



ORIGINAL ARTICLE

Study of tensile properties, fractography and morphology of aluminium (1xxx)/coconut shell micro particle composites



Sefiu Adekunle Bello^{a,*}, Isiaka Ayobi Raheem^b, Nasir Kolawole Raji^c

^a Department of Materials Science and Engineering, College of Engineering and Technology, Kwara State University, Malete, Nigeria

^b Department of Metallurgical and Materials Engineering, Faculty of Engineering, University of Lagos, Lagos, Nigeria

^c Department of Metallurgical Engineering, Federal Polytechnic Yaba, Lagos, Nigeria

Received 6 May 2015; accepted 6 October 2015

Available online 19 October 2015

KEYWORDS

Fractography;
Aluminium (1xxx);
Tensile property;
Enhancement;
Ductility;
Coconut shell

Abstract Aluminium (1xxx)/coconut shell micro particle (Al/CMP) composites have been developed using a compo cast technique. Coconut shells (CSs) were processed with the aid of mortar/pestle and disc grinder and then classified with a set of sieves vibrated with a sine shaker. The CMP additions increased from 2% to 10% at 2% interval. Microstructural/chemical composition analyses were carried out with the aid of scanning electron microscopes (ASPEX 3020) with attached energy dispersive X-ray spectroscopy. Phases were identified using an X-ray diffractometer (XRD). The tensile properties and mode of fracture were studied using Instron extensometer and Avery Denison Impact Testing Machine respectively. Results revealed 99.3% purity of aluminium matrix. The presence of new phases in the aluminium matrix is attributable to chemical interaction between Al and CMPs. The fine grained structure of Al/CMPs composites was confirmed by SEM and optical micrographs. The enhancement in the tensile properties is attributable to the presence of hard phases in the Al matrix and good interfacing bonding between Al matrix and CMP reinforcements. The cone and cup surface appearance with fibrous, dull, dimple and goose grain microstructure of the fracture surfaces of the composites is an indication of ductile fracture. Hence low cost metal matrix aluminium based composites have been developed.

© 2015 The Authors. Production and hosting by Elsevier B.V. on behalf of King Saud University. This is an open access article under the CC BY-NC-ND license (<http://creativecommons.org/licenses/by-nc-nd/4.0/>).

1. Introduction

Metal matrix composites are characterised with high specific mechanical properties such as high strength and ductility per unit volume. Their demands in structural applications for replacement of heavy legacy alloys are now gaining global acceptance. Among non-ferrous metals aluminium attains dominance because of its inherently excellent properties which

* Corresponding author.

Peer review under responsibility of King Saud University.



Production and hosting by Elsevier

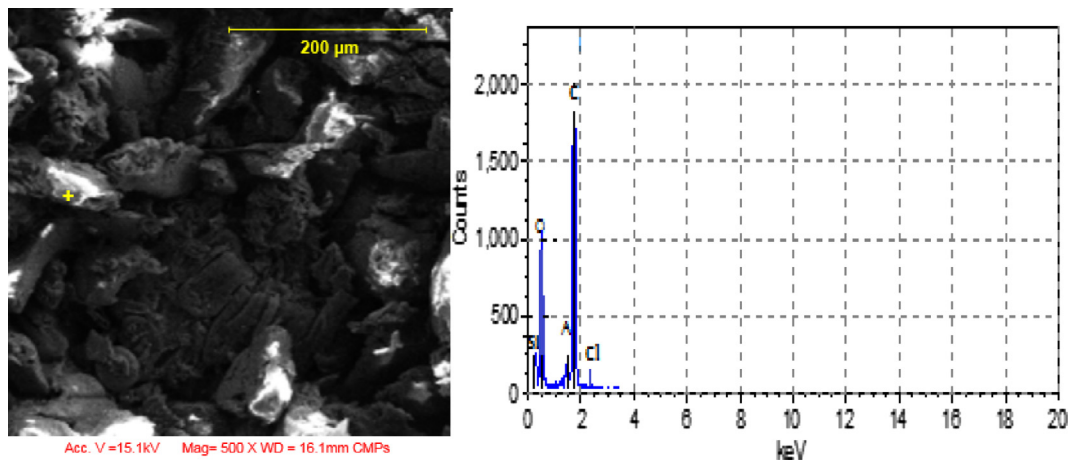


Plate 1 SEM/EDS of the bulk CS.

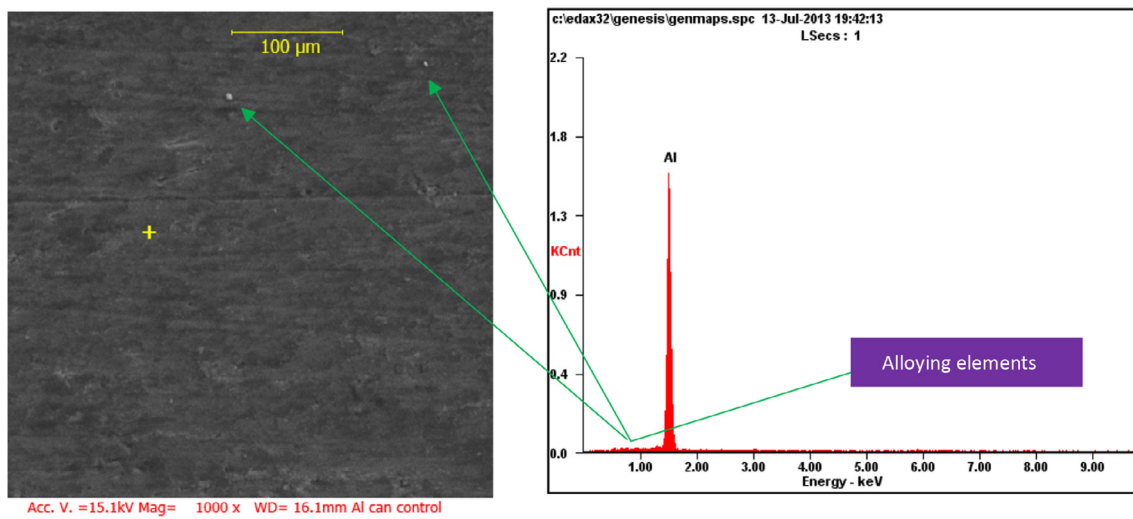


Plate 2 SEM/EDS of the control Al sample.

