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Effect of Step Quenching Ageing on the Wear and Corrosion Properties of Al-Cu-Mg/ 3% Rice Husk Ash Composite

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ABSTRACT

This study investigates the impact of Step Quenching Ageing on the Wear and Corrosion resistance behaviour and microstructure of Al-Cu-Mg/ 3% Rice Husk Ash Content that was fabricated via stir casting technique. The temper conditions of the sample involve pre aging at 90°C for 1 hour and further heat treating to 180°C for 1-5 hours respectively. This was compared with the control sample that was aged at 180°C for 5 hours. The corrosion resistance of the Al-Cu-Mg/3%RHA composite was determined using linear polarization resistance technique, while the wear rate was determined using a pin-on disc tribometer. The findings showed improved corrosion and wear rate behaviour of pre-aged samples having a corrosion rate of 9.727E02 mm/yr when put side by side with the control sample, particularly for the 3 hours trials. It was observed that more precipitates (possibly Al, Cu, Al,CuMg, Mg_2Cu or $MgCu_2$) were established and seen to be more uniformly dispersed in the pre-aged specimens than control sample. It can be affirmed that the step quenching treatment has enhanced the corrosion and wear characteristic of the composite being invested.

Keywords: Age-hardening, Alloying, Reinforcement, Stir-casting, Linear polarization

INTRODUCTION

An alloy is a constituent that has metallic characteristics made via arrangement of two or more chemical elements, with at least a metal. The metallic atoms must govern its chemical composition and the metallic bond in its crystal structure. Commonly, alloys have diverse qualities from those of the component elements (Joseph and Babaremu, 2019 and Suleiman *et al.*, 2018). The properties of an alloy is often enhances depending on the type of metal that it is made from by combination of one or more other metals or non-metals with it. The physical attributes, such as density and conductivity of an alloy may not vary significantly from the individual component element, nevertheless its engineering attributes for instance tensile strength and shear strength could noticeably not be the same from the parent materials (Akinwamide *et al.*, 2020 and Bill and Gareth, 2003).

Aluminum (Al) alloys are widely used in various engineering applications due to their excellent combination of strength, density, formability, and corrosion resistance (Kokani and Kokani, 2004). However, their applicability can be further enhanced by incorporating reinforcements to form metal matrix composites (MMCs). Rice husk ash (RHA), a waste product generated during rice processing, has emerged as a promising reinforcement material due to its low density,

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abundance, and relatively high silica content, SiO_2 (Kumar *et al*., 2012 ; Rioja and Sheppard, 2003). Al-Cu-Mg alloys represent a significant class of age-hardenable Al alloys, where the introduction of copper (Cu) and magnesium (Mg) elements enables precipitation strengthening mechanisms. This process involves the formation of intermetallic phases during a specific heat treatment regime, leading to enhanced mechanical properties (Lil, 2011). Step quenching ageing is a specific heat treatment technique that involves quenching the material from a solutionizing temperature to an intermediate temperature, holding it for a specific duration, and then finally quenching it to room temperature. This approach offers potential benefits compared to conventional single-stage ageing by allowing for more precise control over the precipitation process (Abdulwahab et al., 2011B; Yang *et al*., 2006).

Investigating the influence of step quenching ageing on the wear and corrosion properties of Al-Cu-Mg composites reinforced with rice husk (Al-Cu-Mg/RHA) is crucial for several reasons. MMC wear performance is significantly influenced by the reinforcement phase and the matrix properties. Rice husk ash (RHA), primarily composed of SiO_2, can act as a hard-ceramic phase, enhancing the composite's wear resistance by providing abrasion resistance and promoting load-bearing capacity (Osorio *et al*., 2007; Rana *et al*., 2012). Step quenching ageing can potentially influence the matrix microstructure and the interfacial bonding between the matrix and the RHA particles, impacting wear behavior. The introduction of RHA into the Al matrix can introduce galvanic couples, potentially accelerating localized corrosion. However, the presence of SiO_2 can also act as a physical barrier, hindering the penetration of corrosive agents. Step quenching ageing can influence the distribution and morphology of intermetallic precipitates within the Al matrix, potentially affecting the composite's overall corrosion resistance (Zlaticanin *et al*., 2004; Zhu, 2011).

