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

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ORIGINAL RESEARCH

Abstract- The microstructural properties test was made on Al-2.5%Mg/xSiC_p composites to study the effects of SiC_p coating and the wt.% variation of SiC_p 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 SiC_p and metallic (SiO_2) coating on SiC_p for improved Al-SiC_p wettability in the manufacture of composite. The wt.% of SiC_p was 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 plots confirmed existence of Al with major peaks at 38.5, 45.11, 65.35, and 78.31; while Mg and SiC_p were seen at 34.82 and 60.78 respectively. 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, and higher values of 8.43 – 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 SiC_p 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.% SiC_p for coated samples. Owning to this best performance test results, the coated Al-2.5%Mg/10wt%SiC_p (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 materials 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 SiC_P to meet this engineering applications challenges coupled with the rising demands for materials with weight-saving applications and excellent performance properties.

The problem of an improper distribution of SiC_P in the matrix alloy and less wettability of SiC_P with molten Al are problems usually associated with production processes, methods and materials used among others (Ambali *et al.,*2022; Sijo *et al.,* 2016) are being overcome in this research by varying the process parameters. Heat treatment of reinforcement particles prior to its dispersion into the molten aluminium assist their transfer by creating oxides (Urena *et al.,* 2004). Heating the SiC_P up to 900 °C assists in surface impurities removal and gases desorption, by changing the surface configurations as a result of the formation of a layer of oxide on the surface (Hashim *et al.,* 1999).

Silicon carbide particles (SiC_P) which is one of the frequently used ceramic reinforcing material for composite production owing to its outstanding properties such as availability, high thermal properties, low cost, good corrosion resistance, high modulus and strength, and appropriate compatibility with aluminium matrix alloys (Adebisi *et al.,* 2016) is pretreated with SiO_2 by surface oxidation in an Electric Heat Resistance Furnace at a temperature of 1300 °C prior to its used to overcome the problem of poor wetting with molten aluminium. Surface coating of the SiC_P particles is sometimes essential to enhanced the interfacial bond of the reinforcing phase with the matrix to develop strong adhesion between the two phases. Surface oxidation excite the surface with surface ionic bonds which replaced surface covalent bonds, thereby creating an enhanced adhesive bond amid matrix phases and reinforcement particle, to influence the performances of composites developed (Pázmán *et al.,* 2010). Surface oxidation of SiC_P eliminates the evolution

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of Al_4C_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/xSiC_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 of SiC_Pon the developed composites and compare the characterization behaviours of uncoated and coated Al-2.5%Mg/xSiC_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 the air at ambient temperature (Ambali *et al.*, 2023). The SiO_2 layer formation is in accordance with the equation: $SiC + 2O_2 \rightarrow SiO_2 + CO_{2\,(g)}$ (*Parket al.*, 2016). By forming a layer of SiO_2 between the SiC_P and the metallic Al matrix during heat treatment, destructive Al_4C_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/xSiCp Composites by Stir Casting Method

Commercially available 600Grit SiC_P, Magnesium (metal) turnings and Al alloy (obtained from NOCACO, 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 this study and were as presented in the Table 1 while some of the raw casted composites are shown in Fig. 2.

Table 1: The Alloy and Composite Samples Developed

S/No	Samples	Specification
1.	A	Al-2.5%Mg Alloy
2.	B	Al-2.5%Mg/5wt%SiC_P Composite
3.	C	Al-2.5%Mg/10wt%SiC_P Composite
4.	D	Al-2.5%Mg/15wt%SiC_P Composite
5.	E	Al-2.5%Mg/20wt%SiC_P Composite
6.	F	Coated Al-2.5%Mg/5wt%SiC_P Composite
7.	G	Coated Al-2.5%Mg/10wt%SiC_P Composite
8.	H	Coated Al-2.5%Mg/15wt%SiC_P Composite
9.	I	Coated Al-2.5%Mg/20wt%SiC_P Composite

