MECHANICAL AND WEAR BEHAVIOUR OF HEAT TREATED ALUMINIUM-CLAY COMPOSITE

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ABSTRACT
In this paper, the mechanical and wear characteristics of T6 heat treated Al-Clay composite with 15 wt.% at 250μm particle size produced via stir casting method were studied. The samples were solutionised at 500, 550 and 600˚C then quenched in air, oil and water. The samples were artificially aged at 180˚C for 6 hours. Tensile and Vickers hardness tests were conducted on the sample in accordance with ASTM E8/E8M-13 standards. The wear tests were carried out on both pin-on-disc wear-testing machine and in dry sliding conditions at two different loads (4N and 10N) and three sliding speeds (200, 500 and 1000 rpm). Results show that the water quenched composite samples solutionised at 550˚C demonstrated superior mechanical and wear properties. The wear behaviour of the developed composite shows dependence on load, speed and the volume fraction of clay particles. From the results it is concluded that the mechanical and wear properties of Al 6063-clay composite can be enhancedthroughT6 heat treatment process.

Keywords: Al-Clay composite, heat treatment, hardness, three-body abrasion, mechanical and wear properties

INTRODUCTION

Aluminium metal matrix composites (AlMMCs) are employed due to their excellent properties such as high strength to weight ratio, stiffness and wear resistance. Recently, great interest has been in the automobile industry, aviation industry, building industry, etc. to reduce strength-to-weight ratio of components, improve wear resistance of components, and or enhance fuel efficiency as evidenced by extensive research into aluminium-based composites (Rao *et al.,* 2009; Rawal, 2001; Das, 2004). The development of desirable mechanical characteristics for a material often results from a phase transformation that is wrought by heat treatment. The process consists of heating a metal or alloy to a specific predetermined temperature, holding at this temperature for required time and finally cooling from this temperature. All these operations are carried out in solid state. Sometimes it becomes necessary to repeat these operations to impart special characteristics. Heat treatment is an important process in the final fabrication of many engineering components.

It is established that Al-Si-Cu-based composites system has been widely investigated both in terms of understanding the mechanism of formation and property evaluation (Hecht and Rex, 2014, Asthana, 1998, Hong *et al.*, 1998); the main reinforcements being SiC and Al_2O_3. Although investigations with varying volume fraction of reinforcements have been done, meaningful results have been obtained when the reinforcement percentage has been restricted to 15. Again the size of the reinforcement is dependent on/or restricted to the processing route adopted (Rupa, 2012).

Song *et al.*, (1995) heat-treated 20 vol.% of SiCp (of size 3 and 20 μm) reinforced Al 2014 and Al 6061 matrix composites under the T6 condition, which involved solution treatment at 530 °C for 1 hour, quenching in cold water and then pre-ageing at room temperature for 20 hours. The materials were aged at temperatures between 50 °C and 250 °C, and changes to the wear resistance were measured using a pin-on-drum machine. The result shows that ageing at the lowest temperatures (between ≈50–150 °C) as seen under TEM, revealed the presence of the solute clusters (e.g. Guinier-Preston (GP) zones) and small coherent precipitates in the aluminium alloy matrices, and these were easily sheared by mobile dislocations. Consequently, the hardness and abrasive wear resistance of these under-aged composites were measured to be relatively low. Raising the ageing temperature to ≈200 °C increased the hardness and abrasion resistance of the composites to the peak-aged condition, and this was associated with the precipitation of small intermetallic compounds which were incoherent with the crystal structures of the aluminium alloy matrices. At 250 °C the composites were over-aged, and this resulted in a reduction in hardness and wear resistance due to the coarsening of the intermetallic precipitates. The composites containing 20 μmSiC particles were slightly more wear resistant than those containing 3 μmSiC. Hamed *et al.*, (2001) subjected SiCp-reinforced Al 359 composite to T6 heat treatment which involved solution treatment for 16 hours at 530 °C, quenching at 60 °C and then ageing for 9 hours at 160 °C. The microstructures of the heat-treated 359/SiC/10p composite materials shows a more uniform distribution of SiC particulates, which clearly depicts the positive effect of heat treatment on uniformity in the distribution of reinforced ceramic particles in metal matrix composites.

Balogun *et al.* (2008) heat-treated Al 6063/SiC metal matrix composite at 415 °C for 1-hour soak time. The results of their study showed that the strength, hardness and fracture resistance of the composite increased with increase in volume fraction of SiC particles in the aluminium MMC. The results further showed that

when ductility is the requirement coarse particulate of SiC would be preferred at low volume fraction of particles. Also with decrease in grains size improved ductility can be achieved by increasing the volume fraction. Both volume fraction and particle size were reported to affect the strength, hardness, Young modulus and fracture resistance of the tested composites.

