-21.
11. Harnby, N., Edward, M.F., and Nienow, A.W. (1985), Mixing in process industries, Butterworths, London.
12. Ghosh P.K. and Ray, S. (1988), Indian J. Technology, vol. 26, p. 83.
13. Surappa, M.K. (1997), J. Mater Proc. Tech., vol. 63, pp. 325-333.
14. Hashim, J., Looney, L., and Hashmi, M.S.J. (1999), J. Mater. Process. Tech., vols. 92-93, pp. 1-7.
15. Matsunaga, T., Kim, J.K., Hardcastle, S., and Rohatgi, P.K. (2002), Mater. Sci. Eng., vol. A325, pp. 333-343.
16. Delannay, F., Froyen, L., and Deruythere, A. (1987), J. Mater. Sci., vol. 22, p.1.
17. Rohatgi, P.K., Asthana, R., Yadav, R.N., and Ray, S. (1990), Metall. Trans., vol. 21A, p. 2073.
18. Asthana, R. and Tewari, S.N. (1993), Comp. Manufacturing, vol. 4, p. 3.
19. Ilegbusy, O.J. and Szekely, J. (1988), J. Coll. Interface Sci., vol. 125, p. 567.
20. Stefanescu, D.M., Moitra, A., Kakar, A.S., and Dhindaw, B.K. (1990), Metall. Trans., vol. 21A, p. 231.
21. Ghosh, P.K. and Ray, S. (1984), Trans. Jpn. Inst. Met., vol. 25, p. 440.
22. Samuel, A.M., Gotmare, A., Samuel, F.H. (1995), Comp. Sci. Tech., vol. 53, pp. 301-315.
23. Sahin, Y. (2003), Material and Design, vol. 24, pp. 671-679.