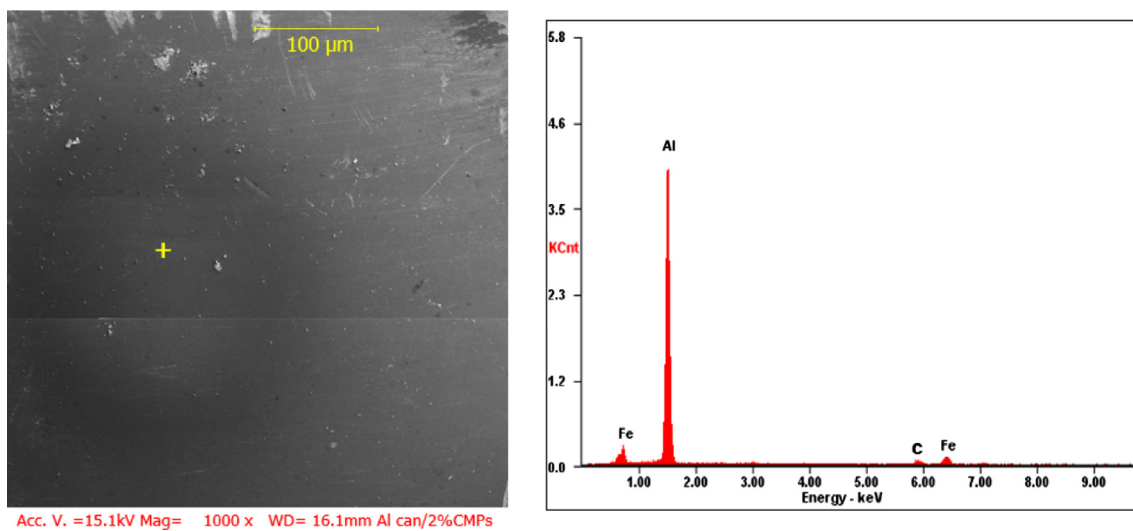


Plate 3 SEM/EDS of the Al/2%CMP composite.

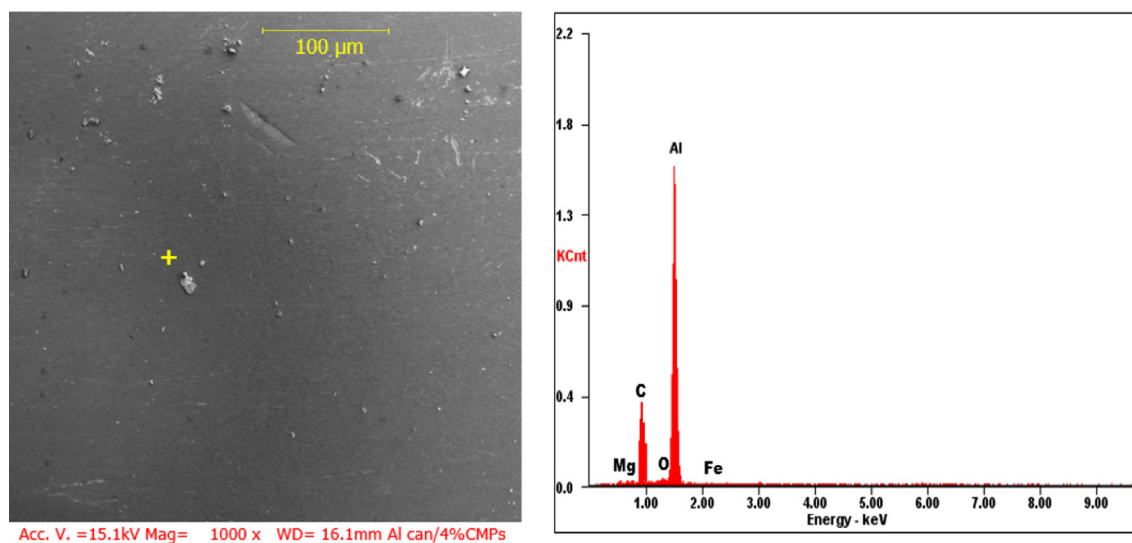


Plate 4 SEM/EDS of the Al/4%CMP composite.

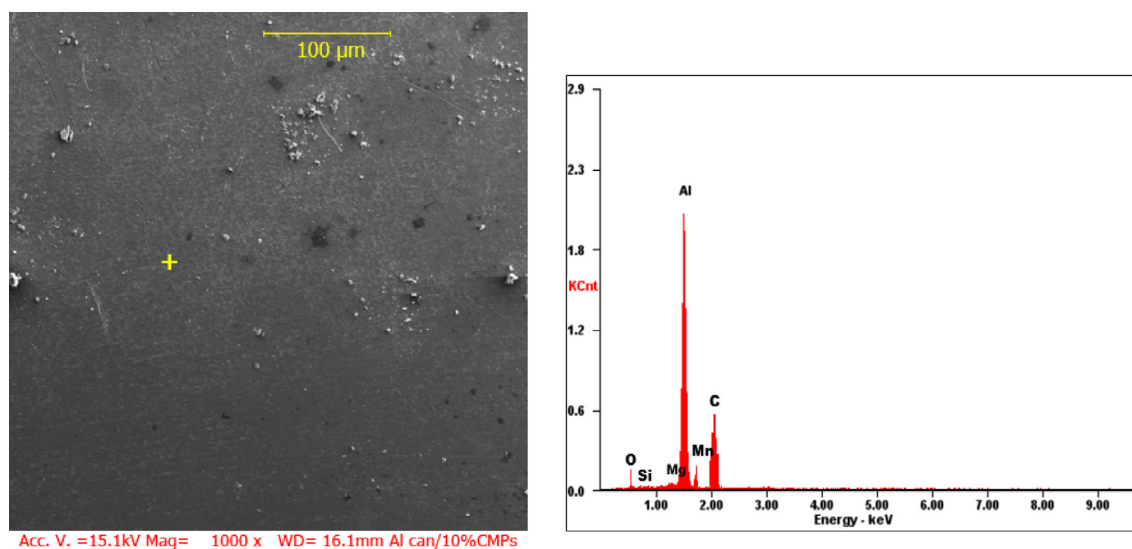


Plate 5 SEM/EDS of Al/10%CMP composite.