Understanding the interplay between step quenching ageing, the presence of RHA, and the resulting wear and corrosion properties of Al-Cu-Mg/RHA composites is essential for their potential application in various wear and corrosion-prone environments. This project aims to bridge this knowledge gap by systematically investigating the effect of different step quenching ageing parameters on the wear and corrosion behavior of Al-Cu-Mg/RHA composites. The findings can contribute to the innovation of new Al-Cu-Mg/RHA composites by way of enhancing the wear and corrosion resistance for tribological and corrosive usage. Even though the Al-Cu-Mg alloys offer many benefits, the long-term effects of thermal ageing, via techniques like step quenching, on their corrosion and wear properties, and especially when reinforced with RHA, are not fully understood. This knowledge gap deters the progression of Al-Cu-Mg/RHA composites designed for uses demanding both better accomplishment and enduring strength at high temperatures.

MATERIALS AND METHODS

The materials investigated comprised: high purity Aluminium (Al) wires acquired from Northern Cable Company (NOCACO) Kaduna-Nigeria, Aluminium-Copper ligand (Al_2Cu), Magnesium powder (Mg), Rice Husk Ash (Ash), distilled water, Sodium hydroxide (NaOH). Also the equipment adopted were Muffle electrical resistance furnace (employed in heat treatment having a capacity of 1200^0C), Ball on disc tribometer (implemented for wear rate determination), Lathe machine (useful in turning and facing), Hacksaw, Hand file, Vernier caliper, Ball mill, Metallurgical microscope, Sieve aperture and Polishing machine.

Rice Husk Ash Production

The rice husk ash was heated in the furnace, at a temperature of 700°C, to produce the ash. The rice husk burns to char and further heating turns it to ash. The ash was then ground by means of ball mill. The milled ash was sieved with a 150 microns sieve aperture. The chemical composition of the rice husk is as shown in Table 1.

Table1: Chemical composition of Rice Husk Ash (%Wt)

Chemical Composition	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	N_2O	K_2O	LOI
Percentage Composition (%)	94.04	0.249	0.136	0.622	0.442	0.023	2.49	3.52

Source: (Umaru *et al*., 2016A)

Preparation of Al-Cu-Mg/3%RHA Composites

In line with the technique illustrated by Umaru *et al* (2016A), stir casting method was employed in the production of the composite. The Charge calculation used in determining the compositions required to produce the alloy was shown in Table 2 and that of the composite in Table 3. Aluminium cable wire was first charged into the wire muffle furnace and was heated to a temperature above the melting point of aluminum (> 700°C) to ensure the wire melts completely. The Aluminium-copper ligand was then charged into the crucible and was also allowed to melt after heating for 15 minutes. The magnesium powder was then added and the molten composition was stirred using a stirring rod. The slag formed was removed using slag scooper. The ash was added and the mix was manually stirred for about 5mins. The melt was then superheated at 800°C and stirred thoroughly before pouring into the prepared sand molds to produce three bars of the cast composite of size 20x300mm.

Table 2: Charge composition of the Al-Cu-Mg alloy (%wt.).

Element	Al	Cu	Mg
Percentage Composition (%)	94.9	3.7	1.4

Table 3: Charge composition of Al-Cu-Mg/RHA composite produced

Chemical Composition	Matrix (Al-Cu-Mg)	Reinforcement
Percentage Composition (%)	97	3

Machining of the Produced Composite

The Al-Cu-Mg/RHA composite produced was machined to corrosion and wear sizes in accordance with ASTM standards.

Heat treatment of Produced Composite

A total of eighteen pieces for wear, corrosion and microstructure were machined and solution heat treated. The solution heat treatment involved heating the samples to 540°C and holding at that temperature for one hour in an electrical heat treatment furnace. The samples were then rapidly quenched in water (at room temperature). The quenched samples were then given step quenching aging (DTAT) by pre aging at 90°C for one hour followed by aging at 180°C for 1-5 hours at 1hour intervals respectively.

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Corrosion analysis of the heat-treated Al-Cu—Mg/ RHA Alloy

The electrochemical corrosion technique used in this research was linear polarization corrosion technique.

Linear Polarization Technique

A total of six (6) samples with the control sample were subjected to electrochemical linear polarization technique to characterize the corrosion rate (current density). The reference electrode, working electrode and auxiliary were assembled together with the samples facing the reference electrode and in contact with the solution, and then the setup (potentiostat) was coupled to a computer system. linear polarization test was carried out in a 0.5M NaOH simulated seawater solution at room temperature (RT) in a static solution for a period of 30 minutes each using a potentiostat and the corrosion rate was determined using open circuit potential (OPC). Corrosion parameters such as corrosion potential (Ecorr), corrosion current density (icorr), anodic slope (ba) and cathodic slope (bc) were obtained from the Linear polarization curves. Results of Linear polarization studies of control sample and samples aged to 1-5 hours which were all immersed in 0.5M NaOH solution are then tabulated. The corrosion rate was then calculated using Eq. (1).

$$\text{CR (mm/yr)} = \frac{3270\text{xMxicorr}}{\rho\text{xZ}} \quad (1)$$

where 3270 is a constant that defines the unit of corrosion rate, icorr is the corrosion current density in A/cm2, p is the density of the corroding material (g/cm3), M is the atomic mass of the metal, and Z is the number of electrons transferred per atom (Abdulwahab *et al*., 2012; Abdulwahab *et al*., 2016).