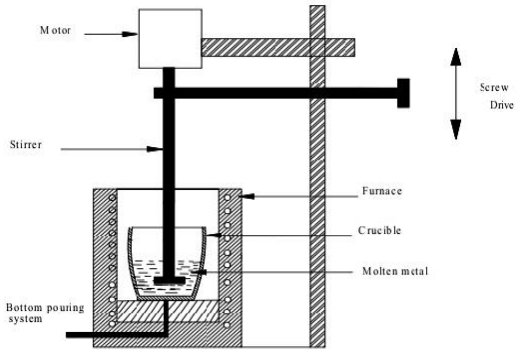

Fig. 1: Stir Casting Process. (Naresh 2006; Gowri et al., 2013)

B. Characterization of Composite Materials

The characterization of the Al-2.5%Mgmatrix 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.

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(a)

(b)

Fig, 2: Raw Cast Composite Materials

i. Scanning Electron Microscope (SEM)

SEM uses electrons for imaging. It is used for direct study of material surfaces as it projects and scans a focused stream of electrons over a surface to create an image. The sample and the electrons inside the beam interact to produce various signals that are utilized to obtain details of the surface's structure and composition. The test was conducted using JOEL-JSM-7600F SEM with EDS Detector (Ambali *et al.,* 2023).

ii. X-Ray Fluorescence (XRF)

XRF is a non-destructive test, an analytical method for obtaining material elemental composition, by an interrelationship of the materials under test with the X-rays emitted. This is achieved by calibration of emitted fluorescent X-rays (secondary) from the samples which is stimulated by the primary source of X-ray. The XRF method operates on the principle of atomic physics and quantum chemistry, where the specimens were shown to entire spectrum of photons consisting of primary radiations emitted from a standard X-ray tube (Ambali *et al.,* 2023).

With pellet technique (PROT-ELE03-v01), the samples were developed and test conducted (analysed) with use of the Philip PW 1210 type λ dispersive XRF spectrophotometer.

iii. X-ray diffraction (XRD)

XRD is utilized to study crystal structures of material. XRD is a fast analytical system mainly employed for crystalline sample phase identification which provide data on the unit cell dimension. X-ray diffraction system is based on interference of a crystalline material sample and X-rays. It has three basic component parts: the X-ray tube, the sample/specimen holder and the X-ray detector. Generally, diffraction systems relied on production of X-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 SiC_p (5%, 10%, 15%, and 20%) to developed Al-2.5%Mg matrix alloy and uncoated and coated Al-2.5%Mg/xSiC_p composites. The characterization of the 9 samples developed were conducted using XRD, SEM and XRF analysis.

3.1 XRD of Uncoated SiC_p and SiO_2 Coated SiC_p

With Rigaku Miniflex Diffractometer, data were collected over a 2 tetha (2θ) range of 5.2545° to 79.9802° at a step size of 0.02°. The phases display in the uncoated (as received) SiC_p and the oxidized SiC_p at 1300°C were both characterized and compared to examined the consequences of SiO_2 coating on SiC_p. XRD pattern of the coated and uncoated SiC_p given in Figure 3 displays sharp diffraction peaks, signifying the samples' crystalline structure. The SiO_2 coated SiC_p displayed more sharp and slighter apex compared to uncoated SiC_p 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 SiO_2 coated and uncoated SiC_p shows existence of the new diffraction peak on SiO_2 coated SiC_p at 22.5. This denotes the major reflection of SiO_2based on the JCPDS card No. 96-900-6292 used in treating SiC_p. The obtained result is in line with the findings of previous studies that characterized SiC_p and obtained similar reflections (Ambali *et al.,* 2023; Mohit *et al.,* 2019; Fakhri *et al.,* 2017).

