Morphology of Al-2.5%Mg/xSiCp Composite developed by Stir Casting Process

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**ORIGINAL RESEARCH**

**Abstract** The microstructural properties test was made on Al-2.5%Mg/xSiCp composites to study the effects of SiCp coating and the wt.% variation of SiCp on the aluminium composites developed. The research was conducted to enhance and solve the issues associated with low wettability and insufficient bonding between aluminium and silicon carbide that usually associated with stir casting process. The study used magnesium, heat treatment of SiCp, and metallic (SiO2) coating on SiCp, for improved Al-SiCp wettability in the manufacture of composite. The wt.% of SiCp varied from 5 wt.% to 20 wt.% (uncoated and coated) at 5 wt. % intervals. The characterization conducted are XRD, XRF and SEM on the samples developed. XRD confirmed existence of Al with major peaks at 38.5, 45.11, 65.35, and 78.31; while Mg and SiCp were seen at 34.82 and 60.78 respectively. The XRF analysis revealed that major elements are within the range proposed for research with Aluminium contains the highest weight percentage of 70.05 – 86.69 wt.%, followed by Magnesium with 2.46 - 2.57 wt.%, and silicon having varying values of 0.91 wt.% for Sample A, 5.91 – 21.01 wt.% for uncoated samples B – E, and higher values of 6.83 – 24.43 wt.% for Samples F – I compared to the control sample, while all other minerals present are in such negligible proportion. The SEM/EDS results showed a fair dispersion of the SiCp particles in the samples with weight percentages of Al between 54.3 – 72.7 wt.%, Mg with 2.33 wt.% for control sample and a higher of values of 3.90 – 8.22 wt.% for composite samples, and Si with 0.10 – 1.32wt.%. The optimum results have been obtained for 10 wt.% SiCp coated samples. Owning to this best performance test results, the coated Al-2.5%Mg/10wt%SiCp (Sample G) may be adopted as an alternate monolithic alloy to the existing AlMn, AlMg and AlMgSi alloys for structural, heavy machineries and marine applications where light weight is required.

**Keywords** Silicon Carbide, Surface Coating, Surface Oxidation, Composite, Stir Casting, Interface

**1 INTRODUCTION**

It has been observed that attention of scientists and engineers has shifted substantially away from usage of conventional material to the adoption of new emerging engineering material in recent years (Ahmad and AbdulAleem 2002). As some of the recent advances in technologies demands material that possesses uncommon blends of properties that are not obtainable in the usual and conventional metal matrix alloys, especially for light weight applications found in transportation and structural industries (Jayashree et al., 2013).

Fabrication of a metal matrix composite materials is meant to produced materials with enhanced performances compared to its matrix alloy (Wlodarczyk-Fligier et al., 2008). It is observed that aluminium alloys are primary material of consideration for light weight structural application but owning to their low strength, they have been unsuccessful in service to meet the ever-increasing demands for high structural application (Ambali et al., 2022; Odiwo et al., 2021; Birajendu et al., 2019; Abdulwahab et al., 2016; Moses et al., 2016). Thus, Al matrix alloy is reinforced with SiCp to meet this engineering applications challenges coupled with the rising demands for materials with weight-saving applications and excellent performance properties.

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Section C: MECHANICAL/MECHATRONICS ENGINEERING & RELATED SCIENCES


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of Al$_2$O$_3$ formation from the chemical reaction in the manufacture of composite by producing silicon oxide layer between silicon carbide reinforcement particles and aluminium magnesium matrix alloy (Odiwo et al., 2021; Pázmán et al., 2010), thereby influencing the properties and behaviours of developed composites. Also, adoption of magnesium (which is a surface-active element) and heat treated SiC$_p$ particles (coating) before dispersal to molten Al matrix alloy improves the wetting of matrix alloy (Al) with reinforcement (SiC$_p$), producing quality mix of Al-SiC$_p$ particles and easily retaining SiC$_p$ in developed composite samples (Sahoo and Das, 2019; Xie et al., 2019).