include high ductility, ease of formability and resistance to atmospheric corrosion. Aluminium alloys and composites have found use in processing industries as packaging materials; transportation as vehicles' structural parts, automobile engine blocks and in architectural applications (Labberton, 2014; Harwood 2008). Increasing demand of aluminium alloys/composites in various applications has called for research all over the world which focuses on aluminium (Al-Haidary and Jabur Al-Kaaby 2007). The only primary production of aluminium from bauxites is considered as a high energy consuming process. Also, the worse environmental issues associated with the primary production of aluminium places barriers on the process. However, volumes of discarded aluminium products due to their life span expiry/failure in service are on increase, littering our environments. Recycling of discarded aluminium products can serve as an alternative to aluminium production

from bauxite. This recycling technique offers two advantages which include great amount of energy saving and closes the chance of greenhouse gas emissions peculiar to primary aluminium production from its natural ore.

Previous studies revealed the use of aluminium scraps for the green production of aluminium alloys/composites for engineering applications. Results from such studies revealed that the properties of the produced alloy/composites are comparable with those from primary aluminium. Hassan and Aigbodin (2015) used discarded high purity aluminium wire for the production of Al–Cu–Mg/eggshell particulates composites. Their results revealed that the use of carbonised eggshells gave better physical and mechanical properties than uncarbonised eggshell particles. Bello (2014) studied the development and characterisation of aluminium can–coconut shell composites. Results of his findings showed an improvement

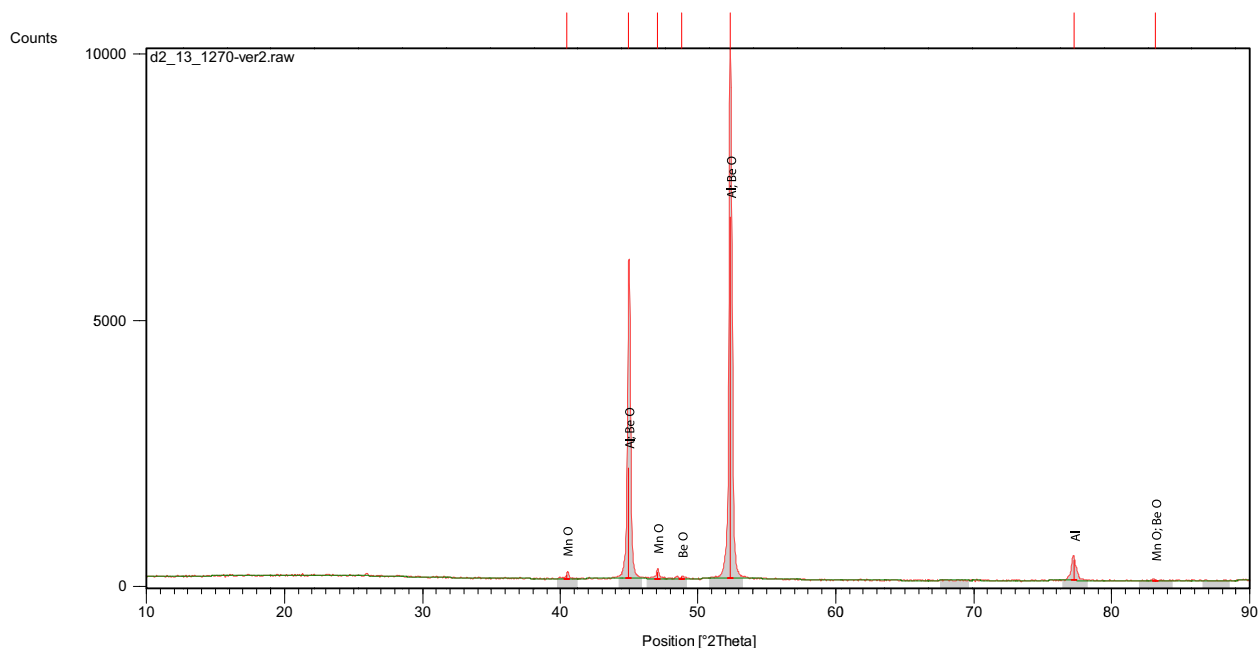


Figure 1 XRD of the control cast of aluminium can sample.

