Influence of SiO_2 from the rice husk ash and coal fly ash particles on the mechanical behaviour and grain characteristics of the stir-casted hybrid composites

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Abstract

The grain characteristics and mechanical properties of aluminium composites reinforced with Rice Husk Ash (RHA) and Coal Fly Ash (CFA), produced by double stir casting in a sand mould, were investigated. As-cast Al.6063 alloys contained eutectic silicon with sharp, long elongated laths which reduces strength and limits their applications. Cold rolling operation was selected to break those eutectic laths and refine the grains for the purpose of property enhancement. RHA and CFA were chosen because they are economical, possess low density and are available in enormous quantities as solid waste by-products. They contain a substantial concentration of SiO_2 and serve as a viable reinforcement for improving mechanical properties of aluminium composites. The reinforcements were added in varying concentrations (1.5 and 3.0 wt.%). Six specimens were fabricated of which five were cold-rolled. Image J and Origin software was used in evaluating the average grain size of the fabricated alloys and its composites. The results were discussed with the help of grain size distribution and profile plots. Structural and composition analysis were done using scanning electron microscopy (SEM), EDX and XRD. Hardness and tensile tests were conducted on the aluminium composites. The results indicated that Al.6063-3 wt.% CFA composite demonstrated the best balance of mechanical properties with hardness of 79 HV, tensile strength of 151 MPa and average grain size of 11.39 μm . This improvement is linked to the effect of SiO_2 in CFA and plastic deformation of the specimens.

Introduction

Metal matrix composite (MMC) is an indisputable blend of a metallic matrix and reinforcing phase(s) which is adapted towards attaining some specific properties which include good strength and low density [1]. Aluminum is one of the most abundant elements on earth. It has a reasonable specific strength, corrosion and fabrication properties [2]. Aluminium matrix composites have gained and will continue to enjoy much usage as structural materials because of the ease in influencing their mechanical and other properties through the addition of reinforcements [3]. The flexibility of aluminium composite materials for their proposed or target applications enhances their usability in several engineering and structural purposes such as in the aerospace and automotive sector which generally resulted into energy saving and cost effectiveness [4]. The nature and superiority of the composite materials is unswervingly subjective to attaining a homogeneous circulation of reinforcement within the matrix [5]. It has been discovered that important parameters such as the volume fraction, size and distribution of reinforcing particles in the aluminium matrix occupy an imperative role in improving or off-putting the overall properties of the composite materials [6]. In fact, the fabrication of aluminium composites by using both industrial and agricultural waste which in turn reduces environmental wastes and lowers the cost of composite development is now a new trend in the composite world [7]. For example, Daramola et al., 2018 [8] extracted SiO_2 from RHA by sol-gel process and used it as a reinforcement (2, 4, 6 and 8 wt.%) for Al.6063 alloy. The composite melt was cast in a sand mould by double stir casting process. They discovered an improvement in the hardness, yield strength and ultimate tensile strength with a consequential decline in the percentage elongation. They attributed this behaviour to the hard ceramic SiO_2 particles, which effectively restricted activities of dislocation, while making the material brittle. They concluded that the cost of development of the composite was reduced. Bharathi et al., 2017 [9] developed aluminium matrix composite reinforced with 2.5 and 5 wt.% fly ash using stir squeeze cast method. They tested the mechanical and abrasive wear resistance of the synthesized composites and discovered that the strength, hardness and stiffness improved with increasing fly ash content but wear rate increased with increase in load and speed due to the breakage of bonds between the reinforcement particle and the matrix material and also due to enhanced friction at the contact surfaces. Many researchers have worked on the addition of agro-wastes which include coconut shell ash, fly ash, rice husk ash, palm kernel ash, snail shell [10 – 15]. They have been noted to be used as reinforcing materials to improve the engineering properties of aluminium composites but a very small number of studies are dedicated towards quantifying and ascertaining the effect of cold rolling and agro-waste particles on the grain characteristics of the developed composites. This research aims to consolidate existing understanding of the position of agro-waste particulate reinforcement and cold rolling on the grain characteristics of Al-6063 alloy and its composites. This project will likewise center on scrutinizing the upshots of addition of RHA and CFA on the grain characteristics and mechanical behaviour of Al.6063/SFA and Al.6063/RHA composites.

