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MATERIALS ENGINEERING | RESEARCH ARTICLE

Wear characteristics of aluminium matrix composites reinforced with Si-based refractory compounds derived from rice husks

Adeolu Adesoji Adediran^{1,2}*, Kenneth Kanayo Alaneme^{2,3}, Isiaka Oluwole Oladele² and Esther Titilayo Akinlabi⁴

Abstract: This study investigates the wear behaviour of aluminium metal matrix composites reinforced with 10 wt.% Si-based refractory compounds (SBRC) derived from rice husk (RHs). The wear test was conducted using a pin-on-disk tribometer under varying loads with a fixed sliding distance. Scanning electron microscope was used to characterize the worn-out surface and the wear debris. From the results obtained, as the applied load increases, the coefficient of friction (CoF) value reduces to a significant extent. This reduction might be associated with the presence of graphite phase in all the composites developed. The results showed that for samples subjected to 5 N load, T1650 had the optimum value of wear volume amounting to 25–93% increase in wear volume against other samples. Additionally, for 8 N load, K1650 showed a higher response in wear volume having 26–74% improvement in wear volume. The specific wear rate of the composites developed at 5 N load application can be ranked in the following order: T1600 > K1250 > T1650 > K1650 > T1250. A severe agglomeration,

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PUBLIC INTEREST STATEMENT

Incremental research documents are available on the use of synthetic materials in the development of aluminium metal matrix composites. Regrettably, there are a large number of agrowastes materials not fully utilized in Nigeria. The current study is a contribution to our previous report on the development of silicon-based refractory compounds from agro-wastes. The wear behaviour of aluminium metal matrix composites is reported in this study. A conventional universal tribometer was used for this examination in an ambient environment. The results presented showed a good potential application in transport structural parts.





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possibly caused by fragmentation of the clustered debris, dominated the morphology of the worn-out surface. From the EDS spectra, the iron content appears to be low while the oxygen content very high, this is an indication that the tribolayer island was oxidized. An optical photograph showing the wear profile was also taken. It is inferred that the initial wear mechanism of the composites is adhesive, this later converted to abrasive.

Subjects: Mechanical Engineering; Tribology; Manufacturing Engineering; Materials Science

Keywords: morphology; wear; EDS; agglomeration; refractory compounds; metal matrix; composites