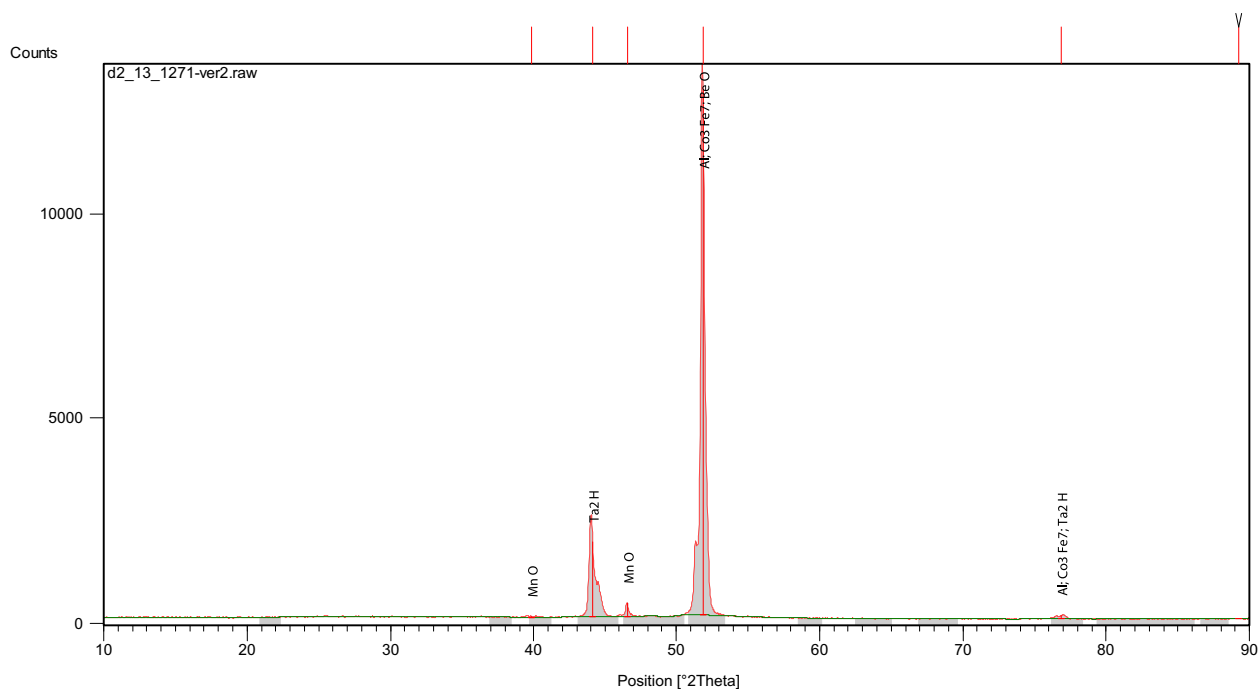


Figure 2 XRD of the Al can/2%CMP composite.

in the wear and mechanical properties of the matrix. [Adewuyi and Omotoyinbo \(2008\)](#) produced aluminium alloys through sand and die casting techniques from aluminium scrap. Their results showed that samples obtained from air cooled dies had better mechanical properties than those produced by sand casting. Also hardness values can be varied by changing the cooling media.

High cost of production of conventional composites due to expensiveness of synthetic fillers has limited their versatile applications ([Bello 2014](#) and [Faye et al. 2009](#)). This has given room for research in the production of low cost reinforcement from agricultural wastes. [Hassan and Aigbodion \(2015\)](#), [Agunsoye et al. \(2014\)](#), [Bello \(2014\)](#), [Ikpambese et al. \(2016\)](#), [Sarki et al. \(2011\)](#), [Hassan et al. \(2013\)](#), [Idris et al. \(2015\)](#),

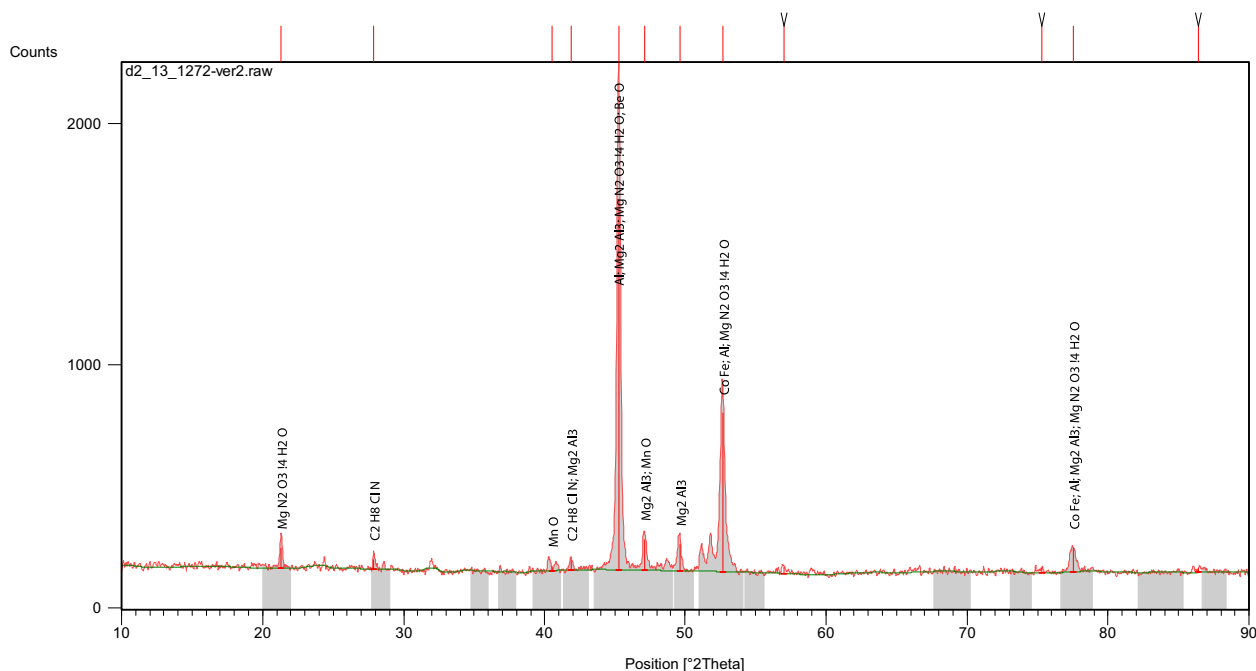


Figure 3 XRD of Al can/10%CMP composite.

