

Thermal, Hardness and Microstructural Characterization of Al-Si-SiC_p Composites

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Abstract

This study investigated the effects of silicon and silicon carbide particles contents on the thermal, hardness and microstructural behaviour of Al-Si-SiC_p composites. 16 samples of the composite produced by stir casting technique were of silicon contents of 1, 2, 3 and 4% by weigh, and silicon carbide contents of 0.5, 1, 1.5 and 2% by weight for each composition of silicon. Each of the samples were subjected to homogenizing annealing heat treatment. Differential thermal analysis (DTA), hardness test and microstructural analysis were then performed on the samples from each composition. The results obtained showed that the hardness of the composite increased gradually as the silicon and silicon carbide particles content increased. The micrographs obtained revealed the presence of silicon carbide, silicon precipitates and aluminium carbide (Al₄C₃) within the metallic matrix. The amounts of these phases varied with the silicon and silicon carbide content. All the samples gave DTA curves with major endothermic peaks between 550 – 570 °C and two sets of exothermic peaks between 580 – 610°C for the first set and between 565 – 570°C for the second set. It was inferred from the study that although varied silicon and silicon carbide contents affected the thermal, hardness and microstructural behaviour of the Al-Si-SiC_p composites, the variation of the SiC_p content had a more pronounced effect on the hardness value of the Al-Si-SiC_p composite.

1. INTRODUCTION

A metal matrix composite (MMC) is a composite material with at least two constituent parts, one being a metal. The other material may be a different metal or another material, such as

ceramic or organic compound (Kelly and Davies, 1965). Metal-matrix composites (MMCs) are of interest today because they offer opportunity to tailor a material with a combination of properties unavailable in any single material. For instance, combining various types of fibres of very high tensile strength and modulus of elasticity with the low density metals such as aluminium, titanium or magnesium often lead to a composite material with a higher strength-to-weight or modulus-to-weight ratio than any single known alloy. Despite the fact that aluminium and its alloys have found remarkably wide applications in the field of engineering, a major limitation to their applicability is their poor wear resistance property and the inability to retain their strength under high temperature service conditions (Pay and Deborah, 1995). This is probably responsible for the development and characterization of various aluminium based composites using various reinforcements that can impart good wear resistance and improved high temperature strength.

By carefully controlling the relative amount and distribution of the ingredients of a composite, its properties can be further improved. Hot pressed $Zr_2(Al(Si))_4C_5/SiC$ composites containing 0 – 30 vol.% silicon carbide (SiC) particles have been reported to have improved hardness value, flexural strength, and fracture toughness with increasing SiC volume contents (Guiqing *et al.*, 2010). Furthermore, it has been reported that in Al–Si–Fe/SiC particulate composites, the addition of silicon carbide reinforcement increased the hardness values and apparent porosity by 75 and 39%, respectively. However, density and impact energy decreased by 1.08 and 15% respectively, with increased SiC content in the alloy (Aigbodion, and Hassan, 2006).

Due to the wide industrial application of Al-Si alloys, the effect of varying the amounts of Si and SiC on the thermal, hardness and microstructural properties of the alloys has been investigated in this study.

2. EXPERIMENTAL DETAILS

The major materials used during the course of the study are aluminium scraps, silicon, silicon carbide particles and etchant. The Al-Si-SiC_p composite material was prepared at the Federal Institute of Industrial Research Oshodi (FIIRO), Lagos, Nigeria, by stir casting method. Sixteen different compositions of the composite was cast by varying the silicon and silicon carbide particle (600 grits) contents. The silicon contents were 1, 2, 3 and 4% by weights, and for each composition of silicon, the silicon carbide content was varied from 0.5 to 2.0% by weight at interval of 0.5% wt. The melted composite materials were then cast into cylindrical rods. The furnace charge calculations are as shown in Table 1.

The cast samples were annealed in order to homogenize the composition. They were heated in an OMSZOV electrical furnace which was set to a temperature of 420°C. They were then soaked at this temperature for five hours and furnace cooled.