1. Introduction

Aluminium-based metal matrix composites (AMCs) are a class of metal matrix composites (MMCs) that are now used in aerospace, structural and automobile industries. Their choice as metal matrix material is informed by the good tribological, physical and mechanical properties which they possess and can sustain under normal service conditions (Alaneme & Sanusi, 2015; D.S. Prasad & Shoba, 2014; K.K. Alaneme et al., 2013). Refractory compounds such as SiC, Al₂O₃, TiC, among others are common reinforcement materials used in the design of AMCs. These reinforcements contribute significantly to the improvement in service performance of Aluminium-based systems, particularly with reference to wear and mechanical properties (Singh et al., 2018; Zhiqiang et al., 2005). However, most of the reinforcement materials are synthesized using processes and technologies such as microwave sintering method (Zhan et al., 2019); chemical vapour deposition (Chen et al., 2019); reactive melt infiltration (Caccia et al., 2018); thermal plasma (Yao et al., 2014). These processes are not readily domesticated in most developing countries. Thus, there is a high reliance on importation to procure these reinforcements at somewhat prohibitive costs of Sibased compounds were because of high foreign exchange to the detriment of the local market (Escalera-Lozano et al., 2008; K. K. Alaneme & Olubambi, 2013). To breach this gap, the use of ashes processed from agro-waste products has been explored as partial replacements for the conventional reinforcements such as silicon carbide and alumina (Alaneme & Sanusi, 2015; Bodunrin et al., 2015). However, hardly could up to 40% replacement with the ashes yield comparable properties with the solely conventional reinforcement-based composites (Alaneme et al., 2018). Recently, research efforts have shown the possibility of synthesizing directly Si-based refractory compounds from carbothermal processing of agro-wastes, notably, rice husks, bamboo leaves and coconut husks (Adediran et al., 2019b; Adediran, Alaneme, Oladele, Akinlabi et al., 2009a, 2018; Alweendo et al., 2019). The mechanical, physical and wear properties of AMCs reinforced with these Si-based refractory compounds are still being pursued to establish its suitability as a complete replacement for the well-known ceramic-based reinforcements used in AMCs. The present study is focused on the wear properties of AMCs reinforced with these Si-based refractory compounds processed under varied treatment conditions. Costa et al. noted that when two mating surfaces are in contact, they experience friction, hence, the formation of debris (Costa et al., 2017). Moreover, the progressive loss of a material on such surface as a result of sliding is known as wear. The assessment of wear properties in AMCs is an essential materials evaluation, because the areas of application of AMCs are such that wear is often encountered in service and operating conditions (brake drums, connecting rods, cylinder liners etc). This has led to a lot of interest in understanding the wear properties and underlying mechanisms of wear damage in AMCs. For instance, Alaneme et al. utilized a 5, 7.5 and 10 wt.% reinforcement of rice husks ash (RHA) and silicon carbide (SiC) in the development of AMCs (Alaneme et al., 2014). They reported that the formation of an abrasive wear surface with high friction coefficient characterized the worn-out surface. Similarly, about 0.92 coefficient of friction (CoF) was reported by (Bembalge & Panigrahi, 2019) for ultrafine grained nanocomposite (UFNC) at a speed of 0.5 m/s in a 30 N load. Fereidouni et al. reported a range of CoF of 0.75-0.80 for a two-stage increasing experiment for

wear assessment (Fereidouni et al., 2018). The effect of sliding velocity and load on the tribological performance of Aluminium-SiC composites was reported by (Kwok & Lim, 1999). From their findings, the wear rate improved when the load and the sliding velocity were increased. In another report, an enhanced value of wear rate was observed when the load, particle size and sliding distance were increased using a statistical model as investigated by (Salin, 2003). Liquid metal-lurgy process was investigated by (Rao et al., 2008) in the development of AMCs. From their work, the velocity was kept constant at a varied load; these gave a higher wear resistance and a corresponding lower coefficient of friction. It was observed by (Ghosh et al., 2012) that the modification of SiC content weight ratio in the production of AMCs among other factors; significantly influenced the wear rate as well as the load. The effect of load (>5 N) on the tribological behaviour of AMCs developed via squeeze casting was reported by (Mindivan et al., 2008). The wear and friction properties were deeply pronounced at higher load application based on their findings.

From the literature survey, hard ceramic particles, particularly carbides materials such as SiC, appear to be a general reinforcement material used for the production of AMCs (Shaikh et al., 2018; S.D. Prasad & Krishna, 2011). Since rice husk is rich in silica, Si-based refractory compound was synthesized from it and used as potential reinforcement material in this study. The interest in studying the wear characteristics of AMCs reinforced with Si-based refractory compounds derived from rice husk is motivated by the quest for the development of Si-based refractory compounds from agro-waste as against the conventional refractory materials. The findings from this work will form a database to the existing literature on aluminium matrix composites developed via conventional refractory compounds derived from rice husk. The effect of parameters such as load, sliding distance and speed on the wear volume, wear rate and coefficient of friction was explored. The morphology of the worn-out surfaces and the wear debris were examined.