Materials And Method

2.1. Materials

The base material for the investigation is as-cast Al.6063 alloy with the chemical composition shown in Table 1. CFA and RHA were used as reinforcement. Borax ($\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$), was added for improvement of the bonding at the matrix/particle interface.

Table 1
Chemical composition of Al.6063 alloy adopted for this research

Element	Si	Fe	Cu	Mn	Mg	Cr	Zn	Ti	Al
Wt.%	0.45	0.22	0.03	0.03	0.51	0.02	0.03	0.01	Bal.

2.2. Method

The rice husk was obtained from a rice mill in Akure, Ondo State, Nigeria. Thorough washing of the rice husk with distilled water was done in order to remove adhering soil and dust. After that it was dried in an oven at 100 °C for 24 hours. Then the dried husk was treated with 2M of HCl acid before calcinations to increase silica content. After the leaching process, the treated husks were washed with distil water and then dried again. The treated husk was then subjected to calcinations at 700 °C for seven (7) hours in a Morgan ventilated lift-out melting furnace. The RHA was grounded in a ball mill. The final product is shown in figure 1. The microstructure of the treated RHA is shown in figure 2a. The chemical composition of the RHA is shown in Table 2.

Table 2
Chemical composition of treated RHA.

Element	SiO ₂	Al ₂ O ₃	CO ₂	CaO	P ₂ O ₅	MgO	Fe ₂ O ₃	Pb ₂ O ₃	Na ₂ O	K ₂ O	CsO	Rb ₂ O	Other oxides
Wt.%	94.29	4.31	0.39	0.08	0.05	0.04	0.09	0.07	0.05	0.03	0.01	0.01	Balance

The fly ash was obtained from a coal mine in Okaba town in Ankpa local government area, Kogi state, Nigeria. The chemical composition of the CFA is listed in Table 3. The microstructure of the fly ash particles is shown in figure 2b

Table 3
Chemical composition of CFA

Element	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MgO	CO ₂	SO ₃	K ₂ O	CaO	MnO	Other oxides
Wt.%	44.1	22.7	24.2	4.25	2.05	1.4	0.02	0.05	0.96	Balance

2.2.1. Stir casting

The double stir casting processing parameters was adopted in this work. Standard charge calculations were done in order to quantify the amount of the Al. 6063, CFA and RHA required for producing the composites. Borax was added as a wetting agent. It was then heated to 250 °C for 20 minutes to remove moisture, after which it was mixed with the specified amounts of TiO₂. The Al. 6063 was charged into the furnace and melting was allowed to progress until a uniform temperature of 779.3 °C was attained. At this stage, the CFA, RHA and pre-heated borax mixture was introduced into the melt and stirring of the slurry was done using a mechanical stirrer. The stirring operation was performed for 22.5 minutes at 574 rpm. An external temperature probe was utilized in all cases to monitor the melt temperature. After stirring, the composite slurry was reheated and maintained at a temperature of 779.3 °C and then mechanical stirring was performed again. The stirring operation was performed for 22.5 minutes at an average stirring rate of 574 rpm. Casting was then carried out on prepared sand moulds at a pouring temperature of about 780 °C [16].

2.2.2 Cold rolling and heat treatment

A miniature cold rolling machine with capability for rolling of flat and cylindrical profiles was utilized for the cold deformation processing of the Al.6063 alloy its composites. The specimens produced in the form of cylindrical rods were firstly machined to 18 mm diameter and 200 mm length. The samples were then cold rolled using the round grooves of the rolling machine from an initial diameter of 18 mm to a final diameter of 16.2 mm. The rods were cold deformed using four passes (translating to 0.45 mm reduction in diameter per pass) through the rolling dies until the final diameter of 16.2 mm was achieved. The cold rolling was done to reduce volume defects such as voids, blow holes and pores that may have developed during sand casting. The cold-rolled alloy and its composites were then heat treated at 100 °C for 30 minutes in order to release internal stresses generated during cold-rolling operations. It was done at a low temperature and short time of 100 °C for 30 minutes respectively in order to prevent the formation of precipitates [17].