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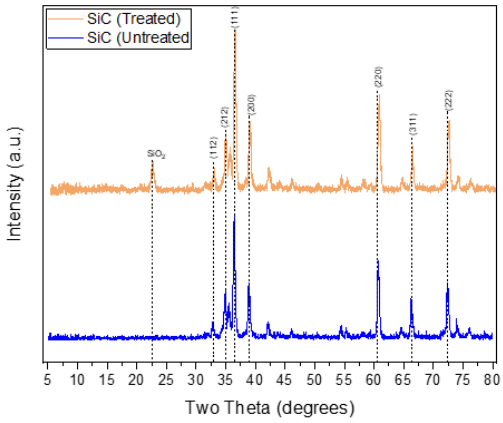

Fig. 3: XRD Pattern of Uncoated SiC_P and SiO_2 Coated

3.2 XRD Pattern of the Samples Developed

With Rigaku Miniflex Diffractometer, data were collected over a 2 tetha (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/xSiC_P composites and coated Al-2.5%Mg/xSiC_P composites were analysed and compared. Figure 4 is XRD patterns of developed samples, which revealed that rise in the composition of SiC_P for the coated and uncoated composites resulted in rise in the reflections of SiC_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_P were also seen at 34.82 and 60.78 respectively. From the XRD plots, the existence of Al, Mg, and SiC_P were affirmed 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_P were used as reinforcement of developed composites. The peak intensities of SiC_P are distinctly visible, however, instead of the intensities increasing with rise in wt.% SiC_P for uncoated samples, Sample F with 5 wt.% coated SiC_P displayed highest intensity of SiC_P. This implied that the SiC_P used as reinforcement was thoroughly liquefied in Al-2.5%Mg alloy, in line with the findings of Pichumani *et al.*, (2018).

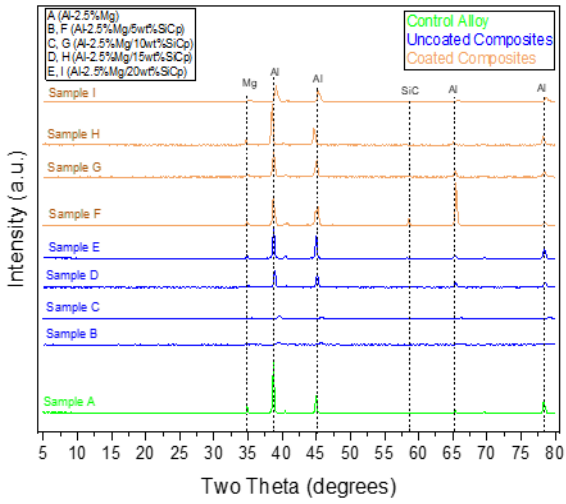

Fig. 4: XRD Pattern of the Samples Developed

3.3 SEM with EDS Analysis of Al-2.5%Mg/xSiC_P Samples Developed

In the work of Ambali *et al.*, 2023, the scanning electron microscopy (SEM) analysis was conducted with JOEL-JSM-7600F using backscattered and x-ray microanalysis with an Oxford EDS detector. The energy of the incident electrons was set to 15 keV. The microstructural investigation of the samples in the Scanning Electron Microscopy (SEM)/Energy Dispersive Spectrometer (EDS) is displayed in Figures 5-7 at a magnification of x28000 and 150 µm full length of the scale. The result (see Figure 5) shows that control matrix alloy without any form of SiC_P reinforcement contains particles of the Al_3Mg_2 phase as dark grey and forms chains along the Al/Al particles in line with the discoveries of Straumal *et al.*, 2010.

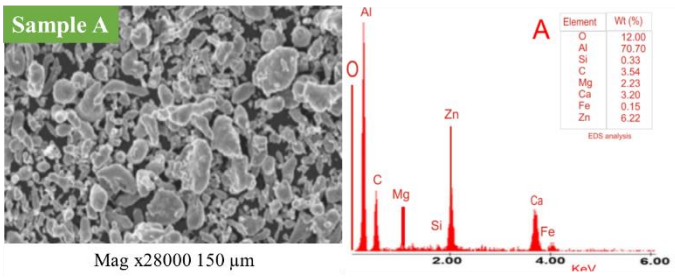

Fig. 5: SEM of Al-2.5%Mg alloy (Sample A)

However, with incorporation of SiC_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_P particles in Al matrix,

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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 depreciates the composites mechanical properties (specifically, abrasive resistance) due to lose bond between reinforcement and the matrix. Coating of SiC_p resulted in an excellent bond between uniformly distributed SiC_p 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.