In this work, SiC$_p$ was heated up to a temperature of 1300°C in a furnace where a thin layer of SiO$_2$ was formed on its surface. The SiO$_2$ treated (coated) SiC$_p$ and untreated (uncoated) SiC$_p$ were used as reinforcement in the production of Al-2.5%Mg/SiC$_p$ composites by varied the percentage weight of reinforcement from 5 wt.% to 20 wt.% SiC$_p$ (uncoated and SiO$_2$ coated) at 5 wt.% intervals to produced eight (8) composites samples by stir casting process. The aims of the work including examination of effects of coating of SiC$_p$ and variation of wt.% of SiC$_p$ on the developed composites and compare the characterisation behaviours of uncoated and coated Al-2.5%Mg/SiC$_p$ composites developed.

2 EXPERIMENTAL METHODS
2.1 TREATMENT OF SiC$_p$

A measured SiC$_p$ was heated in an Electrical Resistance Heating Furnace (Model: XD – 1700M, manufactured by Zhengzhou Brother Furnace Co. Ltd already set to a temperature of 1300°C required for the surface oxidation to take place, where a thin layer of SiO$_2$ was formed on its surface. The furnace was set to heat the samples at a heating rate of 10°C/min. The sample was kept at this set temperature for about 2 hours before it cooled down in a heating the air at ambient temperature (Ambali et al., 2023). The SiO$_2$: layer formation is in accordance with the equation: SiC + 2O$_2$ → SiO$_2$ + CO$_2$(g) (Parket et al., 2016). By forming a layer of SiO$_2$ between the SiC$_p$ and the metallic Al matrix during heat treatment, destructive AlC$_3$ formation was prevented in the composite. (Ambali et al., 2022; Odiwo et al., 2021; Pázmán et al., 2010; Ambali et al., 2023). This surface oxidation test was conducted at Glass and Silicate Technology Department., Ahmadu Bello University, Zaria, Nigeria.

A. Production of Al-2.5%Mg/xSiC$_p$ Composites by Stir Casting Method

Commercially available 600Grit SiC$_p$, Magnesium (metal) turnings and Al alloy (obtained from NOCAICO, Kaduna, Nigeria) were utilized for the study. Using stir-casting method (Figure 1), Al-2.5%Mg alloy and Al-2.5%Mg/xSiC$_p$ composite materials used for this research were produced at the Foundry Workshop of Hydraulic Equipment Development Institute (HEDI), Kano, Nigeria. Ambali et al., 2023 reported that at temperature of about 660 °C, pure aluminium was melted and mixed with 2.5 wt.% Mg to produce aluminium matrix alloy sample. This mixture was discharged into a prepared rectangular mould and allowed to cooled down and solidified at an ambient temperature in the mould, to developed a rectangular shaped ingot, sample A. To reduce surface tension, the remaining aluminium matrix alloy left in the pot was heated to a temperature of 800 °C above its melting point temperature for good wetting of SiC$_p$ reinforcement which was added. The composite mixture (Al-2.5%Mg/xSiC$_p$) were mixed thoroughly with stirrer, poured in other prepared moulds and allowed to cooled down and solidified at room temperature, for samples B to E respectively. SiC$_p$ initially preheated and pretreated at 1300 °C for surface oxidation prior to incorporation into aluminium matrix alloy were poured into mould, to developed coated composite samples F to I respectively. The work developed Al-2.5%Mg/xSiC$_p$ composites with uncoated and coated 5 to 20 wt.% SiC$_p$ at 5 wt.%intervals (Ambali et al., 2023). The nine samples produced were adopted in the study and were as presented in the Table 1 while some of the raw casted composites are shown in Fig. 2.

