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ASSESSMENT OF THE DENSITY AND MECHANICAL PROPERTIES OF PARTICULATE PERIWINKLE SHELL-ALUMINIUM 6063 METAL MATRIX COMPOSITE (PPS-ALMMC) PRODUCED BY TWO-STEP CASTING

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Abstract: This work investigated the density, porosity, some mechanical properties and microstructure of PPS-ALMMC and compared the properties of the composites and those of the aluminium 6063 (Al60603) alloy. Periwinkle shells were milled to particle sizes of 75 μ m and 150 μ m and used to produce PPS-ALMMC at 1,5,10 and 15wt% filler loadings using two-step casting technique. The density, porosity, mechanical properties and microstructure of the composite materials were compared with those of the Al6063 alloy. The addition of PPS to aluminium alloy reduced the density of the composite. It was observed that the filler distributes uniformly in the matrix due to the two-step casting technique. The porosities of the composites were within acceptable level of 0-5% except for the composite with 15wt% PPS of 150 μ m particle size. Improved strength, ductility, hardness and modulus were obtained when the filler was used to reinforce the alloy. However, using a filler of higher particle size resulted to higher porosity, reduced tensile strength, ductility and toughness.

Keywords: Composites, periwinkle shell, two-step casting, mechanical properties

INTRODUCTION

Researchers have shown interests in the development of aluminum metal matrix composite (Al-MMCs) because of their potential applications in industries such as aerospace, automotive, thermal management, electrical and electronic as well as sports. Al-MMCs are engineered materials made by incorporating non-metallic reinforcement(s) into aluminium or its alloy so as to tailor the properties such as strength, hardness, stiffness, electrical and thermal conductivity as well as other properties of the material. Al-MMCs offer high strength to weight ratio and high stiffness to weight ratio [1]. In the composite, the good properties of the metal such as light weight, high ductility, electrical and thermal conductivities are combined with the properties of the reinforcement such as low coefficient of thermal expansion, high stiffness, and strength and abrasion resistance to produce material with desired properties. The reinforcement could be in the form of

continuous and discontinuous fibres, whiskers or particulate [2]. The applications of Al-MMCs are limited by high cost and hence the search for cheap agricultural materials as reinforcements to enhance their applications [3]. Particulate Al-MMCs (PAL-MMCs) are less expensive compared to continuous fibre reinforced Al-MMCs (CFRAL-MMCs) and are usually produced by either the solid state (powder metallurgy processing) or liquid state (stir casting, infiltration and *in-situ*) processes [2]. The particulate ceramics materials used to reinforce aluminium are usually carbides, oxides and borides such as SiC, Al₂O₃, TiB, TiC, etc. [4]. The properties of the material are affected by factors such as the type of reinforcement, the method of production, the volume or mass fraction of reinforcement, the particle size of the reinforcement, the shape and distribution of the reinforcement in the matrix. For example, the impact strength and hardness of particulate Al-SiC MMC have been reported to increase