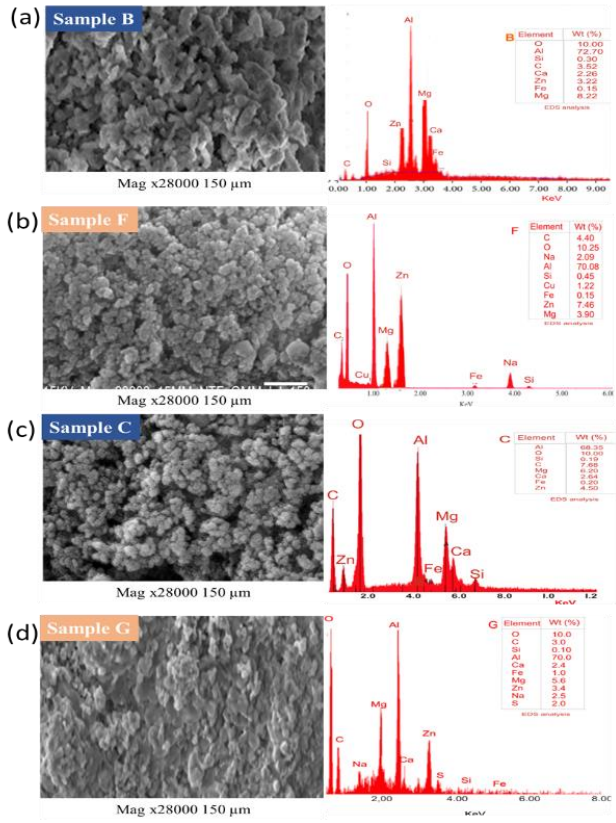

Fig. 6: SEM of (a-b) Al-2.5%Mg/5wt%SiC_p (c-d) Al-2.5%Mg/10wt%SiC_p, uncoated and SiO_2 coated developed samples

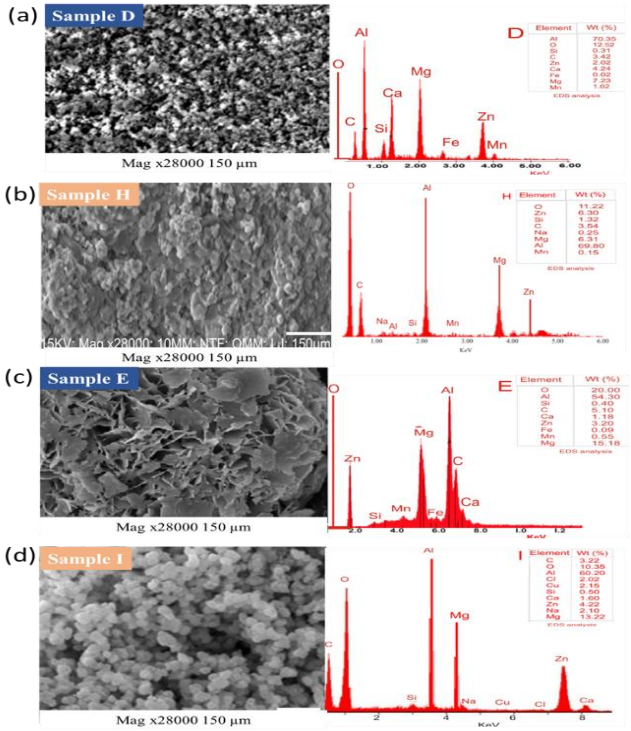

Fig. 7: SEM of (a-b) Al-2.5%Mg/15wt%SiC_p (c-d) Al-2.5%Mg/20wt%SiC_p, uncoated and SiO_2 coated developed samples