2.2.3. Hardness Test

The specimens are prepared as per the ASTM E92-17 standards [18]. 10x 10 mm² cross section was used as shown in figure 1. The specimens are machined according to the specified dimensions and then polished to eradicate any discontinuity or irregularities on the surface. The hardness test on the specimens was carried out on a Vickers hardness testing machine. Five repeat tests were done to guarantee reliability of results.

2.2.4. Tensile testing

Room temperature uni-axial tension tests were performed. Tensile samples machined from the Al.6063 alloy and composites with the following dimensions. 6 mm diameter and 30 mm gauge length as shown in figure 1. The testing was performed using an Instron universal testing machine, operated at a constant cross head speed of 1 mm/s and the procedure adopted was in conformity with ASTM E8M-91 [19] standards. A minimum of three repeat tests were performed for each test condition to ensure the consistency of the data generated. Ultimate tensile strength and tensile elongation are data that were obtained from this test.

2.2.5. Microstructure

Microstructural exploration was made using a Zeiss scanning electron microscope and JSM 7600F Jeol ultra-high resolution field emission gun scanning electron microscope (FEG-SEM). The as-cast and artificially aged samples for the optical and scanning electron microscopy characterization were thoroughly polished using series of emery papers of different grit sizes ranging from 60 grits to 1500 grits, while fine polishing of the samples were performed using suspensions of polycrystalline diamond with ethanol solvent. The samples for the test were metallographically polished and etched with 1HNO₃:1HCl solution by adequately swabbing it on the samples surface and leaving it for about 3 - 6 minutes which is then accompanied by rinsing in water and drying it before observation in the scanning electron microscope.

Results And Discussions

3.1 Microstructural examination of the as-cast alloys and composites.

In Tables 2 and 3 as well as figures 2, it can be inferred that treated RHA contained a larger percentage of silica than the CFA. The treated RHA yielded a higher concentration of SiO₂ as shown in Table 2 than the untreated CFA as shown in Table 3. This can be directly linked to the exhaustive processing route which involved a conscientious washing of the rice husk with distilled water, drying in an oven at 100 °C for 24 hours, treating with 2M HCl, calcinations for 7 hours at 700 °C helped in reducing or eliminating unnecessary impurities from the RHA [20]. The treatment given to RHA helped reduce drastically the presence of foreign and deleterious elements from the reinforcement. As regards the CFA, several impurities (traces of other elements although in very small quantity) were observed. From the SEM micrograph of RHA and CFA shown in figure 2a and

2b respectively. treated RHA had smaller grains that are closely packed together while the CFA has elliptical and rounded grains that are quite large and somewhat loosely packed.

The SEM images of the as-cast aluminium alloys and its composites are shown in figures 3a. In figure 3b, the surface morphology of as-cast Al.6063 alloy at higher magnifications is shown. There is the presence of eutectic silicon which was observed in the microstructure of the as-cast alloys and its composites. The eutectic silicon is characterized by sharp elongated rod-like structures vividly shown in figure 3b. There is nucleation of eutectic Si on the β -(Al, Si) phase in the solute field ahead of the primary aluminium, nucleation of eutectic Al on eutectic Si, and growth of eutectic Al. The alloys and composites contain the silicon phase in the form of large plates with sharp sides and ends [21]. Figures 3 (c-f) showed a well-compacted microstructure with reduced micro-pores and voids due to cold rolling operation earlier conducted on the alloys. However, the lath-like dendrites have been broken by the large rolling deformation and the grains are elongated along the rolling direction for all the samples [22].