with increasing weight fraction of reinforcement and at 25wt% of the reinforcement; there was over 100% increase in strength and about 90% improvement in the hardness of the composite over those of the pure aluminium [5]. The method of stirring also affected the dispersion of the reinforcement in the matrix [5]. Also, the density, strength and hardness of Al6061-SiC and Al7075-Al₂O₃ were compared at 2, 4 and 6wt% addition of reinforcements and it was reported that the experimental densities of the composites were similar to those of the theoretical densities, but however, the addition of Al₂O₃ into the Al matrix resulted to improved strength, hardness and density slightly higher than the improvement obtained with SiC addition; the addition of harder reinforcements also improved the wear resistance of the composites over that of the unreinforced Al alloy [6]. Low density, low coefficient of thermal expansion, good mechanical strength and hardness, as well as good thermal and electrical conductivities are some of the properties that make Al-MMCs functional electronic packaging and thermal management materials especially for weight sensitive applications over conventional copper tungsten (CuW) and copper molybdenum alloys [1,7]. Some works have recently been reported on the utilization of agricultural wastes as filler for Al-MMCs. Agricultural wastes are cheap compared to carbide, oxide and boride fillers. They constitute environmental problems, hence, the need to find useful applications for them. Rice husk ash (2-3µm) has been used to reinforce Al6061 aluminium alloy and it was reported that the reinforcement distributes uniformly in the matrix and enhanced the tensile strength and hardness with increase in mass fraction of the reinforcement up 8% over the unreinforced alloy [3]. Particulate coconut shell was used to reinforce recycled aluminium cans to improve the tensile strength and wear resistance [8]. The use of rice husk ash (RHA) as the reinforcement for aluminium (AlSi10Mg)-RHA composite was investigated; it was reported that there is filler distribution in the matrix, tensile strength, compressive strength and hardness increased with the increase in weight fraction of the reinforcement and the properties are better at lower particle sizes [9]. Further, the properties of Al-7%Si-Rice Husk Ash and Al-7%Si-Bagasse Ash composites were compared and rice husk ash offered better reinforcing properties than bagasse ash [10]. Also, Fly ash has also been used to produce fly ash reinforced aluminium alloy (Al6061) composites [11].

Periwinkle (*Turritellacommunis*) is a type of edible sea snail which is dark, oval in shape with hard shell. Periwinkles are abundant on rocky shores in hinterlands in the South-South of Nigeria which include Cross-River, Rivers, Akwa-Ibom and Bayelsa. They are

sold in various markets across the country. After consumption, the shells are discarded and add to solid wastes in the metropolis. Some researchers have investigated the use of periwinkle shell as reinforcement for cashew nut shell liquid [12-13], polyester [14-15] and phenolic resin [16-17]. In all these, particulate periwinkle shell was reported to improve the tensile strength, compressive strength, wear resistance and also lowers the density. Higher mechanical properties were achieved with lower particle sizes.

In this work, we evaluated the effect of particle size and weight fraction of particulate periwinkle shell filler on the density and mechanical properties of PPS-AlMMC.

MATERIALS AND METHODS:

Materials

The major materials required for this work are aluminium 6063 alloy (Al6063) and periwinkle shells. The alloy with chemical composition shown in table 1 was purchased from the Nigerian Aluminium Extrusion Limited (NIGALEX), Lagos, Nigeria. Periwinkle shells were sourced from the local market at Otueke, Bayelsa State, Nigeria. The other materials are the consumables for density and metallographic assessments.

Table 1: Composition of the aluminium ingot

Element	Al	Si	Fe	Cu	Mn	Mg	Zn	Cr
Average content	98.18	0.5953	0.4635	0.0117	0.0244	0.3459	<0.002	0.0107
Element	Ni	Ti	Sr	Zr	V	Ca	Be	
Average content	0.0347	0.0566	<0.000	0.0772	0.0114	>0.070	<0.000	

Materials Preparation

» Production of PPS

Periwinkle shells were, washed, boiled in water at 100°C for 40 minutes, allowed to cool, thoroughly washed to remove sand particles and dirt and thereafter dried under the sun for two days and heated in an oven at 110°C for thirty minute to remove all moisture. The shells were crushed with hammer mill, pulverized with a ball mill and sieved to 75µm and 150µm particle sizes at the Mineral Processing Laboratory, Federal University of Technology Akure (FUTA), Nigeria.

» Stir Casting

The facilities at the Foundry Workshop of the FUTA were used for the production of the composite materials. Two-step casting as described by [18] was used to produce the composite materials. The quantity of Al6063 and PPS required to produce composites having 1, 5, 10 and 15 weight percent of the PPS were

calculated. The aluminium ingot was charged into a gas-fired crucible furnace and heated to $730^{\circ}\text{C} \pm 30^{\circ}\text{C}$ which is above the liquidus temperature of the alloy and the liquid was allowed to cool in a furnace to a semi-solid state of temperature about 600°C . The calculated PPS was added at this temperature and the semi-solid mixture was stirred manually with a spindle for five minutes. The composite slurry was re-heated to 730°C and stirred vigorously for five minutes and the molten composite was cast in metallic die. Unreinforced Al6063 was also cast as the control specimen. The compositions of the various composite specimens cast are shown in table 2.