Rajasekaran *et al*., 2012, studied the effect of aging on the corrosion resistance, the natural and artificial aging behaviour of 15 vol. % 6061 Al-SiCP composites using the T4 and T6 Treatment aging treatment and the Brinell hardness measurements. They observed that aging temperature history has profound effect on the mechanical and corrosion behaviour of 6061 Al/SiC composite. In order to understand the aging curves for the composite (T6 treated) were determined at various aging temperatures such as room temperature, 140, 160, 180, 200, 220, and 240 ºC. According to the peak hardness variation with temperature profile, it is found that the composite is underaged at 140 and 160 ºC. Peak aging takes place at 180˚C with maximum peak hardness ∼1450 MPa. Overaging takes place at 200, 220, and 240 ºC. The natural aging characteristics of the composite (T4 treated) are also studied using the Brinell hardness measurements and the results show that the peak hardness is reached after 500 hours of aging at room temperature. The peak hardness is around 1097 MPa.

The present investigation focus on the effects of heat treatment onthe mechanical properties and wear behaviour of Al-Clay composites.

MATERIALS AND METHODS

In the present work, Al-clay composite of 15 wt. % clay produced via stir casting process was heat treated using T6 process, where the samples were solutionised at 500, 550 and 600ºCand quenched in air, oil and water to determine the effect of solutionising temperature and quenching medium on the mechanical and wear properties of the developed composites. The samples were artificially aged at 180 ºC for 6 hours. Mechanical, physical and wear properties of the composite were evaluated and compared with as-cast composites.

Materials Characterisation

The tensile test and Vickers hardness test at 200g of samples were conducted using respectively 50kN Instron 3369 L3477 machine and Mitutoyo micro-hardness tester HM-122 in accordance with the ASTM E8/E8M-13 standards. Metallographic examination was carried out using Zeiss EVO MA-15 Scanning Electron Microscope (SEM)/Energy Dispersive Xray (EDX). The sample surfaces

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were ground and polished using alumina suspension, and etched in Weck's reagent for 20 seconds and dried in still air. The microstructure of each samples was examined with a Leica CTR6000 digital optical microscope at 50µm magnification.

Wear Characterisation

The wear characteristics of Al-clay composites in dry sliding conditions were subjected to a series of Denison T62 HS pin-on-disc wear tests as in the set-up in Plate 1. Cylindrical pins of 5 mm diameter were made from the Al-Clay composite. The action of two different loads (4 and 10N), on AISI stainless steel disc at three sliding speeds of 1.05 m/s (200 rpm), 2.62 m/s (500 rpm) and 5.24 m/s (1000 rpm) were investigated as per ASTM: G99-05 standard. The mass loss was measured by weighing the pins before and after the test.

Plate 1: Denison T62 HS pin-on-disc wear-testing machine (Leeds, UK)

RESULTS AND DISCUSSION

The results of the spectrochemical and atomic absorption spectroscopic analyses of the aluminium alloy, the clay and the composites are presented in Tables-1-3 respectively.

Table 1: Optical Emission Spectrometric analysis of Aluminium AA6063

Element	Si	Mg	Fe	Cu	Mn	Zn	Cr	Ti	Al
Wt. % composition	0.429	0.425	0.225	0.004	0.026	0.002	0.006	0.033	98.850

Table 2: Atomic Absorption Spectroscopy (AAS) of clay sample

Parameters	SiO_2	Al_2O_3	Fe_2O_3	MnO	MgO	Na_2O	CaO	K_2O	BaO	SO_3	LOI
Level detected (%)	45.62	33.74	0.43	0.01	0.06	0.05	0.04	0.63	0.01	0.03	4.545

Table 3: Optical Emission Spectrometric analysis of 15 wt. % Al-Clay composite

Element	Si	Mg	Fe	Cu	Mn	Zn	Cr	Ti	Al
Wt. % composition	1.982	0.868	0.225	0.124	0.026	1.042	0.086	0.052	95.595

Plates2 and 3 show the SEM/EDX images of the AA6063 and clay samples used in this work respectively.

The presented results in Table 2 and Plate 3 revealed that the clay sample contains the right proportion of Si, Al, Fe, Mg, Na, Ca, and K that placed it in the range of aluminosilicate clay group as classified by Chesti (1986).