2. Materials and method

2.1. Materials

AA 6063 was used as the matrix material for the composites development with chemical composition displayed in Table 1. The chemical constituents of the rice husk (RHs) used to synthesis the Si-based refractory compounds are as shown in Table 2. The rice husks were locally sourced from Igbemo Ekiti in Ekiti State, South West Nigeria; while the AA 6063 from a local vendor. The RHs were thoroughly washed to eliminate dirt and oven-dried at about 55–65°C. The dried husks were later screened to 125 μ m to remove grains of rice from the husks. The husks were then subjected to carbothermal treatment in a controlled atmosphere at varied temperatures as stated by (Adediran, Alaneme, Oladele, Akinlabi et al., 2018). Prior to the carbothermal processing, the powdered sample were placed in a graphite crucible container with lids as stated by (Adediran

Table 1. Elemental composition of Al-Mg-Si alloy		
Element	Wt.%	
Magnesium (Mg)	0.35	
Silicon (Si)	0.59	
Manganese (Mn)	0.35	
Copper (Cu)	0.012	
Zinc (Zn)	0.002	
Titanium (Ti)	0.057	
Iron (Fe)	0.47	
Nickel (Ni)	0.035	
Aluminium (Al)	Balance	

Table 2. Elemental composition of rice husk ash		
Element	Wt.%	
Silica (SiO ₂)	91.81	
Carbon (C)	4.91	
Calcium oxide (CaO)	1.35	
Magnesium oxide (MgO)	0.50	
Potassium oxide (K ₂ O)	0.41	
Hematite (Fe ₂ O ₃)	0.29	
Others	0.73	

et al., 2019c). Tables 3 and 4 show the composition by weight of the selected reaction products of the Si-based refractory compounds (which were used as reinforcement materials).

2.2. Production of Composites

The production of the Al-Mg-Si alloy matrix composites reinforced with Si-based refractory compounds (SBRC) from rice husks was performed using a two-step stir-casting process as reported by (Alaneme & Sanusi, 2015). The process commenced with the computation of the amount of SBRC (<60 μ m particle size), needed to produce 10 wt.% particle reinforced composites. The coding for the composites and initial history of the reinforcement material are as presented in Table 5. Before the casting process, the SBRC was preheated at 200–280°C to eliminate dampness and increase wettability with the molten matrix as stated by (Alaneme & Aluko, 2012). The AA 60,663 alloys was then charged into the crucible furnace and heated to about 710°C ± 30°C to ensure a complete melt. Later, the melt was allowed to cool to a semi-solid state and stirring was done manually for about 10 min. The semi-solid mixture was again heated and stirred mechanically with a stirrer operated at 350 rpm for another 5–10 min before casting into sand moulds with metallic chills around the mould.

2.3. Wear assessment

The wear tests were carried out using a universal tribometer (RTEC 2441) in accordance with ASTM G 133–05 (ASTM G133-05, 2016), Figure 1 shows the representative tribometer used for the current study. The samples for the wear test were machined to 10 mm × 10 mm × 3 mm dimensions and the surface was prepared to mirror-like polished surfaces metallographically. Prior to testing, the steel balls and sample surfaces were smeared with acetone and allowed to dry in air to remove dust and any potential solid contaminants. The wear depth and coefficient of friction (COF) were monitored continuously for 90 s. A reciprocating motion mode of the sliding against the test samples was obtained at a frequency of 5 Hz with a reciprocating velocity of 1.5 mm/s. Other essential parameters used, such as variation in applied load, the stroke length, time of preload, grade and steel ball diameter, are detailed in Table 6. For reproducibility, triplicate tests were conducted for the wear and COF determination. The wear volume was computed for each composite using the single-trace analysis as stated in Equation (1) by (Adegbenjo et al., 2018).

Wear volume
$$(V_w) = L_s \left[R_s^2 \sin^{-1} \left(\frac{w}{2R_s} \right) - \left(\frac{w}{2} \right) (R_s - Z_w) \right]$$

 $+ \frac{\pi}{3} z w^2 (3R_s - Z_w)$ (1)

Where; L_{s} is the stroke length; Z_{w} is the wear depth; R_{s} is the radius of scar; and W is the scar width.

