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Effect of isothermal heat treatment on the hardness and microstructural study of aluminium/periwinkle shell ash bio composite

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Abstract
Effect of isothermal heat treatment on the hardness and microstructural study of aluminium/periwinkle shell ash bio composite produced by double stir casting method was studied. The samples were heat treated at 540°C for one hour and immediately quenched in warm water (36.5°C) before artificially aged to 180°C for two hours. The hardness of the composite was determined using Rockwell-B scale universal hardness tester, the chemical composition of the Periwinkle Shell Ash (PSA) was studied using X-Ray Fluorescence (XRF) machine, the thermal properties/stability of the composites was studied via Dispersion Scanning Calorimetry (DSC) using a TDG-DSC analyzer and microstructures of the composites were studied via Scanning Electron Microscopy (SEM) equipped with an Energy Dispersion Spectroscopy (EDS) system. From the microstructure (SEM/EDS) result obtained, the PSA in samples B, C & D were found to be homogeneously distributed in the Aluminium Metal Matrix and showed an endothermic DSC curve (i.e. heat absorbed by the composite) which indicated that more energy will be required to cause a dislocation in the metal matrix that led to the failure of materials, while sample E showed least distribution of the periwinkle shell ash in the Aluminium metal matrix which is due to exothermic DSC curve (i.e. heat loss) and indicates that less energy would be required to cause a dislocation movement in the metal matrix of the material. The Hardness value of the composite varies from 7.0 to 7.4 as compared with the 5.0 value of pure Aluminium. It can be deduced that the addition of Periwinkle Shell Ash as a reinforcement to the Aluminium metal matrix has improved the hardness under the studied condition.

Keywords: hardness, microstructure, reinforcement, temperature

Introduction

Aluminum is a silvery white metal, soft, nonmagnetic, ductile and the third most abundant element in the Earth's crust (after oxygen and silicon). Aluminum is remarkable for its low density and ability to resist corrosion. Its alloys are vital in transportation industries and in structures such as building facades and window frames [1-4]. In engineering applications, aluminum cannot be used in its pure form hence the need for reinforcing it to achieve a better use and application. In recent times the use of composites has been of great importance because the addition of high strength fibers to a polymer matrix can greatly improve mechanical properties such as ultimate tensile strength, flexural modulus, and temperature resistance [5-8].

The matrix is the monolithic material into which the reinforcement is embedded. Periwinkle shell is a waste product gotten from the consumption of small marine snail (periwinkle) which is housed in a v-shaped spiral shell and is found in many coastal communities in Nigeria. There are however, large amount of these shells being disposed of as waste thus constituting environmental problems in places where use cannot be found for them [9-11]. Generally, different processing techniques can be used to produce metal matrix composites these include powder metallurgy, stir casting, infiltration casting, direct melt oxidation, hot-dipping and sintering of ball- mill activated mixture of powders etc. [12-16]. The target for development of Aluminum Metal Matrix Composites (AMCs) is optimizing cost reduction and performance level by considering the use of waste (periwinkle shell) as reinforcing materials. This is due to the relative high cost of commonly used synthetic reinforcements such as SiC and Alumina [15-17].

In recent times, researchers in the development of Al based composites using synthetic reinforcements such as silicon carbide (SiC) etc. have shown that the use of synthetic reinforcement is relatively high, and most of the agro waste materials in the world are burnt or buried underground because of the hazardous effect they pose to the environment [18-22]. However, utilization of periwinkle shell as a particulate composite material will help not only in making the environment friendly in areas where this shells are mostly discarded, but also can be used as a reinforcement in the production of aluminium composites.