The spectrometric analysis of the Al-Si-SiC_p composites was carried out at the quality control department of Manaksia Industries Nigeria Limited, Sango Ota. The analysis was carried out to determine the final elemental composition of the composites, after stir casting.

Table 1 : Furnace Charge Calculation for Al-Si-SiC_p Composite with 4% Silicon

| Composite | Al(kg) | Si(kg) | SiC(kg) | T _m (kg) |
|---------------------------------|--------|--------|---------|---------------------|
| Al -4%Si – 0.5%SiC _p | 1.4325 | 0.060 | 0.0075 | 1.5 |
| Al -4%Si – 1.0%SiC _p | 1.4625 | 0.060 | 0.0150 | 1.5 |
| Al -4Si – 1.5%SiC _p | 1.4175 | 0.060 | 0.0225 | 1.5 |
| Al -4%Si – 2.0%SiC _p | 1.4100 | 0.060 | 0.0300 | 1.5 |

The differential thermal analysis was carried out using the NETZCH DTA404PC differential thermal analyser. Each of the samples was put in the sample holder, and the sample and an inert reference material were made to undergo identical thermal cycles by heating to a temperature of 650°C and then cooling to room temperature at a rate of 10°Cmin⁻¹. The differential temperature and temperature signals were then recorded and the plot of ΔT against T was obtained by using a computerized data processing unit.

Samples of the composite materials were subjected to hardness test, using the Brinell Hardness test accessory of the Monsanto Hounsfield Tensometer. Also parts of the heat treated cast samples were cut to suitable sizes. The samples were ground and then polished to a mirror-like surface. The prepared polished surface of each sample was etched with sodium hydroxide solution (10 g NaOH dissolved in 90 ml water), immersed in the etchant for 20 seconds, and then dipped in concentrated HNO₃ to remove stains. The etched samples were mounted on an ACCUSCOPE metallographic microscope and examined using a magnification of X400. Attached to the microscope was an ocular camera and a computer system through which the micrographs were viewed and captured.

3. RESULTS AND DISCUSSION

The results of our investigation have been compiled in three subsections 3.1, 3.2 and 3.3 with respect to the Differential Thermal Analysis, hardness and microstructure, respectively. The elemental composition of the samples with 2% silicon is presented in Table 2.

Table 2: The Chemical Composition of the Al-Si-SiC_p Composite Containing 2% Silicon

| Element | Al | Si | Fe | Ti | Zn |
|----------------|--------|-------|-------|-------|-------|
| Composition(%) | 95.900 | 3.808 | 0.111 | 0.061 | 0.014 |

3.1 Differential Thermal Analysis (DTA)

The DTA curves obtained from the differential thermal analysis are presented in Figure 1. The thermograms show plots of ΔT against T for each composite composition. The red curves (dark background) show the heating process while the purple curves (lighter background) show the cooling process. All the composites gave two exothermic peaks on cooling. However some gave one endothermic peak while others gave more than one. In most of the samples, the major endothermic peak and the first exothermic peak occur between 585 - 595°C.

Al-Si-SiC_p composites containing 1% silicon and SiC_p contents ranging from 0.5 to 2.0% all gave a major endothermic peak each on heating with onsets between 550 and 560°C. However, samples of the same silicon content between 0.5% and 1.5% SiC_p exhibited two other small endothermic peaks with onsets at 555 and 605°C, respectively. Al-Si-SiC_p composite containing 2% silicon and SiC_p contents ranging from 0.5 to 2.0% all gave a major endothermic peak with onsets at about 550°C (Fig. 1). However, sample of this silicon content and 2.0% SiC_p also gave two other small endothermic peaks with onsets at 555°C and 615°C (Fig. 1d). Al-Si-SiC_p composite containing 3% silicon and SiC_p contents ranging from 0.5 to 2.0% all gave a major endothermic peak with onsets between 555°C – 575°C. However, samples of this silicon content and 1.0% and 1.5% SiC_p also both gave two other endothermic peaks with onsets at 555°C and 610°C respectively while that with 2.0% SiC_p also gave two endothermic peaks at 555°C and 595°C. Al-Si-SiC_p composite containing 4% silicon and SiC_p contents ranging from 0.5 to 1.5% all gave a major endothermic peaks with onsets at about 560°C. Also, these samples all gave two other endothermic peaks with onsets at 555°C and 600°C.