The results of spectrometry analysis, SEM micrograph and elemental composition of the 15 wt. % composite sample shown in Table 3 and Figure 1 revealed that there is an increase in the amount of Si, Mg, Cu, and Zn when compare to the conventional AA6063.

The result of the XRD characterisation of the as cast AA6063 and the developed Al-Clay composite are shown in Figure 2.
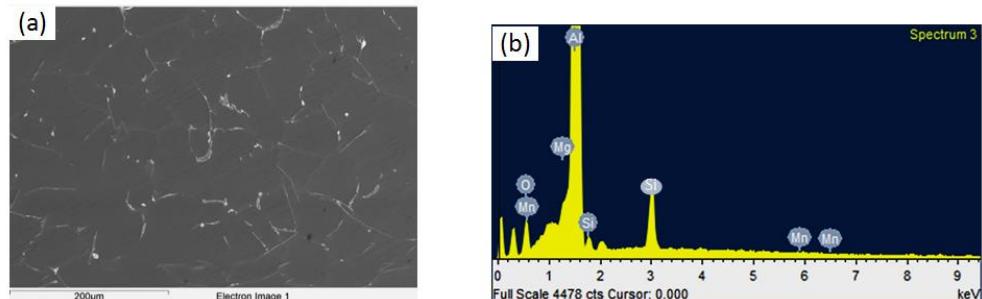
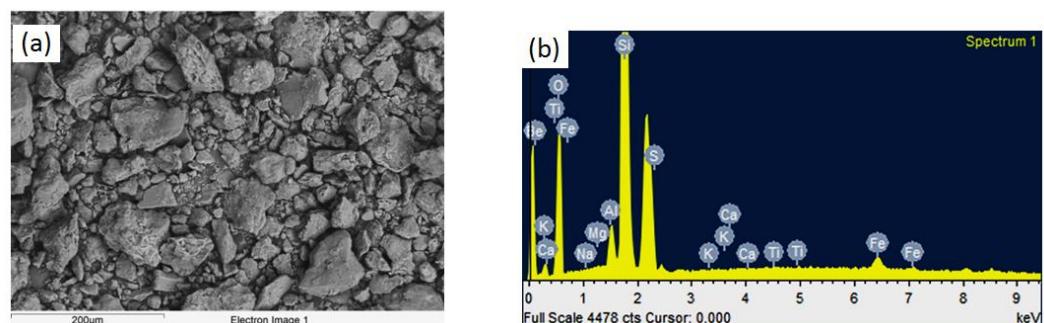**Plate 2: As cast AA6063: (a) SEM photo-micrograph and (b) EDX analysis**

Plate 3: Clay particles (a) SEM photo-micrograph and (b) EDX analysis
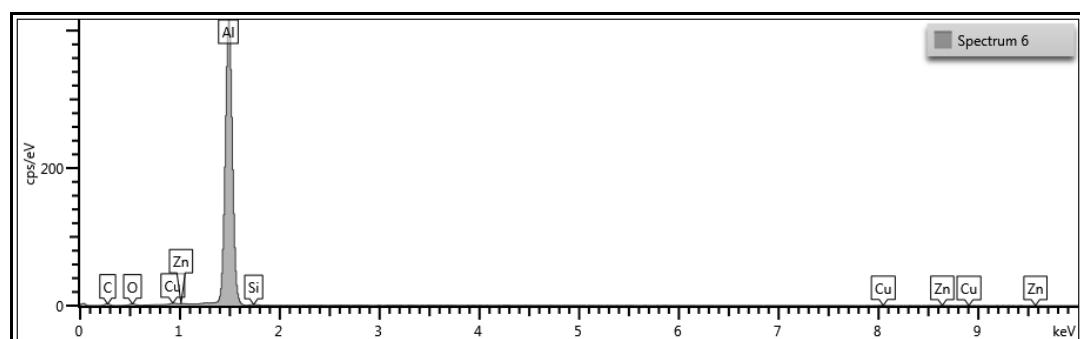

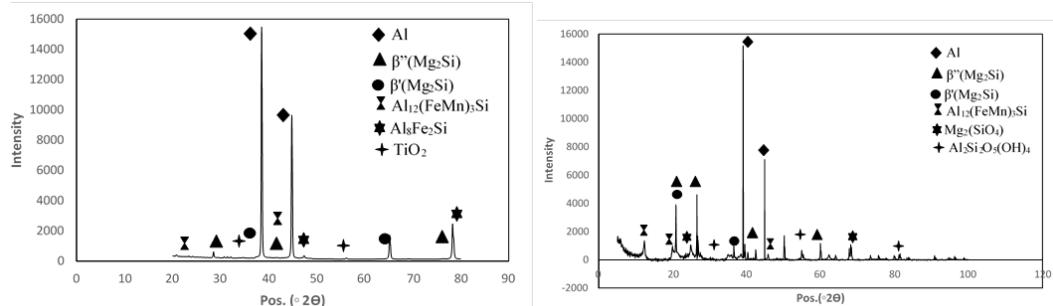**Figure 1: SEM photo-micrograph with EDX analysis of Al/15 wt. % clay composite sample**