Table 3. Variation in yi Alaneme, Oladele, Akir	eld of selected SBRC pro 1labi et al. , 2018)	cessed at higher tempe	rature interphase used i	or the development of	11650, K1650 and 11600) composites (Adediran,
Designation	3 C-SiC	C	2 H-SiC	6 H-SiC	4 H-SiC	SiC
Т	44.96	28.75	6.28	I	I	11.8
×	9.11	1.8	1.02	48.46	7.84	7.97
T1	0.15	4.53	I	3.13	I	I

T2

47.20

19.45

24.17

Table 4. Variat T1250 compos	ion in yield of se ites (Adediran et	lected refractory al. , 2019c)	materials used	for the productic	on of K1250 and
Designation	Cristobalite	Tridymite	Fe ₂ O ₃	Quartz	Graphite
K1	36.86	3.47	1.67	12.71	45.30

1.63

7.55

Table 5. Sample coding, reinforcement in 10 wt.% and reinforcement history		
Sample coding	reinforcement history	
T1650	SBRC was synthesized at 1650°C without catalyst.	
K1650	SBRC was synthesized at 1600°C in a catalytic environment.	
T1600	SBRC was synthesized at 1600°C without catalyst.	
K1250	SBRC was synthesized at 1250°C with the initial powder unconditioned and without catalyst.	
T1250	SBRC was synthesized at 1250°C with the intial powder conditioned in a catalytic environmnt.	

Figure 1. Pin-on disc tribometer.



Table 6. Operating variables for pin-on-disc test procedure		
Parameters	values	
Nominal force	5, 8, 9.5 N	
Acceleration	0.1 m.s ⁻²	
velocity	1.5 m.s ⁻¹	
Stroke	3 mm	
Temperature	28°C	
Motion	Reciprocating sliding	
Preload	10 s	
Steel ball diameter	3.967 mm	
Grade	24, 440 C	

3. Results and discussion

3.1. Effect of time

The variations in coefficient of friction under selected time intervals and loads are as presented in Figure 2.

It is observed that at about 15 s of wear testing, T1650, K1650, T1600 and T1250 appeared to follow the same trend of decline in CoF under different load applications. The formation of a transfer film during sliding appeared to play a significant role for this duration. This trend was also corroborated by (Natarajan et al., 2006; Pradhan et al., 2016). This trend was however not consistent as the time increases, especially for T1250. Futhermore, sample K1250 showed a slightly different trend in CoF pattern under the same time and load when compared with all the composites developed. It is obvious from the result (Figure 2) that the variations in the CoF values might be due to the distribution of the SBRC in the composites developed. Although (Kumar & Balasubramanian, 2010) also attributed such variations to the presence of porosity during composite production. It is likely also that the reinforcement material played a role in the results discussed above.

3.2. Coefficient of friction (CoF)

Figure 3(a-e) shows the representative variation in the CoF over a test period of 90 s. From this plot, it is evident that as the applied load increases, the CoF value reduces to a significant extent. The increase in load supports eliminating the effect of oxide film which in turn reduces friction.

Figure 2. Shows the representative trend of the effect of variation in selected time interval and load on the coefficient of friction. Figure 3. (a-e). Representative

for composites produced.

variation of coefficient of friction



Additionally, the formation of a transfer film which is brought about during sliding and the transfer film is found to be stable for longer durations and a broad range of normal loads (Natarajan et al., 2006). The reduction observed might be associated with the presence of the graphite phase in all the composites developed. They tend to act as lubricant between the mating surfaces, thus reducing the CoF values. It is likely that the increase in load might have resulted to thermal softening caused by the rise in temperature in the mating part.

The plot representing the average values of the CoF is as presented in Figure 4. From the graph, it is revealed that at load application of 5 N, T1650, K1650, T1600 and T1250 all recorded higher average CoF values. While the pattern appears to be slightly different in K1250, it is likely that the variation in CoF values is attributed to the variation in the compositions of the reinforcement materials. Moreover, the direct contact of the pin-on-disc ball might be on the hard SBRC phase (being the reinforcement) in the composites developed.