Al-Si-SiC_p composite containing 1% silicon and SiC_p contents ranging from 0.5 to 2.0% gave the first exothermic peaks with onsets between 590 - 610°C, and the second exothermic peaks with onsets between 565 - 570°C. Al-Si-SiC_p composite containing 2% silicon and SiC_p contents ranging from 0.5 to 2.0% gave the first exothermic peaks with onsets between 580 - 590°C, and the second exothermic peaks with onsets between 565 – 570°C (Fig. 1). Al-Si-SiC_p composite containing 3% silicon and SiC_p contents ranging from 0.5 to 2.0% gave the first exothermic peaks with onsets between 585 - 610°C, and the second exothermic peaks with onsets between 565 – 570°C. Al-Si-SiC_p composite containing 4% silicon and SiC_p contents ranging from 0.5 to 1.5% gave the first exothermic peaks with onsets between 605 – 610°C, and the second exothermic peaks with onsets between 565 – 570°C.

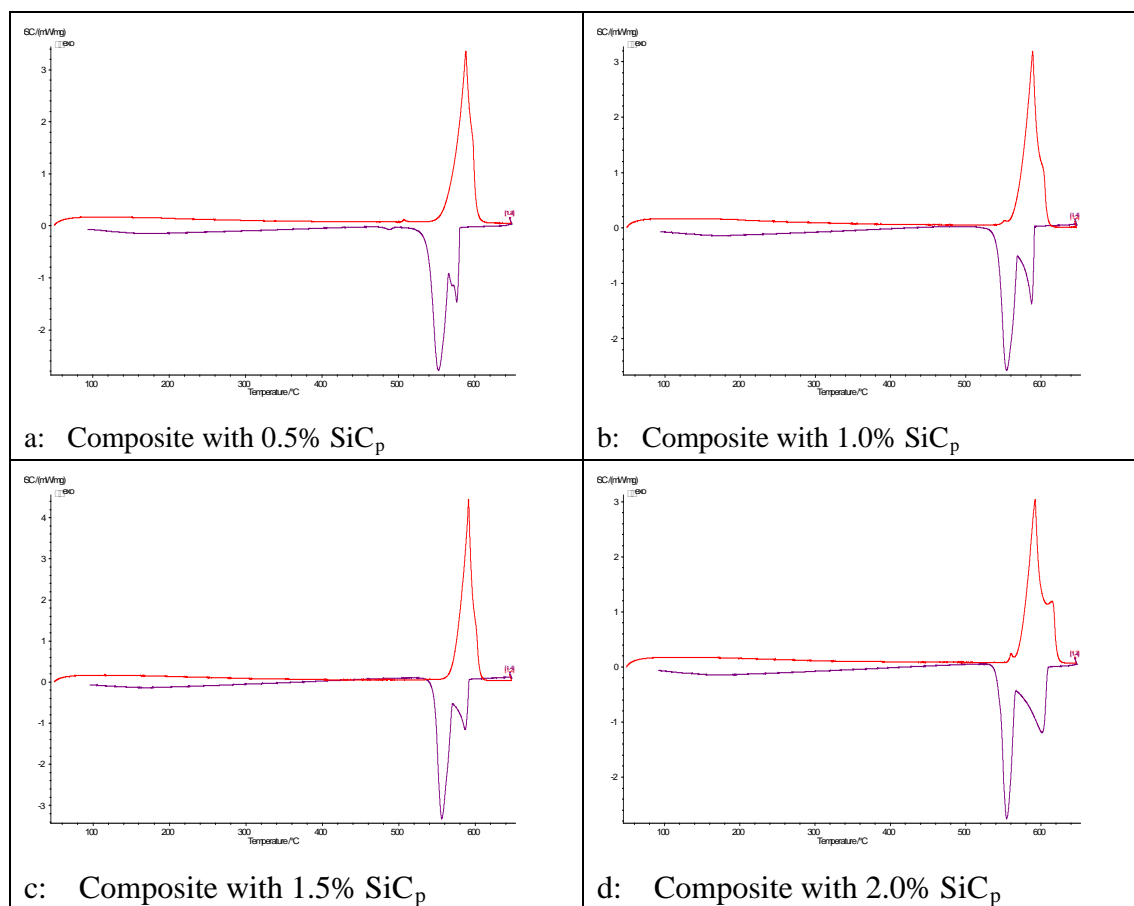


Fig. 1 Thermograms for Al-Si-SiC_p Composites with 2% Silicon Content

The peaks on the DTA curves indicate phase transformations in the composite samples during the heating and cooling cycles. The phases formed during these transformations can be obtained using the aluminium-silicon, aluminium-carbon and silicon-carbon binary phase diagrams. All the samples gave DTA curves with a major endothermic peak during heating with onsets between 550 – 570°C. This could be due to formation of f.c.c Al(α) phase.

All the samples gave two exothermic peaks during cooling. The first exothermic peaks with onsets between 580 – 610°C, could be attributed to the formation of solid solution of Al(α) and Al₄C₃ while the second peaks with onsets between 565 – 570°C, could be linked with the solidification of f.c.c Al(α) phase.

A close look at the DTA curves revealed that composites with 2% silicon content gave thermograms with single endothermic peaks. Composites with 3% silicon content gave thermograms with three endothermic peaks while all the composites with 4% silicon content gave thermograms with multiple endothermic peaks. Therefore, the silicon contents of the Al-Si-SiC_p composite could be said to have affected the number of peaks obtained in the DTA.