3.4 XRF Analysis of Al-2.5%Mg/xSiC_p 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 2. XRF Analysis ofAl-2.5%Mg Alloy and Al-2.5%Mg/xSiC_p Sample Developed

Sample	A	B	F	C	G	D	H	E	I
Al_2O_3 (wt %)	86.69	85.96	83.98	80.35	78.66	74.86	71.08	70.44	70.05
MgO (wt %)	2.46	2.54	2.57	2.48	2.47	2.51	2.48	2.49	2.52
SiO_2 (wt %)	0.91	5.91	8.43	10.41	12.33	15.42	18.99	21.01	24.43
Fe_2O_3 (wt %)	1.34	0.40	0.80	1.08	1.26	0.28	0.24	1.30	1.42
MnO (wt %)	0.40	0.42	0.40	0.04	0.04	0.03	0.04	0.45	0.43
CaO (wt %)	1.73	1.80	1.50	1.70	1.82	1.08	1.07	1.80	1.70
P_2O_5 (wt %)	0.70	0.60	0.40	0.08	0.06	1.25	0.23	0.70	0.52
K_2O (wt %)	0.35	0.30	0.50	0.60	0.50	0.02	0.05	0.40	0.30
TiO_2 (wt %)	0.12	0.15	0.13	0.15	0.15	0.01	0.01	0.10	0.20
SO_3 (wt %)	1.00	0.65	0.47	0.20	0.20	0.12	0.10	1.02	1.02
Na_2O (wt %)	0.002	0.002	0.002	1.65	1.45	0.18	0.20	0.002	0.002
Cl (wt %)	0.20	0.22	0.22	0.02	0.02	0.24	0.22	0.20	0.30
LOI (wt %)	0.60	0.80	0.70	0.88	0.86	0.90	0.90	0.70	0.60
RbO (wt %)	0.01	0.01	0.01	0.20	0.20	0.02	0.22	0.01	0.01
ZnO (wt %)	0.18	0.06	0.04	0.64	0.62	0.12	0.12	0.10	0.10
Cr_2O_3 (wt %)	0.04	0.04	0.02	0.09	0.07	0.10	0.10	0.04	0.04
SrO (wt %)	0.50	0.50	0.60	0.40	0.42	0.02	0.02	0.50	0.40
NiO (wt %)	0.03	0.03	0.02	0.04	0.06	0.10	0.10	0.03	0.03

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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 SiC_P from 5 to 20 wt.% for both SiO_2 coated and uncoated samples increases the attention of SiO_2 among the samples. However, coated samples showed higher compositions of SiO_2 than the corresponding samples without coating. This is ascribed to SiO_2 used in coating the coated samples. The result affirmed XRD analysis that showed the peak of SiO_2 in the coated SiC_P patterns. The presence of MgO and SiC_P increases the tensile and hardness properties of Al-alloy respectively.

4 Conclusion

This work has effectively altered SiC_P surface by an oxidation method for the production of Al-2.5%Mg/xSiCp composites. Good wetting was achieved between oxidized SiC_P particles and Al-2.5%Mg alloy in contrast with as-received SiC_P particulate reinforced composites. The oxidized SiC_P 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 SiC_P also distinctly visibly noticed. XRD plots confirmed existence of Al, Mg, and SiC_P 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 SiC_P), and higher values of 8.43 – 24.43 wt.% for Samples F – I (probably due to the addition of SiO_2 coat in SiC_P 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 SiC_P particles in the samples. The dendritic disposition of matrix alloy with the SiC_P 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 SiC_P adopted in the study, the best results have been obtained at 5 wt.% $SiC_P$for uncoated samples and at 10 wt.% $SiC_P$for coated samples respectively. The sample with coated 10wt SiC_P has the overall best performance. Owning to this best performance test results, the coated Al-2.5%Mg/10wt%SiC_P (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.

Acknowledgments

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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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