<table>
<thead>
<tr>
<th>S/No</th>
<th>Samples</th>
<th>Specification</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>A</td>
<td>Al-2.5%Mg Alloy</td>
</tr>
<tr>
<td>2.</td>
<td>B</td>
<td>Al-2.5%Mg/5wt%SiC$_p$ Composite</td>
</tr>
<tr>
<td>3.</td>
<td>C</td>
<td>Al-2.5%Mg/10wt%SiC$_p$ Composite</td>
</tr>
<tr>
<td>4.</td>
<td>D</td>
<td>Al-2.5%Mg/15wt%SiC$_p$ Composite</td>
</tr>
<tr>
<td>5.</td>
<td>E</td>
<td>Al-2.5%Mg/20wt%SiC$_p$ Composite</td>
</tr>
<tr>
<td>6.</td>
<td>F</td>
<td>Coated Al-2.5%Mg/5wt%SiC$_p$ Composite</td>
</tr>
<tr>
<td>7.</td>
<td>G</td>
<td>Coated Al-2.5%Mg/10wt%SiC$_p$ Composite</td>
</tr>
<tr>
<td>8.</td>
<td>H</td>
<td>Coated Al-2.5%Mg/15wt%SiC$_p$ Composite</td>
</tr>
<tr>
<td>9.</td>
<td>I</td>
<td>Coated Al-2.5%Mg/20wt%SiC$_p$ Composite</td>
</tr>
</tbody>
</table>

![Fig. 1: Stir Casting Process. (Naresh 2006; Gowri et al., 2013)](https://example.com/figure1.png)

B. Characterization of Composite Materials

The characterization of the Al-2.5%Mg matrix alloy and uncoated and coated Al-2.5%Mg/xSiC$_p$ composite materials developed were done by careful examination of their microstructure and elemental analysis. These were achieved using X-ray Diffraction (XRD) for phase identification, JOEL-JSM-7600F Scanning Electron Microscope (SEM) with Energy Dispersive Spectroscopy (EDS) Detector for physical analysis and X-Ray Fluorescence (XRF) for chemical analysis.

Table 1: The Alloy and Composite Samples Developed
rays in X-ray tube being directed to the materials under test, and the rays diffracted in return. The X-rays are filtered to give monochromatic radiation, which are made parallel to focus, and directed towards the specimen/sample under test. The angle formed by incident rays and diffracted rays is a vital feature of all XRD system (Ambali et al., 2023).

Bragg’s Law describes the connection between the electromagnetic radiation wavelength, angle of diffraction and crystalline sample lattice spacing. The machine detects, processes and counts the number of generated diffracted X-rays, and converted diffraction apex to d-spacings for easy verification of minerals as each mineral verified is with a set of distinctive d-spacings. Ambali et al., 2023 reported that characteristically, the mineral identification is obtained by comparing d-spacings with the standard reference patterns, such as Joint Committee on Powder Diffraction Standards (JCPDS). The test was conducted using Rigaku MiniFlex Diffractometer (XRD MiniFlex 300).

3 RESULTS AND DISCUSSION

The study has been carried out by varying weight fraction of uncoated and coated SiCp (5%, 10%, 15%, and 20%) to developed Al-2.5%Mg matrix alloy and uncoated and coated Al-2.5%Mg/xSiCp composites. The characterization of the 9 samples developed were conducted using XRD, SEM and XRF analysis.