3.3. Effect of variation of load on wear volume and wear rate

The plot showing the wear volume and the wear rate is as presented in Figures 5 and 6 respectively.



Figure 4. Average CoF for the composites.

It is revealed that the wear volume increases as the load increases, this is in accordance with Archard theory (Archard, 1953). The resultant effects of friction caused by either the higher contact of mating surface or load-bearing particles are attributive factors that led to the increase in wear volume (Avinash et al., 2016; Deuis et al., 1997; Prabhu et al., 2014). However, it appears that load 8 N was the threshold load for samples T1650, K1650 and T1250 respectively. The increment in load from 5 N to 8 N for these samples, led to an increase in the abrasion of the surfaces of these composites. Additionally (Singh et al., 2018) noted a different report which shows that at the wear surface, an abrasion was likely caused by the groove which results in plastic deformation. It is observed that T1600 and K1250 had nearly similar trend at 9.5 N load. However, they showed a different pattern at other load values. It is evident in T1600 and K1250 that load 5 N was the transitional load after which a significant reduction in the wear volume was obtained.

Figure 6 demonstrates the variation in the specific wear rate of the composites. Composite T1600 displayed the optimum specific wear rate (SWR) obtainable at 5 N load application. However, composites T1650 and K1650 all showed an optimum SWR value at 8 N load. It is noted that T1600 and K1250 had an optimum SWR at 5 N load while T1250 was at 9.5 N load. It is evident that the reinforcements play a key role in the SWR of the composites developed.

composites.



Composites



Composites

Figure 6. Plot showing variation in specific wear rate of the composites.

The findings from (Adediran, Alaneme, Oladele, Akinlabi et al., 2018) revealed that more Sibased refractory compounds are found in K1650. This might have led to the improvement in the composites during abrasion, fretting and sliding. The influence of refractory materials on the wear rate of AMCs was also reported by (Sannino & Rack, 1995). Similarly, an increment in SWR value was evident in T1650 and K1650 respectively, while the least SWR was obtained in T1250 from all the composites developed. A comparison of data showing the summary of wear parameters from literature and the current work is as presented in Table 7. It is noted that the performance levels of the composite in the current work compete favourably with those reported in literature especially for T1650 and K1650 respectively.

3.4. Morphological examination of worn surface and wear debris

The representative scanning electron microscopy (SEM) images showing the worn-out surfaces and wear debris are as presented in Figure 7(a-c) respectively.

It is evident from Figure 7(a) the representative wear track of the composites with the "red" colour showing the load; the whitish arrow indicates the wear debris and the blue arrow show the wear groove. The wear debris was further investigated using a secondary electron mode of SEM as presented in Figure 7(b-d). It is revealed from Figure 7(b,c) that a severe agglomeration, possibly caused by fragmentation of the clustered debris, dominated the morphology. It is evident that the wear mechanism of the composite is adhesive, this later was converted to abrasive, similar trend was corroborated by (K. K. Alaneme & Olubambi, 2013). In addition, from Figure 7(d), the EDS spectra show an appreciable amount of oxygen content as a result of the agalomeration earlier mentioned. The oxygen content can also be traced to the silica content of the reinforcement network as previously observed (K.K. Alaneme et al., 2013). Studies from (Chen et al., 2019; Costa et al., 2017; Jiana et al., 1998; Salvaro et al., 2016) reported that prior to the formation of agglomeration, wear debris undergoes a severe fragmentation and deformation. In the current work, it was evident that the amount of the iron content appears to be low and the oxygen content very high. This is an indication that the tribolayer island was oxidized (Costa et al., 2017). It is concluded that the debris obtained in the current findings falls within the sliding and severe sliding type of debris classification (Fitch, 2013; Hong et al., 2018).