Materials and Method

The materials used in this study includes: high purity Aluminum (Al) wire scraps obtained from northern cable company (NOCACO) Kakuri, Kaduna -Nigeria, Periwinkle shell, charcoal, distilled water and silica sand. Some of the equipment employed in this study included: Charcoal fired crucible furnace, stirring rod, split metal mold, slag scooper, pair of tongs, 250µm sieve aperture, SX-5-12 Box resistance furnace, lathe machine, thermocouple, TGA-DSC analyzer and TM 3030 Tabletop microscope (SEM), Rockwell hardness tester. The necessary methods followed are discussed below:

Periwinkle Shell Ash (PSA) Production

The periwinkle shell was washed and dried in the open for two days, it was then poured into a container and placed on the charcoal fired crucible furnace for heating to take place. The burnt periwinkle shell was allowed to cool before grinding and the ash produced was sieved to -250µm.

Composite Production

Double stir casting method was employed in the production of the composite. Charge calculation was carried out and used to determine the various masses of aluminium and the periwinkle ash needed to cast a rod of 25 mm diameter and 30 mm long of required composition as shown in Table 1.

Table 1: Different Composition of the Composite produced

Sample	% composition of Al	% composition of PSA
A_1	95	5
A_2	90	10
A_3	85	15
A_4	80	20
A_5	75	25

400g of Aluminum each was measured using a spring balance and placed in five containers. The ash for each batch was then measured using a digital weighing beam and poured into small containers for each batch and preheat in the box resistance furnace to 450°C in order to increase the wettability of the ash to have a proper adhesion of the ash and the molten Aluminum. After 15 mins the charcoal fired crucible furnace was fired and the measured Al for the first batch was charged into the crucible furnace and heated to a temperature above the melting point of Aluminum (>700°C) to ensure the wire melts completely. The ash for batch one was then remove from the box resistance furnace and charged into the molten Aluminum in the crucible furnace, it was stirred using a stirring rod and allowed for 5 mins in the furnace. After which it was stirred again before pouring into the prepared sand mold. This procedure was carried out for the remaining compositions and five bars of size 25x300*mm* was produced for each composition.

Heat Treatment of Al/PSA composite

A total of thirty-five (35) samples were machined for DSC, SEM and Hardness. The solution heat treatment performed on the samples involved heating to 540°C and holding at that temperature for one hour in the electric arc furnace. The samples were then quenched in warm water of 36.5°C. The quenched samples were given an artificial aging at 180°C for two hours as described in Fig.1 below.

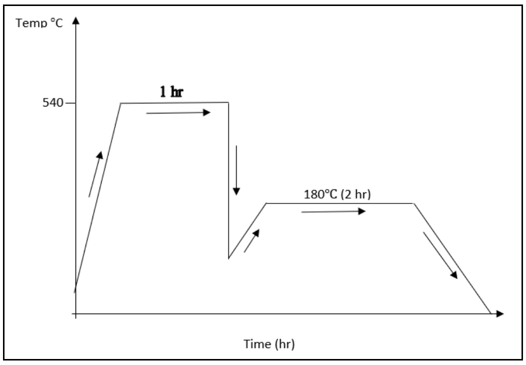

Fig 1: Schematic diagram of the STAT heat treatment cycle indicating solution heat treatment and artificial aging.

Thermal Analysis

Thermal examination of the various composition of the produced composite was carried out via Deferential Scanning Calorimetric (DSC) using the TDG-DSC analyzer. A sample of mass between 3 g and 8 g was required for the DSC examination for each composition, so a file was used to obtain the required amount and a Mettler PM 400 electronic balance was used to determine the mass of the filed sample. The sample is placed in a suitable pan and sits upon a constantan disc on a platform in the DSC cell with a chromel wafer immediately underneath. A chromel-alumel thermocouple under the constantan disc measures the sample temperature. An empty reference pan sits on a symmetric platform with its own underlying chromel wafer and chromel-alumel thermocouple. The start button was clicked and Heat flow for a temperature range between 0ºC to 500ºC was measured by comparing the difference in temperature across the sample and the reference chromel wafers. A graph of heat flow against temperature was plotted. The procedure was repeated for the remaining batches and their various graphs obtained.