Table 2: Charge compositions of the composite materials

Specimen	Particle size of PPS (μm)	Weight percent of PPS (wt%)	Weight percent of Al6063 matrix (wt%)
1	Nil (control)	00	100
2	75	1	99
3	75	5	95
4	75	10	90
5	75	15	85
6	150	1	99
7	150	5	95
8	150	10	90
9	150	15	85

Characterizations:

» Chemical Analysis of PPS

X-ray Fluoresce Spectrometer was used to determine the elemental composition of the PPS. The system detects elements between sodium (Na, Z=11) and uranium (U, Z =92). PPS is found to contain majorly calcium as shown in table 3.

Table 3: Elemental composition of PPS

Element	Ca	Fe	Si	Mo	Al
Content	70.3350	0.5066	0.0724	0.2372	0.1938
Element	P	S	Sn	Sb	Others Elements
Content	0.2746	0.3987	0.4561	0.4511	27.0745

» Determination of Density of PPS

Since PPS does not dissolve in kerosene, the density of PPS was determined using pycnometer, kerosene and digital weighing balance. Empty pycnometer was weighed and the weight recorded as M_0 . The empty pycnometer was filled with kerosene and weighed (M_1) and the mass of kerosene that fills it, M_{k1} , calculated from equation (1)

$$M_{k1} = M_1 - M_0 \quad (1)$$

The volume of kerosene that fills it was calculated from equation (2), where p_k is the density of kerosene calculated from the ratio of mass to volume.

$$V_{k1} = \frac{m_w}{p_k}, \quad (2)$$

The pycnometer was emptied and dried. A quantity of 75 μm PPS was added to the empty pycnometer and the weight of the pycnometer together with the PPS

measured as M_2 . The weight of the PPS, M_{pps} , added is calculated from equation (3).

$$M_{pps} = M_2 - M_0 \quad (3)$$

Kerosene was added and the weight of the whole content, M_c , taken to determine the weight of kerosene added as M_{k2} ;

$$M_{k2} = M_c - M_2 \quad (4)$$

The volume of kerosene (V_{k2}) added is determined from equation (5)

$$V_{k2} = \frac{M_{k2}}{p_k} \quad (5)$$

The volume of the PPS (V_{pps}) put in the pycnometer was calculated from equation (6)

$$V_{pps} = V_{k1} - V_{k2} \quad (6)$$

$$\text{Density of PPS} = \frac{M_{pps}}{V_{pps}} \quad (7)$$

» Determination of Density and Porosity of Composite Materials

The basic method of calculating density is by dividing mass by volume. In this work, experimental density of each specimen was determined by Archimedes' principle. The theoretical densities of the composites were calculated from the rule of mixture as shown in equation (8). The weight percentages of PPS in the composites were converted to volume fractions using the density of PPS calculated in equation (7) and the density of the alloy to convert mass of alloy to volume so as to accurately calculate the theoretical density. The difference between the theoretical and experimental density of each composite specimen was used to estimate porosity using equation (9) [19].

$$p(\text{PPS-AlMMC}) = p(\text{Al6063}) \times V_f(\text{Al6063}) + p(\text{PPS}) \times V_f(\text{PPS}) \quad (8)$$

where: $p(\text{PPS-AlMMC})$ = Theoretical density of composites, $p(\text{Al6063})$ = Density of Al6063 alloy, $V_f(\text{Al6063})$ = Volume fraction of Al6063 alloy, $p(\text{PPS})$ = Density of PPS and $V_f(\text{PPS})$ = Volume fraction of PPS.

$$\text{Porosity} = \frac{\text{Theoretical density} - \text{Experimental density}}{\text{Theoretical density}} \quad (9)$$

» Tensile Testing

Uniaxial tensile test was performed on each specimen at room temperature using Instron Universal Testing Machine at a cross-head speed of 10mm/s. The tensile specimens were machined and tested in accordance with ASTM E8M-91 [20] with the gauge length of 40mm and gauge diameter of 5 mm.