Figure 2. XRD analysis of samples (a) AA 6063 (b) Al/15 wt. % clay composite

Microstructures

The precipitation sequence of 6XXX alloys, as reported by Marioara *et al*., (2003) and Gupta *et al*., (2001) is:

$$SSSS \longrightarrow atomic\ clusters \longrightarrow GP\ zones \longrightarrow \beta'' \longrightarrow \beta' \longrightarrow \beta\ (stable);$$

Where, SSSS is super saturated solid solution.

Generally, hardness and strength increase as grain size decreases. Both properties are proportional to the reciprocal of the square root of the average grain diameter (Abubakre *et al*., 2009). The relatively slower cooling rate is responsible for the formation of platelet-like precipitates in specimen quenched in air. Toa lesser extent, (compared to water quenching) the presence of spherodised grains is observed inspecimen quenched in oil (Plate 4).

Similarly, in agreement with previous works (Marioara *et al*., 2003, Kuijpers *et al*., 2003, Miao and Laughlin, 1999, Gupta*et al*., 2001,Biroli*et al*., 1998, Mrówka-Nowotnik and Sieniawski, 2007) the mechanism of aging in examined alloy during artificial ageing at 180˚C prevent the formation of GP zones. In this case, aging process starts with precipitation of intermediate phases - β" and β'. The β' and β'' particles have a needle-shaped morphology that is fully coherent only along the needle axis parallel to <100>Al (Plate 5). The β" phase is the predominant precipitate in the peak-aged condition, since the presence of such a high density of particles induces large strains in the Almatrix, thus effectively obstructing dislocation mobility. The hardness of the alloy increases as far as equilibrium stable β(Mg$_2$Si) phase is formed (Mrówka-Nowotnik and Sieniawski, 2007).

Plates 4-6 show the SEM photomicrographs of the heat treated samples. Table 4 shows the results of EDS of precipitated phases at the tested places 1-6 as indicated in Plates 4-6. The samples solutionised at 550˚C contain a large volume of white block (β(Mg$_2$Si) phase) particles. The results of EDS analysis in Table 4 indicate that samples solutionised at 550 $^\circ$C contain higher Mg and/or Si, whereas the samples solutionised at 500°C and 600˚C have only less Al–Fe–Si–Mg precipitates. Thus, it is possible to suggest that the reason for the superior mechanical and wear properties exhibited by samples solutionised at 550 $^\circ$C is the presence of more strengthening phases than that of 500 $^\circ$C and 600 $^\circ$C.

Previous studies (Marioara *et al*., 2003, Kuijpers *et al*., 2003, Miao and Laughlin, 1999) have shown that the mechanical properties are highly influenced by the precipitates of hardening β(Mg$_2$Si) phase. The strength is also influenced by the intermetallic phases formed during solidification of the alloys. Treakner, 1977 suggested the use of the equations by which either volume fraction of hardening β(Mg$_2$Si)phase and excessive content of Si can be calculated using a chemical composition of an alloy as in Eqs (1-5). This is used to calculate values of volume fraction of β(Mg$_2$Si) and values of the excessive silicon content that can be precipitated in the as cast AA6063 used in this investigation. The result is shown in Table 5.

Si in precipitates of Al(FeMn)Si type
$$zf(\text{Si}) = 0.25 \,(\%\text{Fe} + \%\text{Mn}) \tag{1}$$
Si in β(Mg$_2$Si) formed in reaction with Mg
$$zm(\text{Si}) = 0.578 \,(\%\text{Mg}) \tag{2}$$
β(Mg2Si) phase content $w(\text{Mg2Si}) = (\text{Mg}) + zm(\text{Si}) \tag{3}$
Remaining content of Si, $nf(\text{Si}) = (\%\text{Si}) - zf(\text{Si}) \tag{4}$
Excessive content of Si, $n(Si) = nf(\text{Si}) - zm(\text{Si}) \tag{5}$