Microstructural Analysis

Five (5) samples of 5 mm, one from each batch was cut and the Microstructural observation of the samples were studied using a Scanning electronic microscope equipped with an energy dispersive spectroscopy (EDS) system at Chemical Engineering Department Ahmadu Bello University, Zaria-Nigeria. The samples were firmly held on the sample holder using a double-sided carbon tape before putting them inside the sample chamber. The SEM was operated at an accelerating voltage of 15 kv and the image was recorded.

Rockwell hardness test

The hardness characteristic of the samples was measured using a Rockwell hardness tester with a minor load of 10 kgf and a total load of 60 kgf. The (scale F: HRF) was used in order to obtain a reliable average hardness reemploying 1/16" (0.0159 m) ball indenter with 100 kg load in accordance with the report elsewhere [23].

Results and Discussion

From the XRF analysis of the periwinkle shell ash shown in Table 2, elements like SiO_2, Al_2O_3, P_2O_3, K_2O and CaO were found to be major constituents of the ash. Silicon dioxide, iron oxides, alumina and titanium oxide are known to be among the hardest substances. Some other oxides viz. Na_2O, ZnO, BaO was also found to be present.

Table 2: XRF analysis of the Periwinkle shell ash

Constituents	Amount Present (wt %)
SiO_2	-
Al_2O_3	0.5000
Fe_2O_3	2.0600
CaO	46.7300
MgO	-
SO_3	1.1000
K_2O	-
P_2O_5	-
TiO_2	0.0760
V_2O_5	0.0100
Cr_3O_5	0.0100
CuO	0.929
ZnO	0.3000
BaO	0.1200
Y_2O_3	0.7800
H_2O^+	44.7400

The presence of hard compounds like SiO_2, Al_2O_3, Fe_2O_3, CaO, MgO and TiO_2 suggested that, the periwinkle shell ash can be used as particulate reinforcement in various metal matrices.

Fig. 2-6 show the endothermic curve (heat absorption) which resulted when the sample was heated at a rate of 12°C/min. from 50°C to 500°C in vacuum flowing at a rate of 5 ml/min. The temperatures of these transitions are function of the metal/bio-composition. However, the minimum and maximum heat flow and temperatures are:

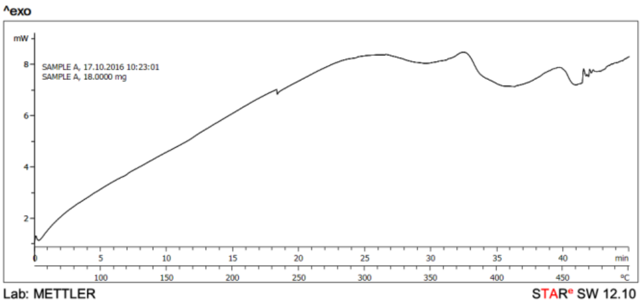

Fig 2: Variation of heat flow with temperature and time of Al/5% PSA addition

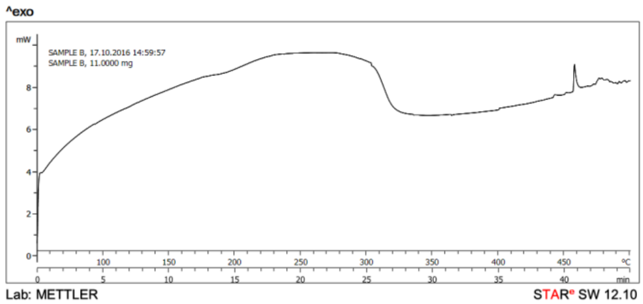

Fig 3: Variation of heat flow with temperature and time of Al/10% PSA addition

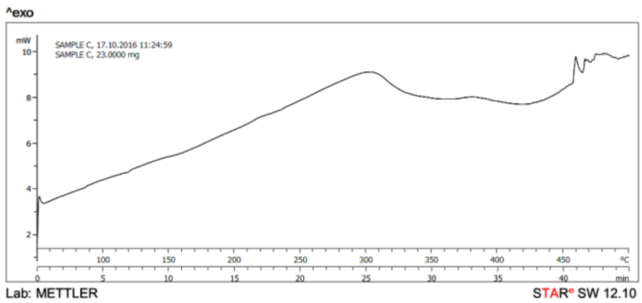

Fig 4: Variation of heat flow with temperature and time of Al/15% PSA addition

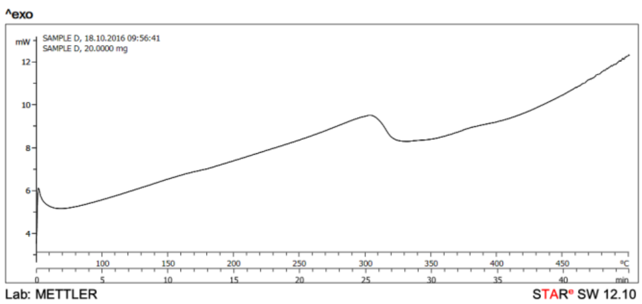

Fig 5: Variation of heat flow with temperature and time of Al/20% PSA addition

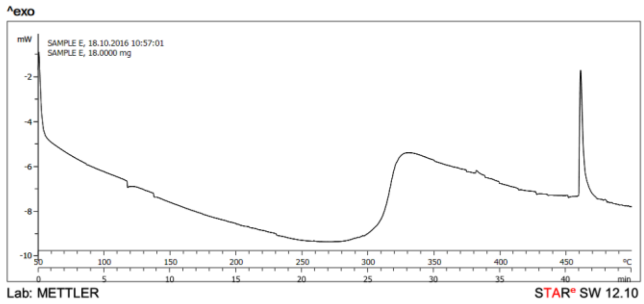

Fig 6: Variation of heat flow with temperature and time of Al/25% PSA addition

From Fig. 2	Min	1.04 *mW* at 50.03 °C;
	Max	8.45 *mW* at 375.04 °C
From Fig. 3	Min	0.58 *mW* at 50.02 °C
	Max	9.61 *mW* at 272.70 °C
From Fig. 4	Min	1.66 *mW* at 50.04 °C
	Max	9.90 *mW* at 482.70 °C
From Fig. 5	Min	3.52 *mW* at 50.09 °C
	Max	12.30 *mW* at 500.38 °C
From Fig. 6	Min	-9.41 *mW* at 271.31 °C
	Max	-0.94 *mW* at 50.47 °C

Endothermic here means that more energy is been absorbed, which indicate that the composite has energy to build up a barrier for dislocation movement as evident in Fig. 2-5. In comparison with Fig. 6 which is exothermic in nature, here the composite is losing more energy to the environment. Also, if more picks occur in the heat treated composite as evident in Fig. 2, it means that the alloy has built up more resistance to the movement of dislocation and hence a higher hardness is achieved. But this did not happen for Fig. 2 in this research. Reasons can be seen from the SEM-EDX analysis.