3.1 XRD OF UNCOATED SiCp AND SiO2 COATED SiCp

With Rigaku MiniFlex Diffractometer, data were collected over a 2 theta (2θ) range of 5.2545° to 79.9802° at a step size of 0.02°. The phases display in the uncoated (as received) SiCp and the oxidized SiCp at 1300°C were both characterized and compared to examined the consequences of SiO2 coating on SiCp. XRD pattern of the coated and uncoated SiCp given in Figure 3 displays sharp diffraction peaks, signifying the samples’ crystalline structure. The SiO2 coated SiCp displayed more sharp and slighter apex compared to uncoated SiCp, signifying better crystallinity (Qiao et al., 2022), which is commensurable to enhanced mechanical characteristics if employed as reinforcement (Mohit et al., 2019). Moreover, the diffraction apexes at nearly 33.12, 35.08, 36.53, 39.03, 60.78, 66.67, and 72.67 can be indexed as 112, 212, 111, 200, 220, 311 and 222 planes respectively. These reflections are in line with the Joint Committee on Powder Diffraction Standards (JCPDS) Card No. 96-900-6287. Furthermore, the comparison of the XRD patterns of SiO2 coated and uncoated SiCp shows existence of new diffraction peak on SiO2 coated SiCp at 22.5. This denotes the major reflection of SiO2-based on the JCPDS card No. 96-900-6292 used in treating SiCp. The obtained result is in line with the findings of previous studies that characterized SiCp and obtained similar reflections (Ambali et al., 2023; Mohit et al., 2019; Fakhri et al., 2017).
3.2 XRD PATTERN OF THE SAMPLES DEVELOPED

With Rigaku Miniflex Diffractometer, data were collected over a 2 theta (2θ) range of 5.2545° to 79.9802° at a step size of 0.02°. The phases present in the developed Al-2.5%Mg matrix alloy, uncoated Al-2.5%Mg/\textit{x}SiC\textsubscript{p} composites and coated Al-2.5%Mg/\textit{x}SiC\textsubscript{p} composites were analysed and compared. Figure 4 is XRD patterns of developed samples, which revealed that rise in the composition of SiC\textsubscript{p} for the coated and uncoated composites resulted in rise in the reflections of SiC\textsubscript{p} with more visible peaks on the coated composites. Furthermore, the XRD plot shows the existence of Al with major peaks at 38.5, 45.11, 65.35, and 78.31 in line with JCPDS Card No. 04-0787. The existence of Mg and SiC\textsubscript{p} were also seen at 34.82 and 60.78 respectively. From the XRD plots, the existence of Al, Mg, and SiC\textsubscript{p} were confirmed in the uncoated (samples B - E) and coated (samples F - I) composites respectively. As reported by Ambali et al., 2023, these findings are in line with previous research results (Anaee et al., 2017). No new peaks appeared in any XRD pattern, indicating that no new phase formation arose through sintering process. It can therefore be recognized that SiC\textsubscript{p} were used as reinforcement of developed composites. The peak intensities of SiC\textsubscript{p} are distinctly visible, however, instead of the intensities increasing with rise in wt.% SiC\textsubscript{p} for uncoated samples, Sample F with 5 wt.% coated SiC\textsubscript{p} displayed highest intensity of SiC\textsubscript{p}. This implied that the SiC\textsubscript{p} used as reinforcement was thoroughly liquefied in Al-2.5%Mg alloy, in line with the findings of Pichumani et al., (2018).

However, with incorporation of SiC\textsubscript{p}, uncoated (labelled with orange colour), and coated (labelled with blue colour) of different wt.% compositions (Figures 6-7), the particles were noticed agglomerated, though uniformly distributed for Samples B to I. This result is in line with the discoveries of (James et al., 2021), who reported that oxide film formed during sample preparation stops aluminium from attaining intimate contact with reinforcement, and the breakdown of the oxide layer at high temperatures is essential to attain wettability. Hence, the high oxygen content averaging 11.79% from the EDS results is noticed as the corresponding effect preventing the wettability of particles. Furthermore, the insufficient stirring speed may be responsible for the agglomeration and uneven allocation of SiC\textsubscript{p} particles in Al matrix,
shown in Figures 6(a-c) and 7(a, c and d). Consistently, reinforcement particles generated agglomerates during composite production. These agglomerates can only be avoided by stirring at high rpm with limited time and appropriate temperature of the melt. Furthermore, particle agglomeration deprecates the composites mechanical properties (specifically, abrasive resistance) due to lose bond between reinforcement and the matrix. Coating of SiCp resulted in an excellent bond between uniformly distributed SiCp and the matrix (as seen in Figures 6d and 7b) helps the reinforcement to be retained by the matrix, hence, resulting in an improved mechanical property as revealed in the yield strength and tensile strength properties of samples G and H with 10 wt.% and 15 wt.% of coated SiCp, respectively. However, with a further increase in wt.% of coated SiCp up to 20 wt.% (see Figure 6d), agglomerations of the particles were observed again, resulting in a drop in the composite mechanical properties.