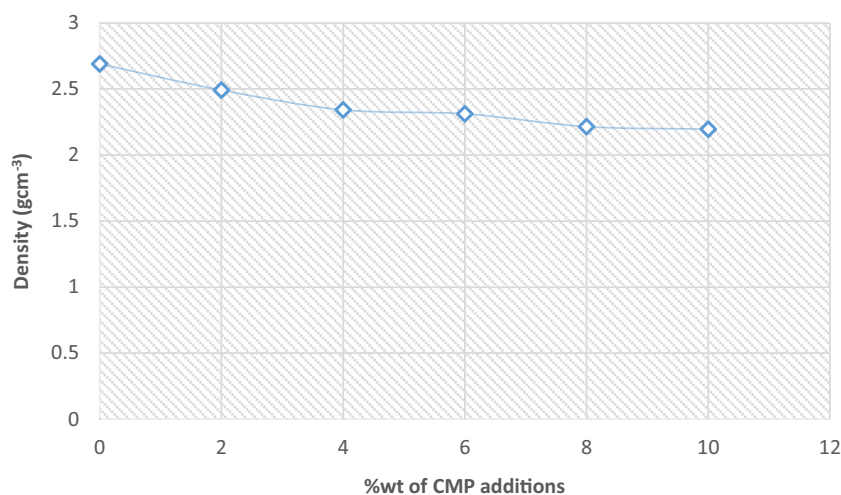


Figure 4 Densities of Al can/CMP composites with %wt of CMPs.

Hassan et al. (2012), Sapuan et al. (2003) and Hassan et al. (2008) have used reinforcements obtained from agricultural wastes for the production of polymer and metal matrix composites. Reports declared the better distribution of the fillers within the matrix and their recovery.

In this present work, metal matrix composites have been produced using aluminium (1xxx) as the matrix and 150 μm sized uncarbonised coconut shell microparticles as reinforcing fillers. This work was aimed at studying effects of %weight (wt) of coconut shell microparticle (CMPs) additions on the tensile properties and morphology of Al/CMP composites. Although a lot of research has been carried out on improvement of

aluminium properties, reinforcement of aluminium (1xxx) with CMPs is very rare.

2. Materials and equipment

Coconut shells (CSs) used in this work were obtained from coconut chip centre, Ibadan, Oyo State Nigeria. Al (1xxx) used was sourced from Al cans packed from event centres, Ibadan Oyo State Nigeria. CSs were sundried for two months after which they were manually broken into smaller pieces using a mortar and pestle. Broken CSs were classified using a 5 mm sized sieve vibrated manually with hands. Those CS pieces

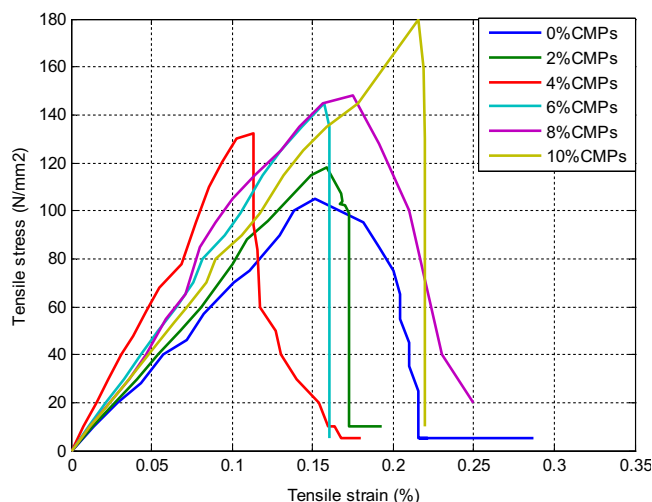


Figure 5 Stress-strain curves of Al-can/CMP composites.

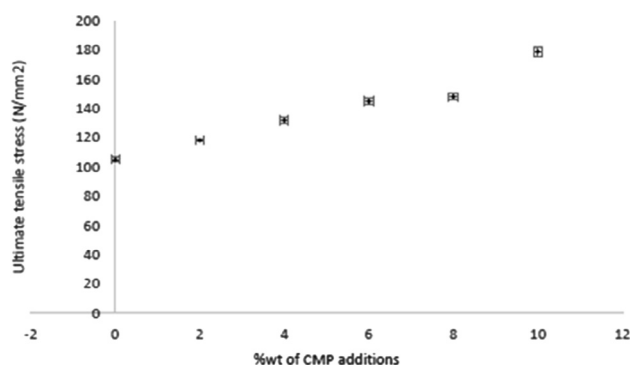


Figure 6 Tensile stress with %wt of CMP additions.

retained below the sieve were pulverised using a two hardened steel disc grinder to obtain CS powder/particles. CS particles were sorted into different mesh sizes ranging from 600 to 75 μm size using a set of sieves placed in descending order of grain fineness number (GFN), vibrated by a sine shaker for 40 min. The CS microparticles (CMPs) that were retained in a 150 μm sized sieve were used as reinforcement in this work. Equipment used in this work includes oil fired crucible furnace, scanning electron microscope with attached energy dispersive

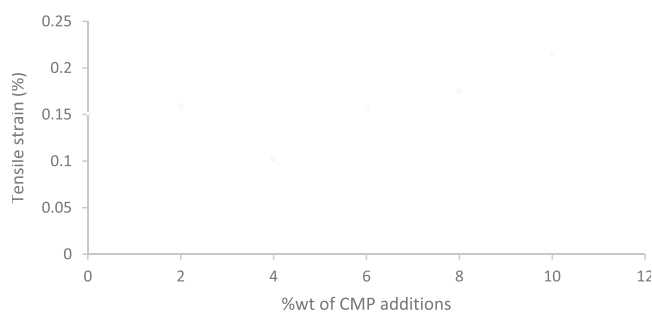


Figure 7 Tensile strain (%) with %wt of CMP addition.