The map of grains and the crystallographic orientation distribution of the developed alloys after thresholding are presented in Fig. 4 (a–h) while the grain size distribution plots are shown in Fig. 5 (a–h). Analysis in Fig. 4a showed that the threshold map of the treated RHA contained smaller grains in comparison to those of CFA. The grain characteristics of the reinforcements (RHA and CFA) were deduced from the grain count measurement of the micrographs. An important parameter in Image J analysis called threshold was adopted in determining the segments in between the grains and also the grain boundaries. With this, the grain size distribution was figured out as this function was able to detect the grain boundaries and or interior [23]. The average grain size for the RHA particles was 17.14 μm at 1259 grain counts while the average grain size for the CFA particles was 29.06 μm at 330 grain counts as shown in figures 5.

3.2. Grain size analysis of the developed as-cast Al.6063 alloys and composites.

By using image J grain size analysis tool, it was discovered that the average grain size for the un-cold rolled as-cast Al.6063 alloy was 12.90 μm at 1537 grain counts while that of the cold rolled as-cast Al.6063 alloy was found to be 3.15 μm at 1534 grain counts. This signified the effect of cold rolling operations on grain characteristics as it helped reduce and compact the overall grain size by plastic deformation of the eutectic silicon laths and collapsing of micro voids and solutes segregated at the boundaries in the alloy [24].

The average grain size of the 1.5 wt.% CFA was found to be 9.82 μm at 1205 grain counts while the average grain size of 1.5 wt.% RHA was found to be 8.16 μm at 1913 grain counts which was found to be lesser than that of the uncold-rolled alloy (12.90 μm). The average grain size of 3.0 wt.% CFA was 11.39 μm while the average grain size of 3.0 wt.% RHA was found to be 10.52 μm . In conclusion, the average grain size of the CFA reinforced composites is larger than that of the RHA reinforced composites. This can be related to the fact that CFA particles have larger grain diameters (29.06 μm) than RHA (17.14 μm) particles and this culminates into the overall increase in grain size for CFA composites. In addition, CFA and RHA particles did not plastically deform with the aluminium matrix during cold rolling [25], in this case, it was only the matrix phase that responded to plastic deformation, so the large grain size of the reinforcements (in this case, CFA) will add to the overall grain size of the composite. The XRD characterization of the six specimens showed in figure 6, confirmed the chemical compositions of the alloys and its composites.

It was observed that the content of SiO_2 and Al_2O_3 increased with increasing concentration of RHA and CFA. The 3 wt.% RHA and 3 wt.% CFA showed higher peaks of silica (SiO_2) and alumina (Al_2O_3) than those of 1.5 wt.% RHA and CFA composites. This increasing concentration of hard ceramic particles will definitely have a resultant effect on the mechanical properties of the composites developed.

3.2 Mechanical properties

3.2.1 Hardness

The hardness of the composites improved compared to the as-cast alloys. The significant improvement in the composites can be attributed to the cold rolling treatment of the alloys [26] and its composites as observed in figures 7. Cold rolling is a plastic deformation process that causes permanent strains in the materials which resulted into compaction of the grains under applied stress and consequently led to a reduction in the grain size as shown in figure 5 [27]. It has been discovered that grain size has a relationship with hardness [28]. As the grain sizes reduce, the hardness of the composites increases [29]. Also, during the cold rolling process, large sized pores, micro-voids and segregated solute atoms at the grain boundaries are collapsed and this consequently improved the hardness of the composites [30, 31]. Al.6063 - 3 wt.% RHA composite showed the best hardness property (84 HV) succeeded by 3 wt.% CFA composite (79 HV) while the un-cold rolled Al.6063 alloy had the least hardness (65 HV). Also, thermal mismatch between the high co-efficient of thermal expansion matrix and the low co-efficient of thermal expansion reinforcements (Al_2O_3 and SiO_2) led to improved hardness as this thermal mismatch aided the generation of dislocations [32]. Finally, the improvement in the hardness properties of the composites can be linked to the increasing concentration of RHA and CFA as shown in figures 6 and 7.