3.4 XRF ANALYSIS OF Al-2.5%Mg/xSiCp SAMPLE DEVELOPED

The XRF analysis of samples showed the major elements in the material. It revealed wt% of Al and Mg, as well as the traces of other elements present.

<table>
<thead>
<tr>
<th>Sample</th>
<th>A (wt %)</th>
<th>B (wt %)</th>
<th>C (wt %)</th>
<th>D (wt %)</th>
<th>E (wt %)</th>
<th>I (wt %)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al2O3</td>
<td>86.69</td>
<td>85.96</td>
<td>83.98</td>
<td>80.35</td>
<td>78.66</td>
<td>74.86</td>
</tr>
<tr>
<td>MgO</td>
<td>2.46</td>
<td>2.54</td>
<td>2.57</td>
<td>2.48</td>
<td>2.47</td>
<td>2.51</td>
</tr>
<tr>
<td>SiO2</td>
<td>0.91</td>
<td>9.14</td>
<td>4.37</td>
<td>14.28</td>
<td>0.78</td>
<td>1.28</td>
</tr>
<tr>
<td>FeO</td>
<td>1.34</td>
<td>0.40</td>
<td>0.80</td>
<td>1.08</td>
<td>1.26</td>
<td>1.28</td>
</tr>
<tr>
<td>MnO</td>
<td>0.40</td>
<td>0.42</td>
<td>0.40</td>
<td>0.04</td>
<td>0.04</td>
<td>0.03</td>
</tr>
<tr>
<td>CaO</td>
<td>1.73</td>
<td>1.80</td>
<td>1.50</td>
<td>1.70</td>
<td>1.82</td>
<td>1.80</td>
</tr>
<tr>
<td>P2O5</td>
<td>0.70</td>
<td>0.50</td>
<td>0.46</td>
<td>0.06</td>
<td>0.86</td>
<td>0.24</td>
</tr>
<tr>
<td>K2O</td>
<td>0.35</td>
<td>0.33</td>
<td>0.46</td>
<td>0.50</td>
<td>0.50</td>
<td>0.50</td>
</tr>
<tr>
<td>TiO2</td>
<td>0.12</td>
<td>0.15</td>
<td>0.13</td>
<td>0.15</td>
<td>0.15</td>
<td>0.15</td>
</tr>
<tr>
<td>SO3 (wt %)</td>
<td>1.02</td>
<td>0.65</td>
<td>0.47</td>
<td>0.20</td>
<td>0.20</td>
<td>0.20</td>
</tr>
<tr>
<td>Na2O</td>
<td>0.002</td>
<td>0.02</td>
<td>0.02</td>
<td>1.65</td>
<td>1.45</td>
<td>0.18</td>
</tr>
<tr>
<td>Cl (wt %)</td>
<td>0.20</td>
<td>0.22</td>
<td>0.22</td>
<td>0.02</td>
<td>0.02</td>
<td>0.24</td>
</tr>
<tr>
<td>LOI (wt %)</td>
<td>0.60</td>
<td>0.80</td>
<td>0.80</td>
<td>0.88</td>
<td>0.86</td>
<td>0.90</td>
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<tr>
<td>RbO (wt %)</td>
<td>0.01</td>
<td>0.01</td>
<td>0.01</td>
<td>0.20</td>
<td>0.20</td>
<td>0.20</td>
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<tr>
<td>ZnO (wt %)</td>
<td>0.18</td>
<td>0.06</td>
<td>0.04</td>
<td>0.64</td>
<td>0.62</td>
<td>0.62</td>
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<tr>
<td>Cr2O3 (wt %)</td>
<td>0.04</td>
<td>0.04</td>
<td>0.02</td>
<td>0.09</td>
<td>0.07</td>
<td>0.10</td>
</tr>
<tr>
<td>SrO (wt %)</td>
<td>0.50</td>
<td>0.50</td>
<td>0.60</td>
<td>0.40</td>
<td>0.42</td>
<td>0.42</td>
</tr>
<tr>
<td>NiO (wt %)</td>
<td>0.03</td>
<td>0.03</td>
<td>0.02</td>
<td>0.04</td>
<td>0.06</td>
<td>0.10</td>
</tr>
</tbody>
</table>