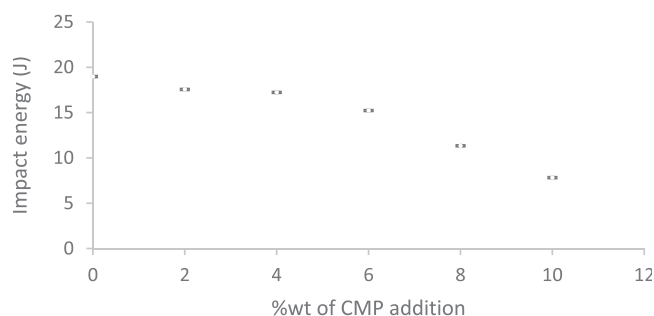


Figure 8 Impact energy with %wt of CMP addition.

X-ray spectroscopy, Instron extensometer, Avery Denison Universal Impact tester, X-ray diffractometer and a cope and drag die cavity mould.

3. Methodology

The composites used in this study were Al-CMP composites containing 2–10% uncarbonised 150 μm CMPs at an interval of 2%. The samples were produced by compo cast technique in accordance with Bello (2014) and Agunsoye et al. (2014). The samples were produced by varying CMP additions to the melt of Al and then stirred to ensure homogenous distribution of CMPs prior to pouring into the preheated metallic mould. A control sample without CMP additions was also produced.

The produced samples were shaped into standard samples for the purpose of tensile property investigation and impact energy determination. The morphology examination and elemental composition analysis of phases present in the produced composite samples were carried out using scanning electron microscope (ASPEX 3020), model SIRIUS50/3.8 with the attached energy dispersive X-ray spectroscopy.

The chemical formulae and compound names of the phases were determined using a Panalytical Empyrean X-ray diffractometer with Pixel detector. The phases were identified using X'Pert High score plus software; PAB-ICSD and ICDD (2014) databases. Samples were placed on a zero background sample holder. The density of 60 $\times$ 10 $\times$ 10 mm of each sample was also measured using direct mass-volume method. A pioneer weighing scale of 210 g capacity and 0.0001 readability by Ohaus Corporation was used for mass measurement.

Tensile samples each of 40 mm gauge length and 5 mm diameter were gripped by jaws of the Instron extensometer, (model: Instron 3369; system ID: 3369S3457). They were subjected to tensile force and this resulted in the gradual stretching of the composite samples at a strain rate of 10^{-3} s^{-1} with the increasing applied stress until the fracture occurred after a considerable amount of plastic deformation. The impact energies of the produced samples were measured with the aid of Avery Denison Universal Impact Testing Machine. Before the test, the pendulum was released to set the scale to zero. The 60 $\times$ 10 $\times$ 10 impact samples of 1 mm notch depth and 0.02 notched radius at an angle of 45° were subjected to a striking energy of 300 J by a pendulum released from the upper position equivalent to charpy impact test. The impact energy absorbed by each sample was noted and recorded. In order

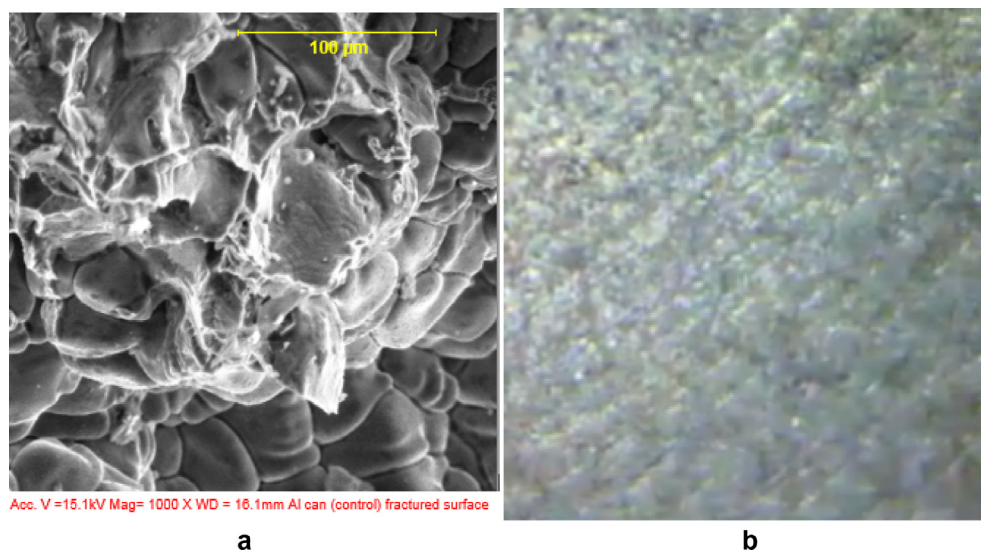


Plate 6 (a) SEM and (b) optical micrographs of the cast of Al can fractured surface.

to study the mode of fracture, the fractured surfaces of the unfilled Al and Al/10CMPs composite were analysed using SEM and Baskar optical microscope (Resolution: 640×840 pixels; Magnification: $10\times$ – $300\times$).

4. Results and discussions

4.1. SEM micrographs/EDS spectrographs

Plate 1 presents the SEM micrograph and EDS spectrograph of the $150\ \mu\text{m}$ sized CMPs. The microstructure reveals the differences in geometry of the particles. Shapes of the CMPs can be classified into dendritic (tree like), prismatic, oval and other polygonal shapes. The EDS elemental analysis declares the presence of C, O, Si, Al and Cl in the CMPs. **Plate 2** presents the SEM micrograph and EDS spectrograph of the unfilled Al sample. The microstructure is very plain. This is evident from

EDS which reveals aluminium with maximum peak. The tiny white particles may represent some alloying elements present in the Al matrix in an amount so small that they cannot have distinct maximum peaks on the EDS spectrograph. Indistinguishable peaks around the Al peak confirm the presence of those elements. The shoulders around the Al peak may be attributable to presence of Al in different forms.