For each specimen, three repeated tests were carried out to guarantee reliability. The tensile properties reported are tensile strength, modulus of elasticity, percentage elongation and energy at break.

» Hardness Testing

The hardness of the aluminium alloy and composites was determined with Vicker Hardness Tester (*LECO AT 700 Microhardness Tester*). The dimension of each specimen for hardness testing is 25x20mm and each specimen was grinded and polished to obtain a flat

smooth surface. During the testing, a load of 980.7mN for 10s on the specimen through square based pyramid indenter and the hardness readings taken in a standard manner. The readings were taken in three different points at the surface of the hardness specimen and the average computed as the hardness of the specimen.

» **Metallography**

Software driven optical metallurgical microscope was used to study the microstructure of the alloy as well as the composites. Prior to viewing of specimens with optical microscope, emery papers of grit sizes ranging from 500µm-1500µm were used to polish the surfaces of the specimens.

Thereafter, fine polishing was performed using a suspension of polycrystalline diamond of particle sizes ranging from 10µm - 0.5µm with ethanol solvent. Each specimen was etched with 1HNO₃: 1HCl solution [18] prior to viewing with the optical microscope for microstructural study.

RESULTS AND DISCUSSION

» **Density and Porosity**

The particle density of PPS determined was 2.3g/cm³ while the density of the Al6063 alloy was 2.68g/cm³. The densities and porosities of the composites are shown in *tables 4 and 5*.

Table 4: Densities and percentage porosities of PPS-AlMMC with 75µm particulate PPS

Specimen	Theoretical density (g/cm ³)	Experimental density (g/cm ³)	Porosity (%)
2	2.676	2.61	2.5
3	2.657	2.56	3.7
4	2.638	2.52	4.5
5	2.615	2.50	4.4

Table 5: Densities and percentage porosities of PPS-AlMMC with 150µm particulate PPS

Specimen	Theoretical density (g/cm ³)	Experimental density (g/cm ³)	Porosity (%)
6	2.676	2.55	4.8
7	2.657	2.53	4.8
8	2.638	2.51	4.9
9	2.615	2.41	7.8

From the results shown in *tables 3 and 4*, the composites have lower densities than the alloy. The density decreases gradually as the weight fractions of the filler increase. The decrease in density is more significant in composites with 150µm PPS filler compared to those with 75µm PPS filler.

The decreased density with addition of the particulate periwinkle filler is a result of the lower density of the filler compared to the matrix. This is a positive development because it will further maximize the utilization of PPS-AlMMC where lighter weight is desired and properties such as specific strength and stiffness will be higher in the composite compared to the aluminium alloy.

Porosity or void in the composite material accounts for the difference between the theoretical and experimental densities of the composite materials. It is observed that porosity increases with increase in filler content and higher in composites with PPS filler of higher particle size. Porosity is as a result of trapped air or poor wettability of the reinforcement. High porosity results to low strength and other mechanical properties. Generally, the porosities of the composite materials are within acceptable range for the entire specimen due to the two-step casting method adopted as earlier reported [18]; except in specimen 9 due to poor wettability of PPS with larger particle size at higher weight fraction in the matrix. More research will be carried out in order areas so as to further improve matrix-filler wettability in the composite.

» **Microstructure**

The optical micrographs of the Al6063 alloy and those of the composites are shown in *figures 1-9*.

It is observed that PPS disperses in Al6063 alloy. There is homogeneity of the microstructure. Also, the coarse grains of the alloy were refined to finer grains by the introduction of PPS.