3.3 Ultimate tensile strength

The ultimate tensile strength of the developed alloys and composite also followed the progressive trend observed in the hardness properties. The improvement in the mechanical properties can be linked to several factors. First, the SiO_2 and Al_2O_3 present in RHA and CFA act as a formidable barrier to dislocation motions [33]. The SiO_2 and Al_2O_3 particles are by far stronger and have a higher elastic modulus (76 GPa and 195 GPa respectively) than the Al.6063 matrix (68.5 GPa). The particles arrest dislocations whenever they are in contact and because dislocations cannot cut through the particles [34], they tend to loop around the particles under applied stress [35]. This continually happens whenever dislocations encounter a particle. This led to entanglements and multiplication of dislocations under applied stress. The increase in dislocation density caused by looping of dislocations as a result of encountering the hard ceramic particles (SiO_2 and Al_2O_3) led to increased strength in the composites as observed in figure 8 [36, 37]. The higher the content of the reinforcement in the alloy, the higher the strength becomes as shown for all the composites in figure 8 [37]. Al.6063 - 3 wt.% RHA composite showed the best ultimate tensile strength (156 MPa) followed by 3 wt.% CFA composite (151 MPa) while the as-cast/un-cold rolled Al.6063 alloy had the least strength (90 MPa). Secondly, it is suggested that difference in the crystallographic orientation of the matrix (fcc) and the reinforcing hard ceramic particles SiO_2 (tetrahedral) and Al_2O_3 (hexagonal) could have caused an improvement in strength [38]. Also, cold rolling of the composites improved the strength significantly by reducing the grain sizes as shown in figure 8a and 8b. This agrees with hall-petch mechanism that grain size of the alloys and its composites has an inverse relationship with strength [39]. The smaller the size of the grains, the better the strength of the composites. Finally, there is load transfer from the matrix to the reinforcement because the ceramic particles possess high elastic modulus than the parent matrix. This is suggested to enhance the strength of the composites [40]. The low strength in the un-cold rolled alloy, as shown in figure 8, is suggested to be caused by their coarse grains and presence of eutectic silicon in the matrix as shown in figure 3 (a-b) [41]. The presence of eutectic silicon in the materials does not necessarily improve hardness.

The tensile elongation of the composite was observed to decline in a steady manner as observed in figures 9. The decline was observed to be most prominent in the 3.0 wt.% RHA and 3.0 wt.% CFA reinforced composites. This can be linked to the high content of the SiO_2 and Al_2O_3 particles evenly dispersed in the composites [42]. This is also confirmed by the XRD results in figures 6. SiO_2 and Al_2O_3 are hard phase ceramics material that hardens the composites when they are dispersed in the matrix. As they restrict mobility of dislocations effectively, it has a resultant effect on the tensile elongation of the composites causing it to fracture with little yielding preceding it. The CFA also contains SiO_2 particles but not as high in content as those found in RHA. The as-cast/un-cold rolled alloy had the least elongations compared with RHA and CFA reinforced composites shown in figure 9. The cold-rolled as-cast Al.6063 alloy had the best tensile elongation (9.7%). This occurrence is associated with the collapsing of the eutectic silicon phase, micro voids and large pores by cold-rolling process [43]. They serve as discontinuities and sites for fracture when little plastic strain is applied. The breaking of these laths and the absence of hard ceramic SiO_2 and Al_2O_3 reinforcements in the cold rolled alloy, helped improve its tensile elongation above other specimens produced for this research.