Plates 3–5 show the SEM micrograph/EDS spectrograph of the Al/CMP composites. **Plate 3–5** display very fine microstructures with good interfacial bonding between Al matrix and CMP reinforcements. CMPs are well and evenly distributed and there is no CMP segregation. The microstructures are free from shrinkage cavities. This is an indication of effective stirring of the melt of the composite mixture prior to pouring and rapid solidification/cooling of the composite melt to room temperature. The rapid solidification is attributable to excellent heat conductivity of the mould material which transferred heat quickly from the melt and dissipated it to its

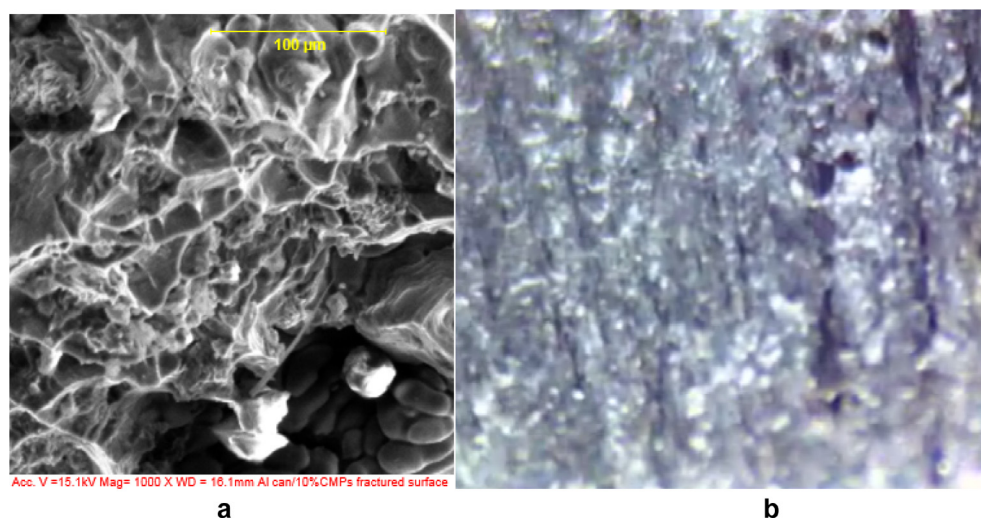


Plate 7 (a) SEM and (b) optical micrographs of Al can/10%CMP composite fractured surface.

immediate environment. The increased packing density is attributable to increment in %wt of CMPs additions. Comparison of EDS spectrographs on Plate 3–5 with that in Plate 2, showed that there were new elements present in the Al matrix. This indicated a chemical reaction between constituting elements in the Al matrix and those in CMPs, leading to formation of new compounds represented by the presence of those new elements on EDS spectrographs.

4.2. XRD diffractograms

XRD diffractograms of the unfilled Al presented in Fig. 1 show that the major diffraction peaks occurred at diffracting angles (2θ) of 44.94° , 52.39° and 52.39° with inter-planar distance of 2.34, 2.03 and 2.03 Å and relative X-ray diffracting intensities of 22, 60 and 62. The respective phases at these peaks are BeO, MnO and Al. This agrees with the EDS spectrograph on Plate 2. Presence of Ta_2H and CO_3Fe_7 in Fig. 2 indicated by peaks at respective diffracting angles of 44.12° and 51.86° with X-ray diffracting intensities 42 and 29 and inter-planar distances of 2.38 and 2.05 Å and Mg_2Al_3 , $\text{C}_2\text{H}_8\text{ClN}$, CoFe and $\text{Mg}_2\text{N}_2\text{O}_3 \cdot 14\text{H}_2\text{O}$ in Fig. 3 indicated by peaks at respective diffracting angles 21.31° , 27.88° , 52.71° and 77.50° with intensities of 9, 3, 57 and 36 and respective inter-planar distances of 4.84, 3.72, 2.02 and 4.84 Å is attributable to chemical reaction between Al matrix and CMP reinforcements in Al/2%CMP and Al/10%CMP composites respectively.

5. Densities

Fig. 4 display the variation in densities of the produced Al/CMPs composites as the %wt of CMPs increased. It is observed from Fig. 4 that the densities decreased as the %wt of CMPs increased. This indicates that CMPs is lighter than Al (1xxx). The density decreased from 2.69 g cm^{-3} of the Al with 0%CMPs addition to 2.196 g cm^{-3} of Al/10%CMP composites equivalent to about 18.36% reduction in density. However, it can be inferred that lighter aluminium based composites can be fabricated with Al (1xxx) and CMPs.

6. Tensile properties

Fig. 5 show the stress–strain curves of the unfilled Al and Al/CMP composites. Different curves in Fig. 5 can be interpreted by Figs. 6 and 7. Fig. 6 presents an increase in ultimate tensile stress (UTS) as the %wt of CMPs addition increased. Fig. 7 described an irregular behaviour with respect to tensile strain as %wt of CMP addition increased. There is a minimum tensile strain at 4%CMP addition. The enhancement in the UTS is attributable not only to the high diffusion rate of CMPs and their deep penetration into the Al matrix, leading to a good interfacial adhesion between the Al matrix and CMPs but also increased packing density of CMPs which were evenly dispersed throughout the Al matrix. Even dispersion of CMPs within the matrix of Al gave room for chemical reaction leading to the formation of 2nd phase compounds such as Ta_2H , CO_3Fe_7 , Mg_2Al_3 , $\text{C}_2\text{H}_8\text{ClN}$, CoFe and $\text{Mg}_2\text{N}_2\text{O}_3 \cdot 14\text{H}_2\text{O}$ (see Figs. 2 and 3) occupying interstices between Al lattice sites. The presence of the newly formed phases caused an elastic straining effect as evident from an increment in the

inter-planar spacing from 2.34 of a phase in the unfilled Al to 2.54 Å of one of the phases in the Al/CMP composites. This straining effect coupled with barriers to dislocation movement contributed to enhancement in the UTS. The barrier/hindrance to dislocation movement became more pronounced as the packing density of the CMPs increased. This led to pile-up of dislocation at the grain boundaries and new compounds' sites such that any further dislocation movement would be induced by additional force application. Hence the composites became more strengthened. Therefore, Al/4%CMP composites at 150 µm size of CMPs can be recommended for application requiring a combination of rigidity and moderate ductility, especially in the car brake padding system and automobile engine block.