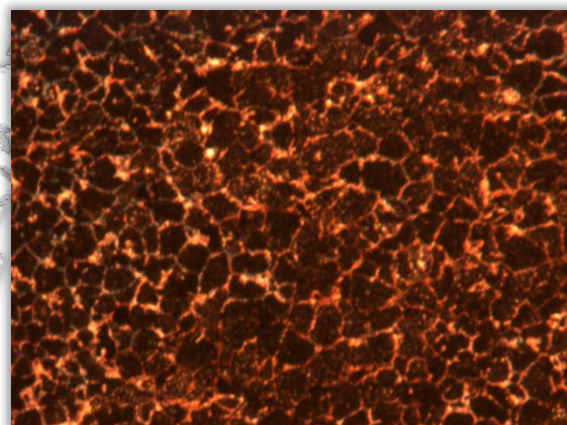


Figure 1: Optical micrograph of specimen 1 (x50)

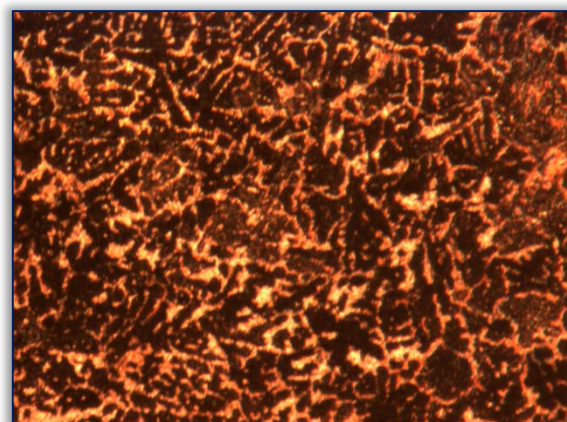


Figure 2: Optical micrograph of specimen 2 (x50)

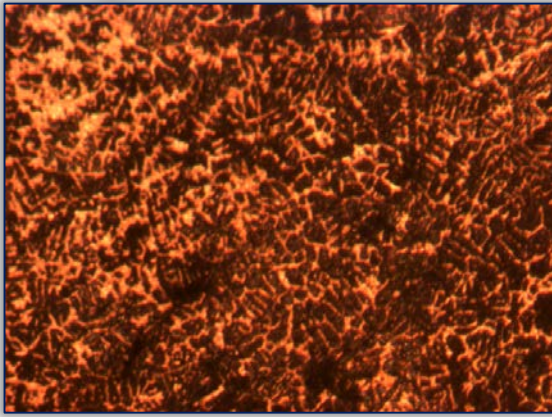


Figure 3: Optical micrograph of specimen 3 (x50)

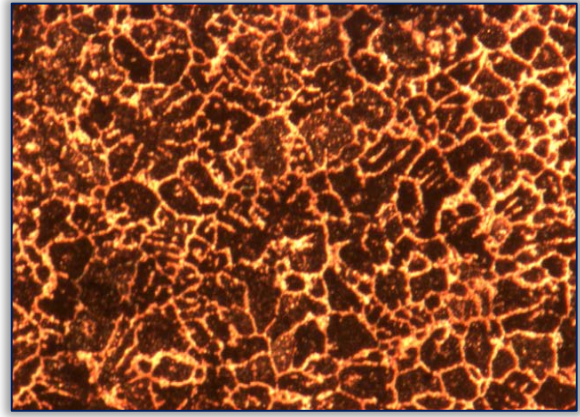


Figure 7: Optical micrograph of specimen 7 (x50)

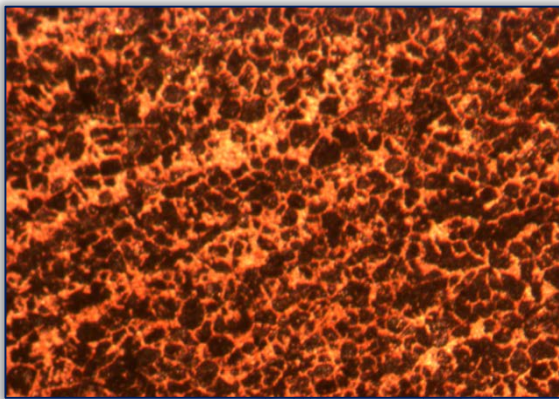


Figure 4: Optical micrograph of specimen 4 (x50)