Wear Rate Analysis of the Heat-Treated Al-Cu-Mg/RHA alloy

The wear analysis was carried out on the surface of the samples using a ball on disc tribometer. This test was conducted according to ASTM G190-15 and ASTM G115-10 standards. In this test, a fixed ball-shaped body (stainless steel) is loaded on top of the rotating disc-lower specimen (Al-Cu-Mg/RHA alloy) under the following conditions load of 10 N, speed of 250 rpm and a distance of 7.95 m in accordance with other works (Abdulwahab *et al*., 2011A; Abdulwahab *et al*.,2018; Umaru *et al*., 2012). The entire test was performed at room temperature and under unidirectional (clockwise) motion.

Microstructural Analysis of the Heat-Treated Al-Cu-Mg/RHA Alloy

Metallographic samples were prepared by cutting them from larger pieces, mounting them in a plastic material, and grinding them down to a smooth surface using sandpaper and water. The samples were then polished using a very fine abrasive powder suspended in water. Finally, the samples were etched using a chemical solution and examined using a scanning electron microscope to determine their surface structure.

RESULTS AND DISCUSSION

Table 4 show the LPR result of the heat treated composite, while Figure 4 show the corrosion rate of the composite as a function of time. Also, Plate 1 show the worn-out track of the composite at various conditions of ageing treatment, while Plate 2 show the SEM analysis of the composite at different ageing condition.

Corrosion Study of the Heat-Treated composite

Corrosion rate of the heat-treated composite of Al-Cu- Mg/ RHA was studied using linear

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polarization technique in a solution of 0.5M NaOH and the result presented in Table 4, while the result of corrosion rate of Al-Cu-Mg/RHA alloy as function of time is presented in Figure 1. From the result, the control sample exhibited the highest corrosion rate, while the heat-treated samples displayed the lowest corrosion rate, with optimum corrosion resistance achieved after 3 hours of aging time.

Table 4: LPR result of the heat-treated composite.

SAMPLE	Ba (V/dec)	Bc (V/dec)	E_{cor} Cal(V)	I_{corr} (A)	Corrosion rate (mm/yr)	Polarization resistance (Ω)	E Begin (V)	E End (V)
1hr	4.504	5.139	-1.28	5.124E03	1.179E03	9	-1.5	1.5
2hrs	4.686	5.481	-1.10	4.646E03	1.069E03	9	-1.5	1.5
3hrs	4.720	5.162	-1.10	4.228E03	9.727E02	10	-1.5	1.5
4hrs	4.223	5.147	-1.30	5.656E03	1.301E03	8	-1.5	1.5
5hrs	4.167	5.201	-1.35	6.414E03	1.476E03	7	-1.5	1.5
Control	4.317	5.239	-1.32	1.082E02	2.489E03	4	-1.5	1.5

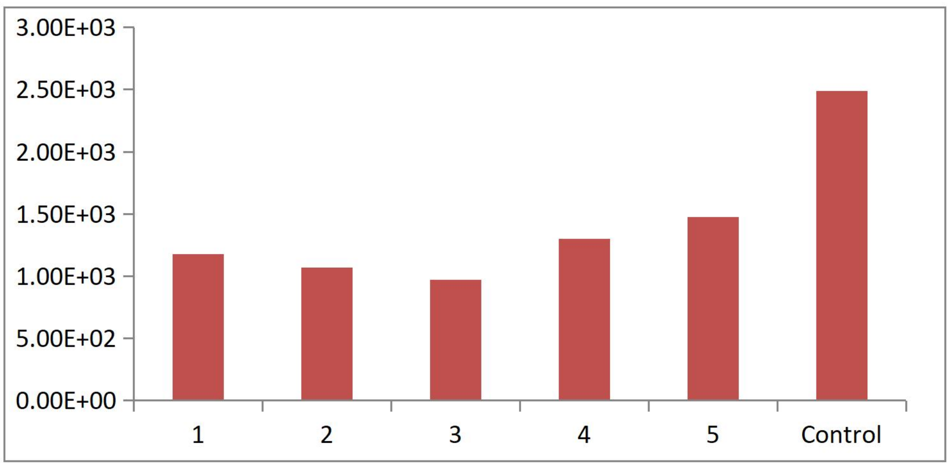

Figure 1: The corrosion rate of Al-Cu-Mg/RHA composite as function of time.