7. Impact energy

Fig. 8 presents a decrease in the charpy impact energies (IE) of the Al/CMP composites with an increment in %wt of CMP additions. The reduction in IE is gradual up to 6 %wt of CMPs. Above 6 %wt of CMP addition, there was a drastic reduction in the IE. The decrease in IE may be attributable to extreme hardness of the new phases which imparts brittleness to the Al matrix.

8. Fractography

Plates 6 and 7 show the SEM and optical micrographs of the fractured surfaces of the unfilled Al and Al/10%CMP composite samples respectively. Plates 6 and 7 reveal that there are differences in the morphology and grain sizes of the microstructures. Grains on Plate 7 are much finer than those on Plate 6. Plates 6 and 7 show that both fractured surfaces appeared goose and dimple, fibrous and dull which indicates that the fracture occurred in a ductile manner with a crack initiation. The porosity free-coarse fibrous grain microstructure of the unfilled Al (without CMP addition) inhibited crack growth, leading to absorption of higher impact energy than Al/10%CMP composites. Hence, the addition of CMPs to the Al matrix enhanced the strength and grain refinement but reduced the impact energy. Hence, this indicates a reduction in fracture toughness of the Al/CMP composites.

9. Conclusions

From results of investigation and discussion of this research work, the following inferences can be made:

- [1] Newly light metal matrix composites suitable for use in transportation, construction, architectural design and packaging have been produced from Al (1xxx) sourced from Al cans and CS.
- [2] Incorporation of CMPs in Al (1xxx) matrix led to a grain refinement in the microstructure of the Al/CMP composites.
- [3] There was a proportionate increment in tensile stresses as %wt CMPs increased.
- [4] There was an irregular increase in tensile strains of the Al/CMP composites as the %wt of CMP filling increased.

- [5] Minimum tensile strain observed with Al/CMP composites at 4 %wt of CMPs additions indicates a critical reinforcement level that gave rise to Al/CMP composites characterised by high rigidity.

Acknowledgements

Authors wish to appreciate Mrs Bello Haneefah for her financial support in making this research worthwhile. Also appreciated are Rasheed Abiodun Jimoh and Abdul Wahab Abass Olayinka of Department of Materials Science and Engineering for their assistance in carrying out microstructural analysis.

References

- Adewuyi, B.O., Omotoyinbo, J.A., 2008. Effects of cooling media on the mechanical properties and microstructure of sand and die casting aluminium alloys. *J. Sci. Technol.* 28 (1), 97–102.
- Agunsoye, J.O., Talabi, S.I., Bello, S.A., Awe, I.O., 2014. The effects of *Cocos nucifera* (coconut shell) on the mechanical and tribological properties of recycled waste aluminium can composites. *Tribol. Ind.* 36 (2), 155–162.
- Al-Haidary, J.T., Jabur Al-Kaaby, A.S., 2007. Evaluation study of cast Al–SiCp composite. *Mat. Sci. Pol.* 25 (1), 155–165.
- Bello, S.A., 2014. Development and Characterization of Aluminium Can/Coconut shell Composites. Master of Science Thesis, Department of Metallurgical and Materials Engineering, University of Lagos, Nigeria, pp. 1–82.
- Faye, S., Graham, S., Alan, H., Ajay, K., Robert, Q., 2009. Technology Needs to Support Advanced Composite in the United Kingdom. The Inter-Agency Group Composite, United Kingdom.
- Hassan, S.B., Aigbodion, V.S., 2015. Effects of eggshell on the microstructures and properties of Al–Cu–Mg/eggshell composites. *J. King Saud Univ. -Eng. Sci.* 27, 49–56.
- Hassan, S.B., Oghenewweta, J.E., Nyior, G.B., Aigbodion, V.S., 2013. The effect of vacuum carbonization of waste maize stalk on the thermal resistance of polyester/maize stalk particulate composites. *Plast. Polym. Technol. (PAPT)* 2 (3), 68–74.
- Hassan, S.B., Aigbodion, V.S., Patrick, S.N., 2012. Development of polyester/eggshell particulate composites. *Tribol. Ind.* 34 (4), 217–225.
- Hassan, S.B., Aponbiede, O., Aigbodion, V.S., 2008. Precipitation hardening characteristics of Al–Si–Fe/SiC particulate composites. *J. Alloys Compd.* 466, 268–272.
- Harwood, G., 2008. Aluminium. The Rest of Now, Manifesta7, Bolzano, Italy.
- Idris, U.D., Aigbodion, V.S., Abubakar, I.J., Nwoye, C.I., 2015. Eco-friendly asbestos free brake-pad: Using banana peels. *J. King Saud Univ. -Eng. Sci.* 27 (2), 185–192.
- Ikpambese, K.K., Gundu, D.T., Tuleun, L.T., 2016. Evaluation of palm kernel fibers (PKFs) for production of asbestos-free automotive brake pads. *J. King Saud Univ. -Eng. Sci.* 28 (1), 110–118.
- Labberton, M.G., 2014. Aluminium packaging recycling: the contribution from Romania towards a more resource efficient Europe. In: *Proceeding of European Recycling Society Annual Conference*, second ed. Bucharest.
- Sarki, J., Hassan, S.B., Aigbodion, V.S., Oghenewweta, J.E., 2011. Potential of using coconut shell particle fillers in eco-composites materials. *J. Alloy. Compd.* 506, 2381–2385.
- Sapuan, S.M., Harimi, M., Maleque, M.A., 2003. Mechanical properties of epoxy/coconut shell filler particle composites. *Arabian J. Sci. Eng.* 28 (2B), 171–181.