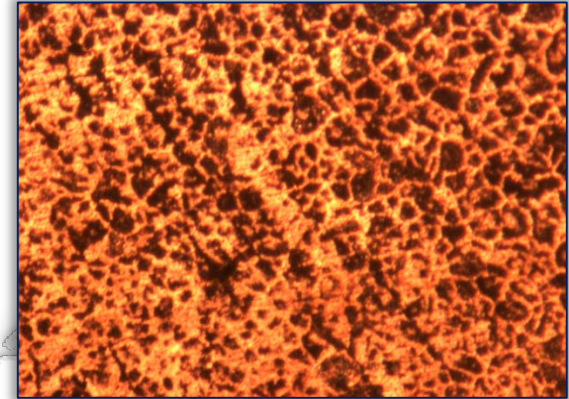


Figure 8: Optical micrograph of specimen 8 (x50)

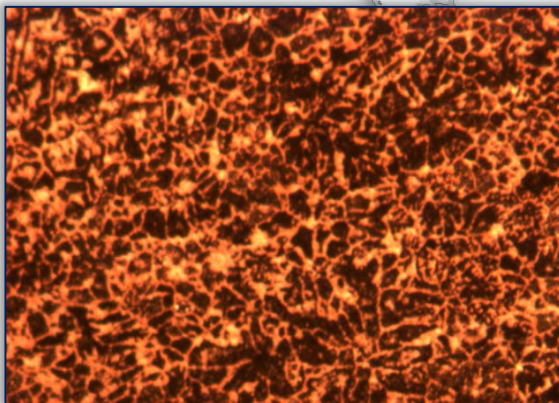


Figure 5: Optical micrograph of specimen 5 (x50)

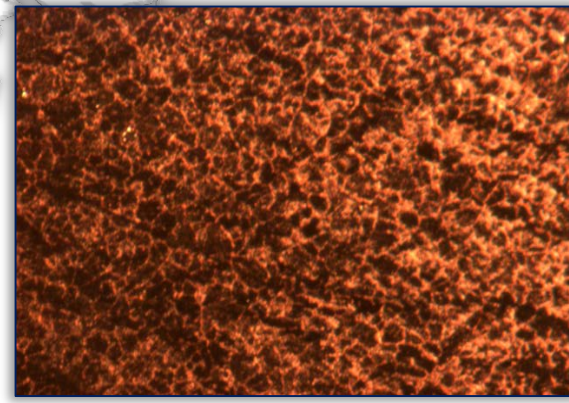


Figure 9: Optical micrograph of specimen 9 (x50)

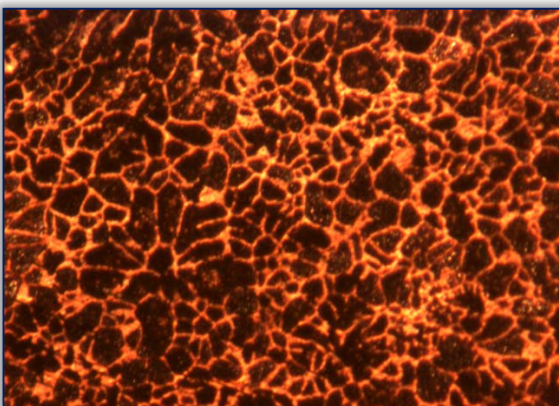


Figure 6: Optical micrograph of specimen 6 (x50)

» Tensile Properties

The tensile strength, elastic modulus, percentage elongation of the alloy and PPS-AlMMC are shown in figures 10-14. The composites possess higher elastic moduli than the alloy. At low particle size, there is improvement in strength up to 10wt% of PPS in the alloy while the strength depreciated above 5wt% PPS in the alloy when the particle size is 150 μ m. The introduction of PPS in Al6063 alloy is observed to improve strength, modulus and ductility at 75 μ m particle size of the filler.