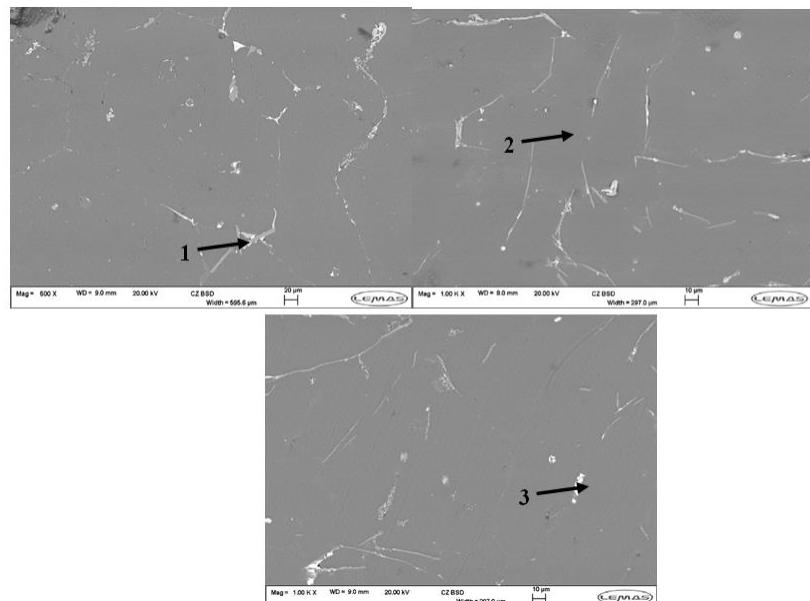**Plate 4:** **SEM photo-micrographs of Al-Clay composites solutionised at 500 ºC quenched in: (a) Air (b) Oil, and (c) water**
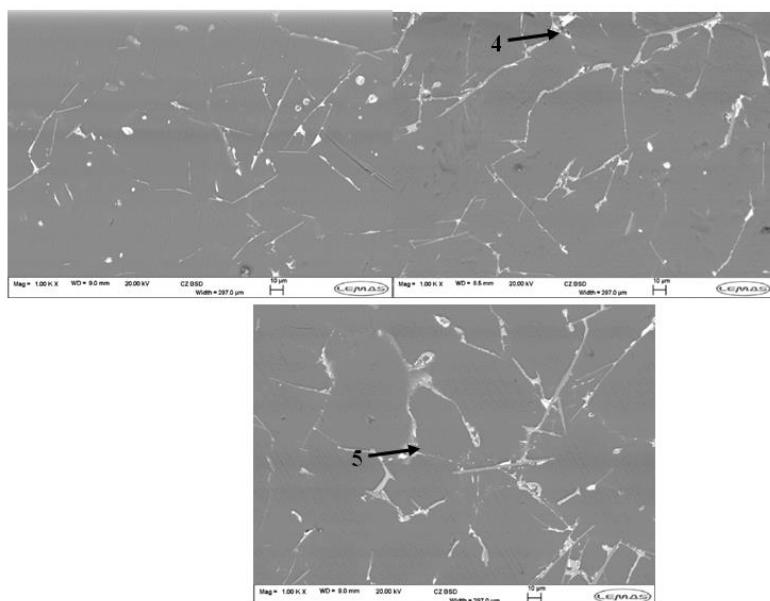

Plate 5: **SEM photo-micrographs of Al-Clay composites solutionised at 550 ºC quenched in: (a) Air (b) Oil, and (c) water**
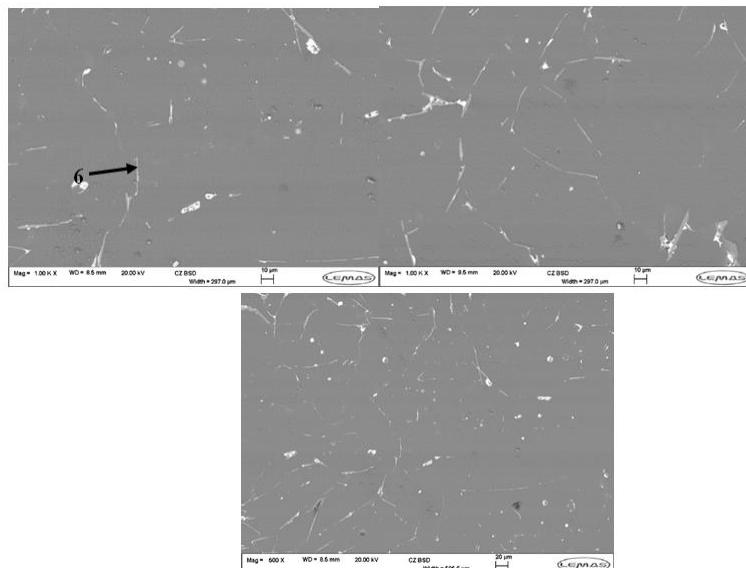
Plate 6: **SEM photo-micrographs of Al-Clay composites solutionised at 600 ºC quenched in: (a) Air (b) Oil, and (c) water**