3.2 Hardness

The results of the Brinell hardness tests are presented in Figures 2 and 3. Figure 2 shows the variation of hardness values of Al-Si-SiC composites with varying SiC contents, while Figure 3 shows the variation of hardness values of Al-Si-SiC composites with varying silicon contents. Observation revealed that for all the composites produced, hardness values varies with increase in SiC_p content (Fig.2) and silicon content (Fig.3). For a particular silicon content, hardness of Al-Si-SiC_p composite increased gradually when the SiC_p content was increased from 0.5 to 2.0% (Fig. 2). Also, for a particular SiC_p content, hardness of the composite increased gradually when the silicon content was increased from 1 to 4%. (Fig. 3). The slopes of the graphs of hardness values against the SiC_p and silicon contents were obtained from the equations of the trend lines of variation of hardness with SiC_p contents and silicon contents, respectively, with a view to determining which of Si and SiC_p has produced more effect on the hardness of the composite matrix. Table 3 shows the slopes of the graphs of hardness values against the SiC_p contents while Table 4 shows the slopes of the graphs of hardness values against the silicon contents. The tables reveal that increase in SiC_p content has a more positive effect on the hardness values of the Al-Si-SiC_p composite than increase in silicon content.

As observed in Figure 2, the hardness value increases as the silicon carbide particle content increases. This agrees with the results obtained by Aigbodion and Hassan (2006) in a study to investigate the effects of silicon carbide reinforcement on the microstructure and properties of cast Al-Si-Fe/SiC particulate composites. The increase in hardness values is attributed to the distribution of hard and brittle ceramic phases in the ductile metal matrix. The microstructures obtained reveal a dark ceramic and light metal phases, which probably resulted to increase in the dislocation density at the particle – matrix interfaces.