The PPS has strengthening capacity and increases the capacity of the composite to carry load. Also, due to its ability to refine the grains, improved ductility depicted

by the higher percentage elongation, strength and toughness are observed in the composites over those of the alloy in line with Hall Petch Equation. However, at higher particle size of the filler, due to low surface area and poor wettability, the porosity of the composite increases, which give rise to lower strength at high percentage of the filler in the matrix.

» **Hardness**

The hardness of Al6063 alloy and PPS-AIMMC are shown in figure 14. In the composite with 75µm PPS filler, there is a decrease in hardness at 1wt% filler addition followed by continuous increase in hardness at 5, 10 and 15 wt% filler.

However, at 150µm PPS particle size, maximum hardness was achieved at 1wt filler loading followed by 5wt 5 filler content and thereafter the hardness becomes lower compared to the alloy.

PPS has the potential to improve the hardness at low wt% with higher particle size filler while with lower particle size filler, improved hardness is achieved at higher wt% filler loading.

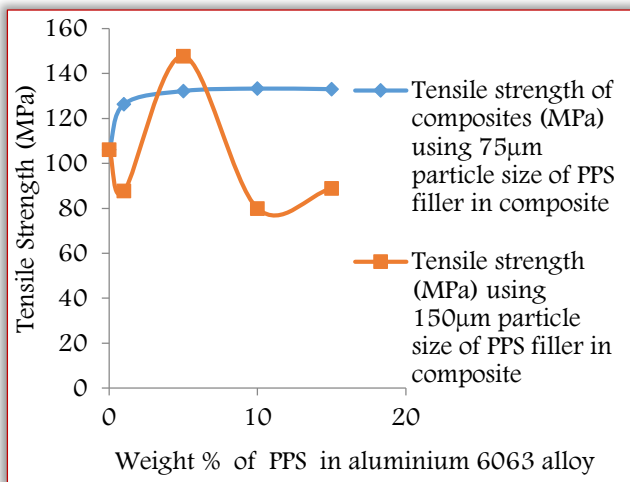


Figure 10: Tensile strengths of Al60603 alloy and PPS-AIMMCs

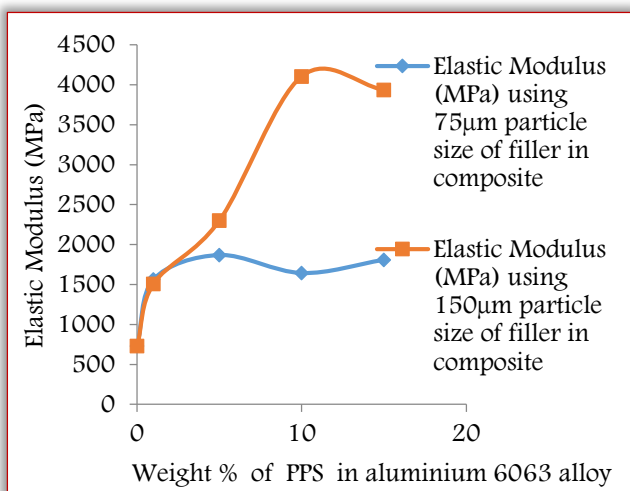


Figure 11: Elastic moduli of Al60603 alloy and PPS-AIMMCs

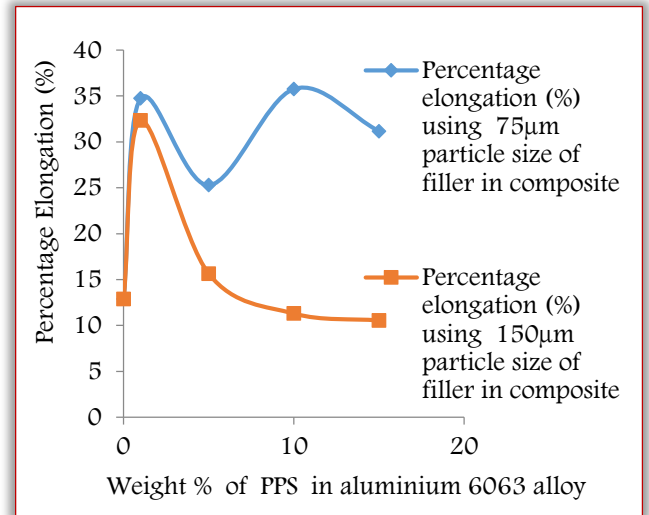