Conclusion

The grain characteristics and mechanical behaviour of the as-cast Al.6063 alloy and its composites reinforced with varying amount (1.5 – 3.0 wt. %) of RHA and CFA were investigated. From the results, the following conclusions were drawn:

- The microstructures for the cold rolled RHA and CFA reinforced Al.6063 composites generally showed grain improvement to be significant.
- The contribution of the RHA and CFA particles to the refining of the Al.6063 matrix grains was very minimal. Grain refinement and collapsing of the eutectic silicon phase majorly occurred by cold rolling process.
- The hardness of the as-cast Al.6063 alloy was found to be lower than those of the composites. (65 HV-70 HV) for the as-cast alloy while higher hardness values (75 HV – 84 HV) for its composites. The increase in hardness of the reinforced composite is linked to the grain size reduction by plastic deformation and high content of SiO₂ and Al₂O₃, acting as barriers to dislocation motion.
- The tensile strength of the as-cast alloy and its composites followed the same trend as the hardness. The UTS of the 3 wt.% RHA and CFA composite had the best strength of 156 and 151 MPa respectively. This improvement can be associated with the hard reinforcing phase and strong interface between the matrix and the reinforcing particles which effectively hindered dislocations.
- However, the percentage elongations was improved by cold rolling treatment compared with its as-cast counterpart that was not cold rolled. The increasing content of RHA and CFA abridged the ductility considerably for the respective compositions.
- It can be inferred from this study that cold rolled Al.6063 composite reinforced with 3.0 wt.% CFA had the best balance of mechanical properties which can find use in automobile frames and wheels.

Declarations

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Authors contribution

O.A. Ogunsanya & O.C. Olorunyolemi: Conceptualization, Methodology, Investigation, Formal analysis, Data Curation, Writing – Original Draft, Writing – Review & Editing. A.A. Akinwande: Methodology, Investigation, Formal analysis, Data Curation, Writing – Original Draft, Writing – Review & Editing. O.A. Balogun: Data curation Methodology, Investigation, Formal analysis, Writing – Data curation, Visualization, Review & Editing, M. S. Kumar: Interpretation, Writing – Review & Editing.

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The authors declare no competing interest with any previous work.

Data Availability

All data employed in support to the outcomes in the study are included in this article.

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Figures

Figure 1

shows (a) ball mill (b) CFA particles (c) treated RHA particles (d) as-cast al.6063 and composites (e) tensile and hardness tests specimen. (f) electric air oven

Figure 2

shows SEM image of (a) treated RHA (b) CFA.

Figure 3

shows the sem and edx images of (a, b) as-cast / un-cold rolled Al.6063 alloy and at higher magnifications (c) as-cast / cold-rolled Al.6063 alloy and edx (d) 1.5 wt.% CFA composite and edx (e) 1.5 wt.% RHA composite and edx (f) 3 wt.% CFA composite and edx (g) 3 wt.% RHA composite and edx.

Figure 4

Map drawing of the grains and crystallographic orientation distribution in (a) RHA, (b) CFA, (c) un-cold rolled / as-cast Al.6063 alloy, (d) cold-rolled Al.6063 alloy (e) Al.6063-1.5 wt.% CFA, (f) Al.6063-1.5 wt.% RHA, (g) Al.6063-3.0 wt.% CFA (h) Al.6063-3.0 wt.% RHA.

Figure 5

Grain size distribution in (a) treated RHA (b) CFA (c) un-cold rolled as-cast Al.6063 alloy (d) cold rolled as-cast Al.6063 alloy (e) 1.5 wt.% CFA composite (f) 1.5 wt.% RHA composite (g) 3.0 wt.% CFA composite (h) 3.0 wt.% RHA composite

Figure 6

XRD of the (a) as-cast/un-cold rolled, (b) cold rolled Al.6063-1.5 wt.% RHA, (c) cold rolled Al.6063-1.5 wt.% CFA, (d) cold rolled al.6063 alloy (e) cold rolled Al.6063-3.0 wt.% RHA (f) cold rolled Al.6063-3.0 wt.% CFA.

Figure 7

Hardness of the as-cast alloys and composites

Figure 8

Ultimate tensile strength of the as-cast alloys and composites

Figure 9

Tensile elongation of the as-cast alloys and composites