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The characterization results of developed samples for major dominant cations using X-Ray fluorescence, are presented in Table 2. It shows that the untreated samples (B, C, D and E) contain exchangeable cations which have close concentrations with treated samples (F, G, H and I). However, comparing the composition of MgO of sample A (control sample) with reinforced samples, an average composition was observed throughout the sample. In addition, it was noticed that increase in the concentration of SiCp from 5 to 20 wt.% for both SiO2 coated and uncoated samples increases the attention of SiO2 among the samples. However, coated samples showed higher compositions of SiO2 than the corresponding samples without coating. This is ascribed to SiO2 used in coating the coated samples. The result affirmed XRD analysis that showed the peak of SiO2 in the coated SiCp patterns. The presence of MgO and SiCp increases the tensile and hardness properties of Al alloy respectively.

4 CONCLUSION
This work has effectively altered SiCp surface by an oxidation method for the production of Al-2.5%Mg/xSiCp composites. Good wetting was achieved between oxidized SiCp particles and Al-2.5%Mg alloy in contrast with as-received SiCp particulate reinforced composites. The oxidized SiCp particulate composite has a continuous interlayer, which produced a high interfacial bond strength due to a reduced interfacial energy, thus improve the compatibility of reinforcement with matrix alloy.

XRD, XRF and SEM studies validated the properties of composites prepared through a coating process. The XRD analysis (plot) shows existence of Al with major peak intensities. Existence of Mg and SiCp also distinctly visibly noticed. XRD plots confirmed existence of Al, Mg, and SiCp in the samples. The XRF analysis revealed that major elements are within the range proposed for research with Alumina contains the highest amount of 70.05 – 86.69 wt.%, followed by Magnesium with 2.46 - 2.57 wt.%, and silicon having varying values of 0.91 wt.% for Sample A, 5.91 – 21.01 wt.% for uncoated samples B – E (probably due to the addition of SiCp), and higher values of 8.43 – 24.43 wt.% for Samples F – I (probably due to the addition of SiO2 coat in SiCp used in the samples) compared to the control sample, while all other minerals present are in such negligible proportion. The SEM/EDS results (analysis) showed a fair dispersion of the SiCp particles in the samples. The dendritic disposition of matrix alloy with the SiCp particle clearly visible in the sample’s micrographs. SEM analysis showed the weight percentages of Aluminium between 54.3 – 72.7 wt.%, magnesium 2.33 wt.% for control sample and a higher of values of 3.90 – 8.22 wt.% for composite samples, and wt.% of silicon as 0.10 – 1.32 wt.%. In contrast to the control sample, for the 600-grit size SiCp adopted in the study, the best results have been obtained at 5 wt.% SiCp for uncoated samples and at 10 wt.% SiCp for coated samples respectively. The sample with coated 10wt SiCp has the overall best performance. Owning to this best performance test results, the coated Al-2.5%Mg/10wt%SiCp (Sample G) may be adopted as an alternate monolithic alloy to the existing AlMn, AlMg and AlMgSi alloys for structural, heavy machineries and marine applications where light weight is required.

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AUTHORS’ CONTRIBUTIONS
Ambali A.O. conducted the study. Oyelaran O.A. and Bolaji B.O. co-supervised the study and assisted in the interpretation of the experimental data obtained. Abdulrahman J., Suleiman O.A. and Araromi O.T. developed the samples according to the specifications and assisted in the laboratory work. All the authors read and approved the final manuscript.

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