Figure 12: Percentage elongations of Al60603 alloy and PPS-AIMMCs

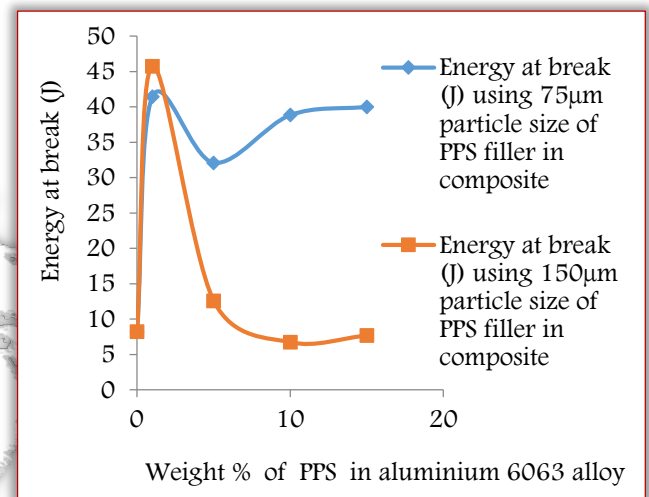


Figure 13: Energies at break of Al60603 alloy and PPS-AIMMCs

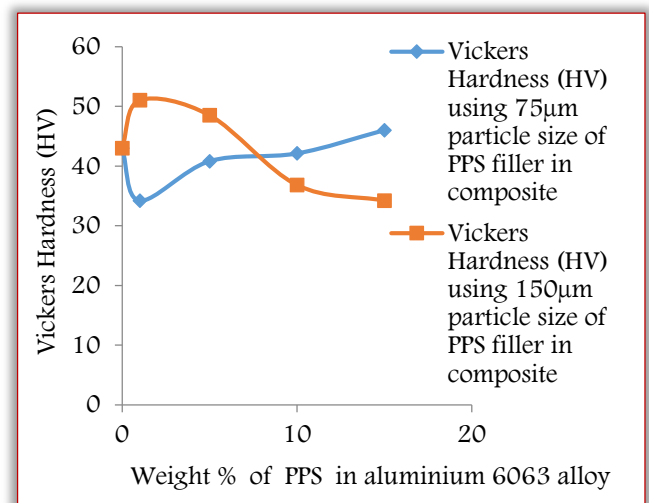


Figure 14: Vickers hardness of Al60603 and PPS-AIMMC composites

CONCLUSIONS

From the results, the following can be concluded:

- ≡ PPS-AlMMC has lower density than Al6063 alloy. The porosity of the composite is low due to filler matrix compatibility and two-step casting process. At higher particle size and filler content, the compatibility of the matrix and filler reduces and porosity increase resulting to lower strength and hardness.
- ≡ PPS distributes uniformly on Al6063 alloy and refines the grains from coarse grains to fine grains.
- ≡ Due to the ability of PPS to refine the grains of Al6063 alloy, the addition of PPS in the alloy improves the strength, elastic modulus, ductility and hardness of the composites. The composites are cheaper than aluminum matrix reinforced with carbide, oxide and boride fillers. The composites can be used in areas where lighter weight and higher strength are required within the aerospace, automotive and electronic industries such as cylinder liners in engines, aluminum calipers and power electronic modules.

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ISSN:2067-3809

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