Table 4: Results of EDS of selected precipitated phases in Plates3-5

Tested place	Si	Mg	Fe	Cu	Zn	Al
1	6.22	1.73	3.11	1.43	0.55	Balance
2	3.34	0.58	2.08	1.25	1.79	Balance
3	4.89	0.81	6.85	0.11	1.63	Balance
4	9.28	1.41	0.12	1.58	1.50	Balance
5	5.75	1.35	0.98	1.03	1.22	Balance
6	3.99	0.66	5.22	2.13	2.11	Balance

Formation of other intermetallic phases as seen in Table 5 have significant effect on decreasing volume fraction of hardening precipitates ofβ(Mg_2Si) phase, thereby decreasing the effectiveness of hardening due to particles of this phase (Zuo and Jing, 2009).

Table 5: Calculated values of volume fraction of β(Mg_2Si) and values of the excessive silicon content in the investigated alloy (Eqs(1-5)).

Alloy	*zf*(Si)	*zm*(Si)	*nf*(Si)	*w*(Mg_2Si)	*n*(Si)
AA6063	0.063	0.246	0.366	0.671	0.12

Mechanical Properties

The results of the mechanical properties are shown in Figures 3-5. These figures give a summary of the Vickers hardness number, ultimate tensile strength (UTS), and percent elongation as a function of the solutionising temperatures and cooling media. It can be seen that the heat treated samples show significant improvement in the mechanical properties of the developed composites as compared to the as-cast samples.

The Vickers hardness values of the T6 treated Al-clay composite are shown in Figure 3. The peak hardness value of 128.3 HV was achieved at water quenched samples with solutionising temperature of 550 $^{\circ}$C. According to the A1-Mg-Si phase diagram melting of ternary eutectic Mg_2Si-(Al)-(Mg) phase takes place at about 550 $^{\circ}$C (Rajasekaran *et al*., 2011).

Clusters of silicon atoms and vacancies appear almost immediately after quenching, and then magnesium atoms precipitate on nuclei (Salvo and Suery, 1994). These nuclei grow to the formation of a needle-shaped phase with growth axis (major axis) lined up on the <100> matrix direction and ranging in length between 20 and 100 nm. Such zones are coherent with the matrix lattice in the <100>direction and progressively evolve toward a more ordered state. During this process mechanical properties are improved due to development of micro strains in the lattice.
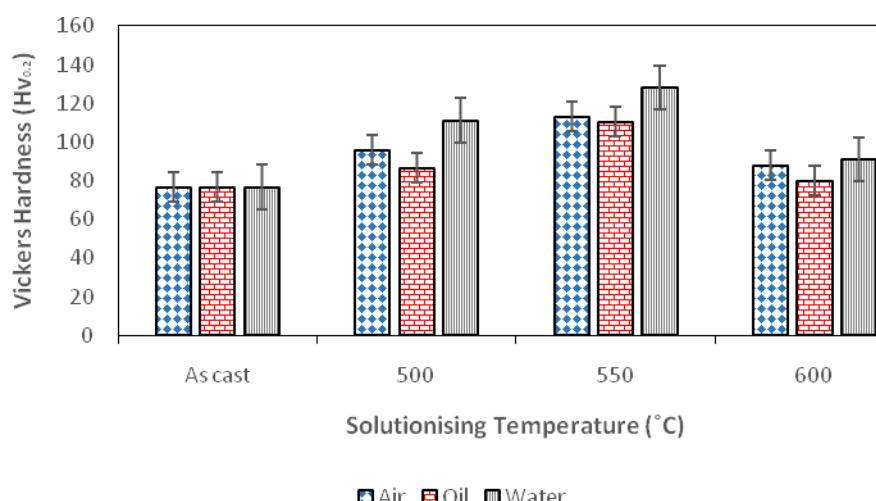

Figure 3: Vickers Hardness (HV) of Al-Clay composite samples at varying solutionising temperature and quenching media