4. Conclusions

The wear characteristics of aluminium matrix composites reinforced with Si-based refractory compounds derived from rice husks were investigated in the current work. The results show that:

- as the applied load increases, the CoF value reduces to a significant extent. This reduction might be associated with the presence of the graphite phase in all the composites developed.
- an increase in wear volume with respect to load condition might be attributed to ease of removal of load-bearing particles and higher contact with mating surface.
- a severe agglomeration, possibly caused by fragmentation of the clustered debris, dominated the morphology of the worn-out surface. From the EDS spectra, the iron content appears to be low and the oxygen content very high. This is an indication that the tribolayer island was oxidized and the presence of silica phase from the reinforcement material.
- the specific wear rate of the composites developed at 5 N load application can be ranked in the following order: T1600 > K1250 > T1650 > K1650 > T1250.

Table 7. Comp	arison of wear d	ata parameters (of existing work	s and current fir	ndings				
Composite	Method of processing	Method of wear test	Load	Period	Sliding distance	Wear volume/ loss	Wear resistance	CoF	Ref
Pure Al + 2–8 wt. % Gr and 10 wt. % ZrO ₂	Sintering	Pin-on-disc	10-40 N	1	400-800 m	Wear loss increases with increase in sliding distance	It increases as the graphite content increases up to 6 wt.% graphite content	1	(2020)
Al 2219 + 8 wt. % B₄C and 3 wt. % Gr	Stir casting	Pin on disc	20-60 N	1	500-2500 m	1	It increases with increase in sliding speed & load	Decreases due to the presence of Gr	(Ravindranath et al., 2017)
LM6 alloy + nano Si ₃ N ₄ and 3.33 wt.% Gr	Gravity die casting	Pin on disc	20-40 N	600 s	1–3 km	Mass loss increase with increase in load	Wear rate increases with increase in load	Increases as load increases	(Ambigal & Prabhu, 2017)
AA 6063 + RHA and Al ₂ O ₃ (10 wt. %)	Stir casting	Pin on disc	25 N	1000 s	1	1	Increases with increase in RHA & Al ₂ O ₃ particles	Increases steadily to 600 s after which a gradual drop was obtained	(K. K. Alaneme & Olubambi, 2013)
LM 13 alloy + ZrSiO4 and SiC (15 wt. %)	Stir casting	Pin on disc	1 kg & 5 kg	1	2800 m	1	Higher at low & high loads. Better wear resistance was attained at 3% ZrSiO4 & 12%SiC	1	(Kumar et al., 2013)
AA 6063 + SiC and RHA (5, 7.5 10 wt. %)	Stir casting	Pin on disc	25 N	1000 s	1	I	Increase in RHA wt.% increases the wear resistance	Lower CoF was recorded by samples with higher RHA	(Alaneme et al., 2014)
AA 6063 + Al ₂ O3, RHA % Gr (10 wt. %)	Stir casting	Pin on disc	25 N	s 006	1	1	Best wear resistance was obtained with the use of Al ₂ O ₃ , RHA % Gr	1	(Alaneme & Sanusi, 2015)
									(Continued)

	oad Period Sliding Wear volume/ Wear CoF Ref distance loss resistance	. 8, 9.5 N 90 s 3 mm It increases Optimum wear Decreases Current work relatively with resistance was significantly as increase in load 0btained at the load 11650 & K1650 increases
	.oad Period	5, 8, 9.5 N 90 s
	hod of Load r test	n disc 5, 8, 9.5
ued)	Method of Met processing wea	Stir casting Pin a
Table 7. (Contine	Composite	AA 6063 + 10 wt. % Si-based refractory compounds derived from rice husks

Figure 7a. (a–d) Shows the wear (a) track and wear debris of the worn-out surface; (e) shows the representative optical micro-scopic image of the wear track at magnification 10×, with the coloured profile indicating the wear depth.



(b)



Figure 7b. (continued).

10 µm

(d)

(c)



Figure 7c. (continued).

(e)



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