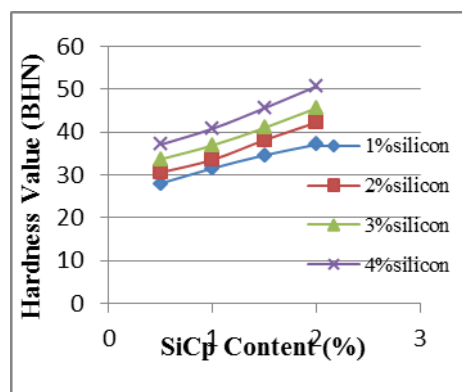


Fig. 2 Variation of Brinell Hardness of Al-Si-SiC_p composite with SiC_p Content

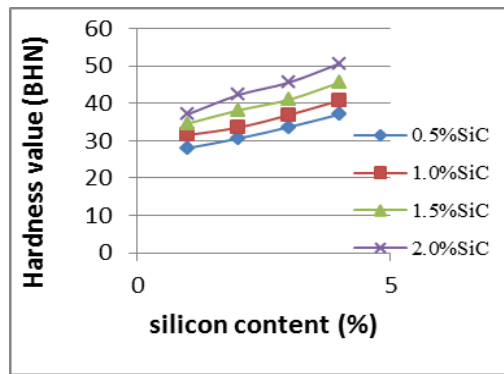


Fig. 3. Variation of Brinell Hardness of Al-Si-SiC_p composite with Silicon Content

Table 3: Slopes of graphs of hardness value against SiC_p contents

| Silicon Content(%) | Slope |
|--------------------------|-------|
| 1 | 6.036 |
| 2 | 7.904 |
| 3 | 7.972 |
| 4 | 9.124 |
| Average(m ₁) | 7.759 |

Table 4: Slopes of graphs of hardness value against Silicon contents

| SiC Content(%) | Slope |
|--------------------------|--------|
| 0.5 | 3.019 |
| 1.0 | 3.114 |
| 1.5 | 3.583 |
| 2.0 | 4.418 |
| Average(m ₂) | 3.5335 |

In the hardness test, severe plastic flow has been concentrated in the localized region directly below the indentation, outside of which material still behaves elastically. Directly below the indentation, the density of the particles increased locally, compared to the regions away from the depression (Manoj *et al.*, 2009).

As the indenter moves downward during the test, the pressure has been accompanied by non-uniform matrix flow along with localized increase in particle concentration, which tends to increase the resistance to deformation. Consequently, the hardness value increases due to local increase in particle concentration associated with the indentation.

Also, the increase in the hardness value of the composites as the silicon contents increase can be attributed to the fact that silicon is a harder phase than aluminium. Tables 3 and 4 show that increase in SiC_p content has a more pronounced effect on the hardness values of the Al-Si-SiC_p composite than increase in silicon content. This can be attributed to the fact that silicon carbide is harder than silicon.

3.3 Microstructure

The micrographs of the cast Al–Si–SiC_p composites are shown in Plates 1a through 1d. The Plates depict micrographs essentially consisting of SiC particles dispersed in a matrix of primary α – Al dendrites and interdendritic α – Al + Si eutectic. Other phases suspected to be present are silicon and Al₄C₃ which are formed as a result of the reaction between molten aluminium and SiC according to the equation:

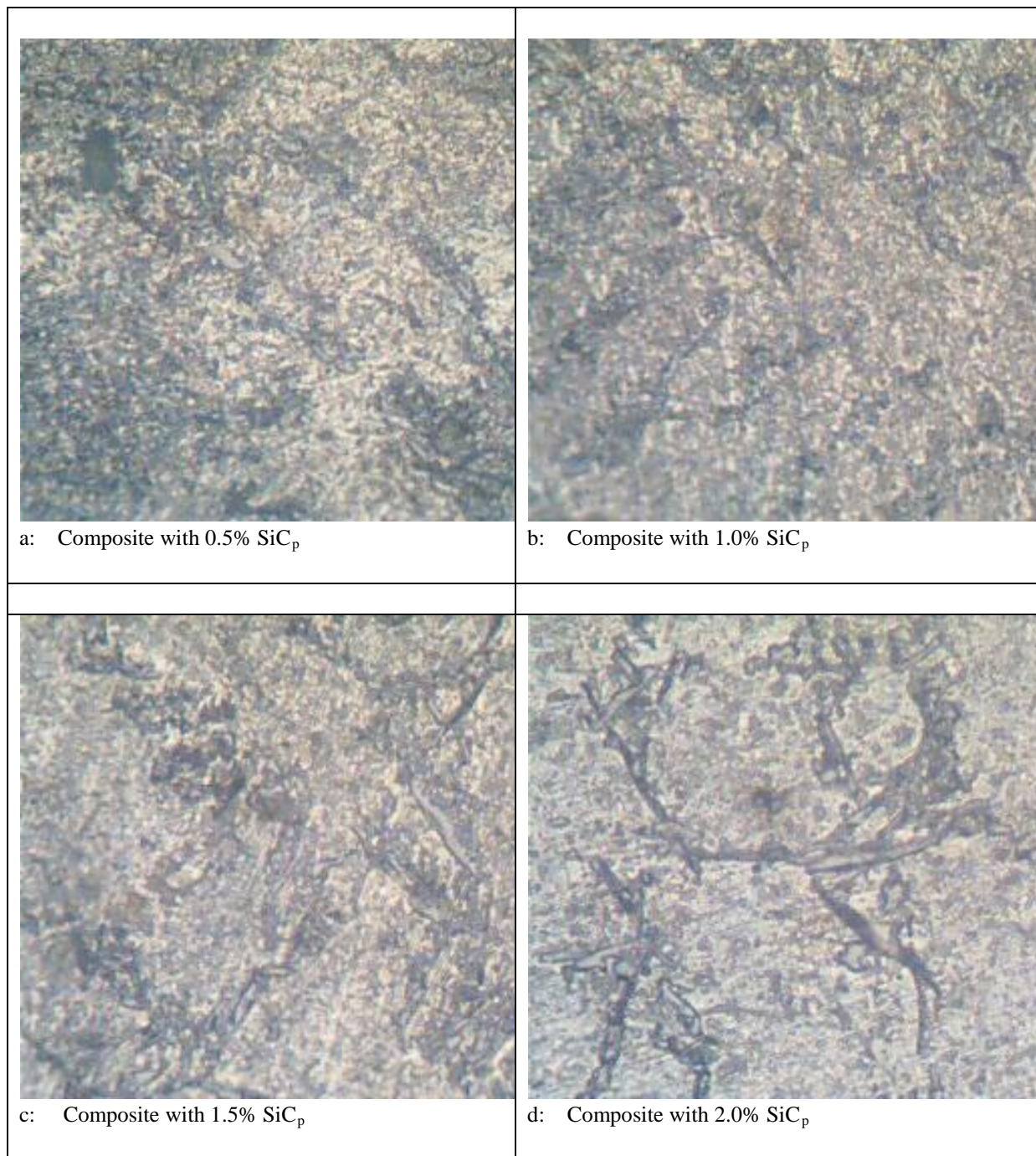


Plate 1: Micrographs of Al–Si–SiC_p composites with 1% silicon after etching with NaOH solution X40 (Dark region represents SiC, while light region represents α – Al)



Plate 1 shows microstructures of cast Al–Si–SiC_p composites with 1% silicon content and SiC_p contents varying from 0.5 to 2.0%. The micrographs reveal homogenous distribution of SiC particles in the aluminium matrix in composites with 0.5 and 1.0% SiC. However, some clustering of SiC were observed in composites with 1.5 and 2.0% SiC.

The micrographs of Al–Si–SiC_p composites with 1% silicon content and 0.5 to 1.0% SiC_p are characterized by homogenous distribution of SiC particles in the aluminium matrix (Plate 1). Therefore, the properties of the composites are expected to be isotropic. Micrographs of Al – Si – SiC composites with 1% silicon content and 1.5% and 2.0% SiC show some clustering of SiC in some part of the composite matrix. This can be explained by the fact that SiC particles have a lower thermal conductivity and heat diffusivity than those of aluminium and therefore, may have been unable to cool down as the aluminium melt. As a result, the temperature of the particles was somewhat higher than that of the liquid alloy. The hotter particles may have heated up the liquid in their surroundings, and thus delay solidification of the surrounding liquid alloy.