The UTS values of the composite peaked at 238.7 MPa with samples quenched in water and solutionised at 550 $^{\circ}$C. The results also indicate the influence of the T6 heat treatment on the ductility of the composite (Figure 5). This is evident with a decrease in percent elongation, which declined to 4 % in water quenched samples solutionised at 600 $^{\circ}$C. Samples quenched in oil are more ductile in relative to the air and water quenched samples due to the presence of spherodised grains which enhances movement of dislocation.
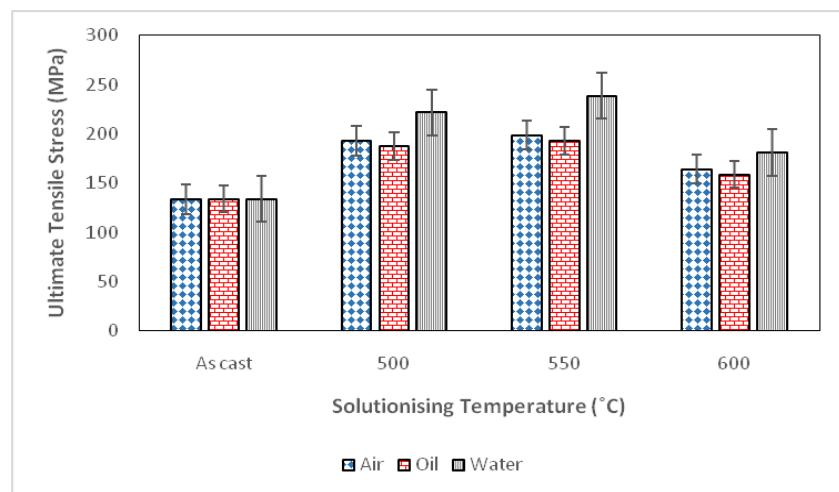

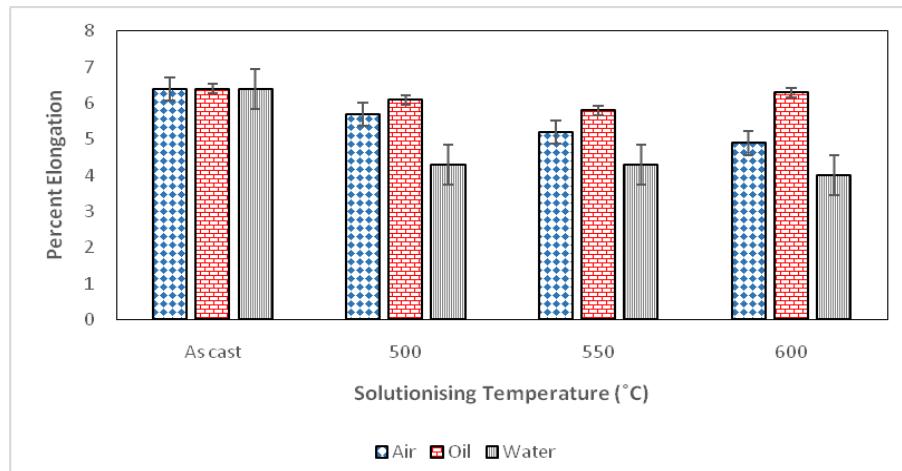**Figure 4:** **UTS of Al-Clay composite samples at varying solutionising temperature and quenching media**

Figure 5: **Percent elongation of Al-Clay composite samples at varying solutionising temperature and quenching media**

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Wear Characteristics

The results of the wear test of both the as-cast and the heat treated composites are displayed in Figures 6-8.

The wear rate was at minimal in water quenched composites solutionised at $550^{\circ}C$. The improved wear resistance observed here is due to the very high rate of heat extraction experienced by the samples which creates the possibility of having more precipitates during the ageing process. The precipitates are also uniformly distributed in AA6063 matrix.
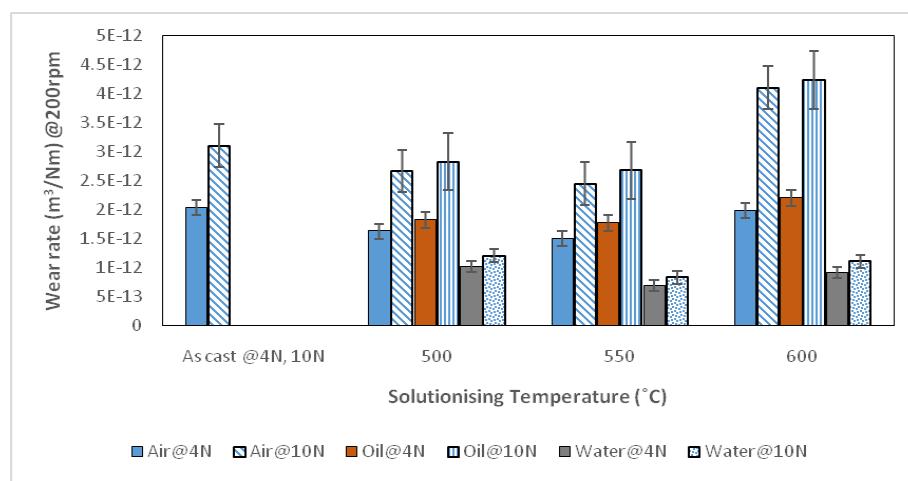