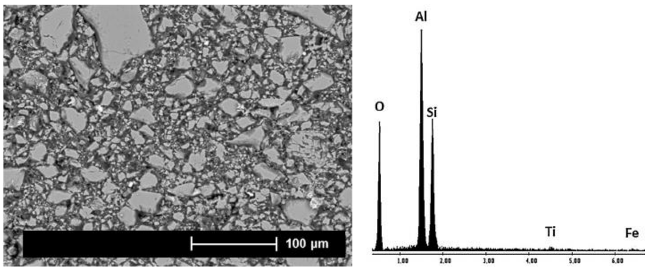

Plate 1: SEM/EDX micrograph of Pure Al

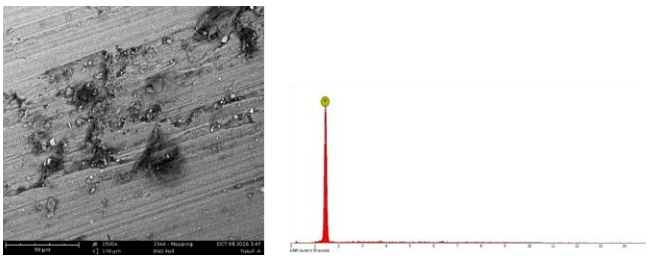

Plate 2: SEM/EDX micrograph of Al/5%PSA composite

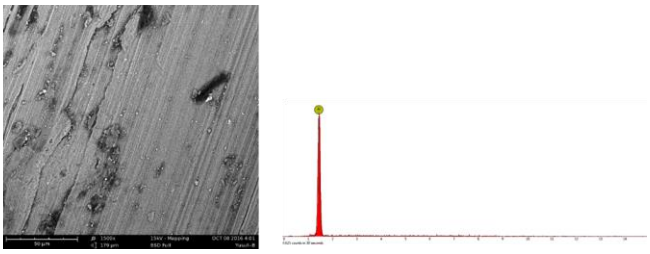

Plate 3: SEM/EDX micrograph of Al/10%PSA composite

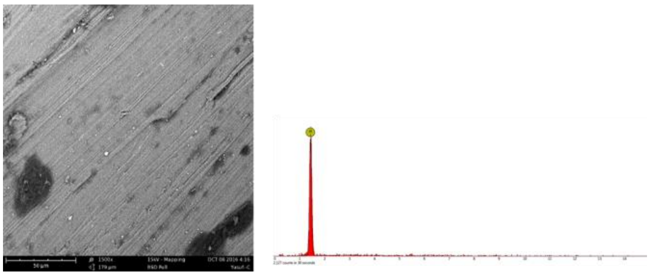

Plate 4: SEM/EDX micrograph of Al/15%PSA composite

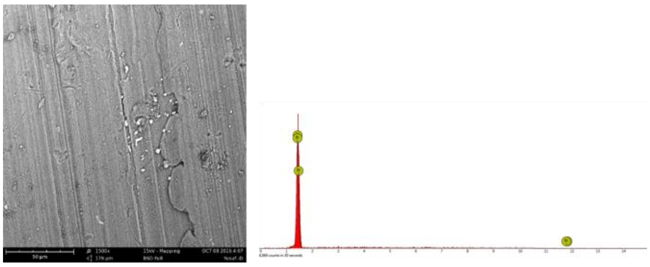

Plate 5: SEM/EDX micrograph of Al/20%PSA composite

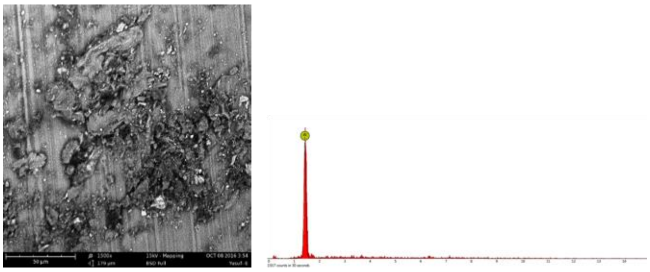

Plate 6: SEM/EDX micrograph of Al/25%PSA composite

Plates 3-5 shows an even distribution of particles, which possibly account for the higher hardness achieved as seen in Table 3. In contrast to this, Table 1, 2 and 6 shows an uneven distribution and larger particle size of the reinforcement, thereby creating a softer phase which possibly accounted for the lower hardness of the composite with this condition of ageing.

Table 3: Hardness result of the composite produced

Composite	HRF value
Pure Al	5.0
Al/5%PSA	7.0
Al/10%PSA	7.3
Al/15%PSA	7.4
Al/20%PSA	7.4
Al/25%PSA	7.0

Conclusion

From the results and discussion made, the following conclusions can be made:

1. Aluminium/periwinkle shell ash particulate composite were synthesized successfully by using stir casting method.
2. Periwinkle shell ash can be used as a reinforcer in metal matrix composites.
3. 10, 15 and 20% periwinkle addition gives better thermal conductivity and hardness value than other proportions (0%, 5%, and 25%). This can possibly be seen from the SEM/EDX analysis showing no particle fragmentation and giving rise to a refined surface, intimate contacts with the aluminum alloy melt formed and obtained a perfect metallurgical bonding after solidification without impurities or gas present at particle-matrix interface.

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