Nucleation of α – Al phase starts in the liquid at a distance away from the particles, where the temperature was lower. The growth of α – Al nuclei lead to enrichment of Si in the melt. The enrichment of Si in the melt around the particles leads to heterogeneous nucleation. Another effect of thermal lag was that the melt around the particles would solidify in the last stage. This would make the particles located between dendrites. In other words, the interdendritic clusters of SiC particles have been partially inherited from inhomogeneous distribution of particles in the original slurries (Manoj *et al.*, 2009).

The micrographs of Al–Si–SiC_p composites with 2% silicon content and SiC_p contents varying from 0.5 to 2.0% show a more pronounced presence of Si precipitates as revealed by clusters of silicon in the microstructures. This may have resulted from the chemical reactions between SiC and molten aluminium, which has been reported in the work of Viala *et al.* (1990). The authors found that Al does not react with SiC up to its melting temperature (933 K), but molten Al reacts promptly with SiC, giving different products depending on temperature. Between 940 and 1620 K, Al₄C₃ is produced according to the reaction represented by equation 1. Also, according to Lai and Chung (1994), the reaction is only significant at elevated temperatures, which are encountered during composite fabrication by liquid metal infiltration or stir casting, or during remelting after composite fabrication.

The micrographs of Al–Si–SiC_p composites with 3 to 4% silicon and SiC_p contents varying from 0.5 to 2.0% revealed a less pronounced presence of silicon and Al₄C₃ precipitates. This may be traced to the fact that, introduction of silicon into the molten Al would displace reaction 1 to the left – hand side (Viala *et al.*, 1990). The higher silicon contents in these composites accounts for how the reaction represented by equation 1 was suppressed to a significant level.

4. CONCLUSIONS

Based on the strength of the results obtained in the study, it can be concluded that varying the silicon and silicon carbide contents affects the thermal, hardness and microstructural behaviour of the Al-Si-SiC_p composite. Although, increase in silicon and SiC_p contents increased the hardness value of the composite, the SiC_p content has produced a more pronounced effect on the hardness value of the Al-Si-SiC_p composite than silicon.

REFERENCES

1. Aigbodion, V.S. and Hassan, S.B. (2006): “ Effects of Silicon Carbide Reinforcement on Microstructure and Properties of Cast Al-Si-Fe/SiC Particulate Composites” *Materials Science and Engineering A*, Vol. 447, Issues 1-2, pp 355-360.
2. Guiqing, C. ,Rubin, Z., Xinghong, Z. and Wenbo, H.(2010): “Microstructure and Properties of Hot Pressed Zr₂(Al(Si)₄C₅/SiC Composites” *Microelectronics International*, Vol. 27, No. 1, pp21-24.
3. Kelly, A. and Davies, G.J.(1965): “The Principles of the Fibre Reinforcement of Metal,” *Rev.* Vol.10, No. 37, p 1.
4. Lai, S and Chung D.L.(1994): “Fabrication of Particulate Aluminium-matrix Composites by Liquid Metal Infiltration”. *Journal of Materials Science*, Vol. 29, pp 3128–3150.
5. Manoj, S., Dwivedi, D. D., Lakhvir, S. and Vikas, C. (2009): “Development of Aluminium Based Silicon Carbide Particulate Metal Matrix Composite”. *Journal of Minerals and Materials Characterization and Engineering*, Vol. 8, No. 6, pp 455-467.
6. Pay, Y and Deborah, D.L.(1995): “Powder Metallurgy Fabrication of Metal Matrix Composites Using Coated Fillers”, *The International Journal of Powder Metallurgy*, Vol.31, No. 4, Pp 335-390.
7. Viala, J. C., Fortier, P. and Bouix, J. (1990): "Stable and Metastable Phase Equilibria in the. Chemical Interaction between Aluminium and Silicon Carbide", *Journal of Materials Science*, Vol. 25, pp 1842.