Figure 6: **Wear rate at200 rpm of Al-Clay composite samples at varying solutionising temperature and quenching media**
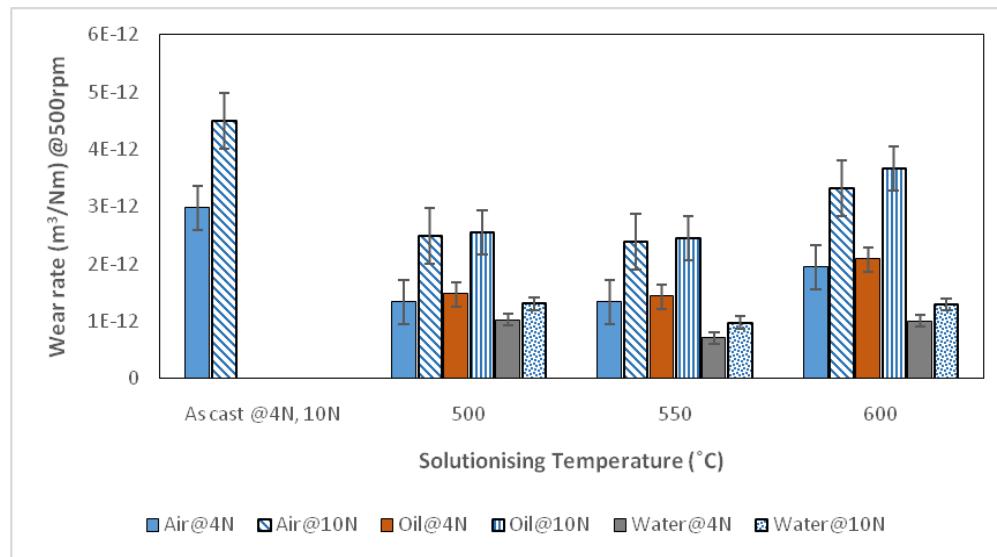
Figure 7: **Wear rate at 500 rpm of Al-Clay composite samples at varying solutionising temperature and quenching media**
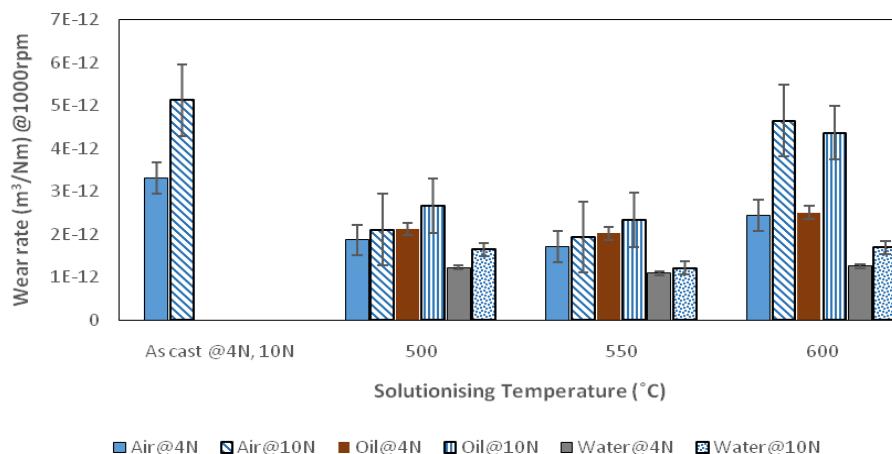

Figure 8: **Wear rate at 1000 rpm of Al-Clay composite samples at varying solutionising temperature and quenching media**

CONCLUSIONS

It can be concluded that:

(i)   The wear behaviour of the developed Al-Clay composites show dependence on load, speed, solutionising temperature and the quenching media.

(ii)   The mechanical properties and wear behaviour of the T6 heat treated Al-Clay composite samples were significantly enhanced.

(iii)   The formation and distribution of the block-like $\beta(Mg_2Si)$ phase particles is uniformed around the grain boundaries of the microstructure and generally improve the mechanical and wear properties of the developed Al-Clay composites.

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