The superior corrosion performance of the heat-treated samples can be attributed to a reduction in grain boundaries observed on their surfaces compared to the control sample (as shown in Plate 2). The presence of more grain boundaries in the control sample is directly correlated with a higher susceptibility to corrosion.

Wear Study of the Heat-Treated Composite

Wear rate of the heat-treated composite of Al-Cu- Mg/ RHA was studied using pin-on disc technique, carried out with a uniform load of 10N and presented in Figure 2. The result reveals that the non pre-aged (control) sample had the highest wear rate. In contrast, samples that underwent pre-aging displayed significantly lower wear rates. The sample pre-aged for 1 hour exhibited the highest wear resistance, with a wear rate of 0.149 g/m compared to 0.245 g/m for the control sample.

Scanning electron microscopy (SEM) analysis further confirmed the significant influence of pre-aging time and temperature. The control sample's higher wear rate suggests that pre-aging at 90°C positively affects wear resistance. Plate 1 visually demonstrates this, showing a deeper wear track indicating more material loss compared to the sample pre-aged for 1 hour.

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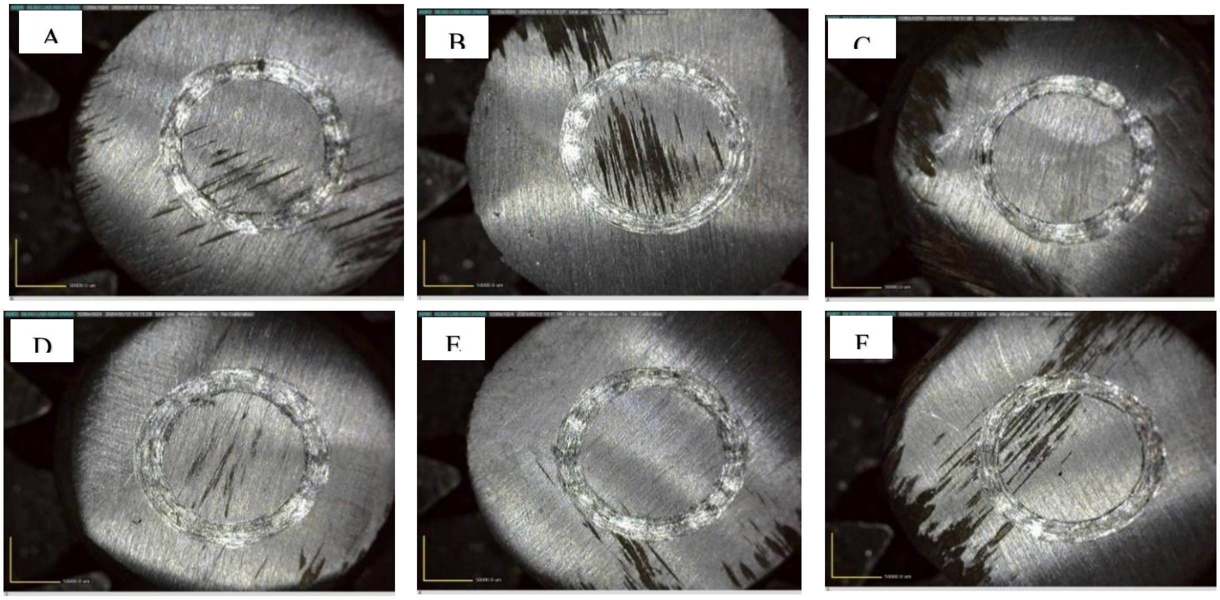

Plate 1: Worn track of A-the control sample, B- Sample pre-aged at 90^0C & aged at 180^0C/ 1hr, C- Sample pre-aged at 90^0C & aged at 180^0C/ 2hr, D- Sample pre-aged at 90^0C & aged at 180^0C/ 3hr, E- Sample pre-aged at 90^0C & aged at 180^0C/ 4hr and F- Sample pre-aged at 90^0C & aged at 180^0C/ 5hr

The passage suggests that a higher pre-aging temperature combined with a shorter pre-aging time might promote the precipitation of solute atoms, leading to improved wear resistance. This aligns with findings reported in other research (Abdulwahab *et al*., 2011B; Umaru *et al*., 2013).

From Figure 2, the relationship between wear rate and time for the Al-Cu-Mg/RHA alloy is nonlinear. The wear rate increases significantly between 1 and 2 hours, and then plateaus until 4 hours. After 4 hours, the wear rate increases again until 5 hours and control sample, the wear rate remain relatively constant. Moreover, there were some variations in the wear rates among aged samples, suggesting that factors beyond aging time might also influence wear behavior.

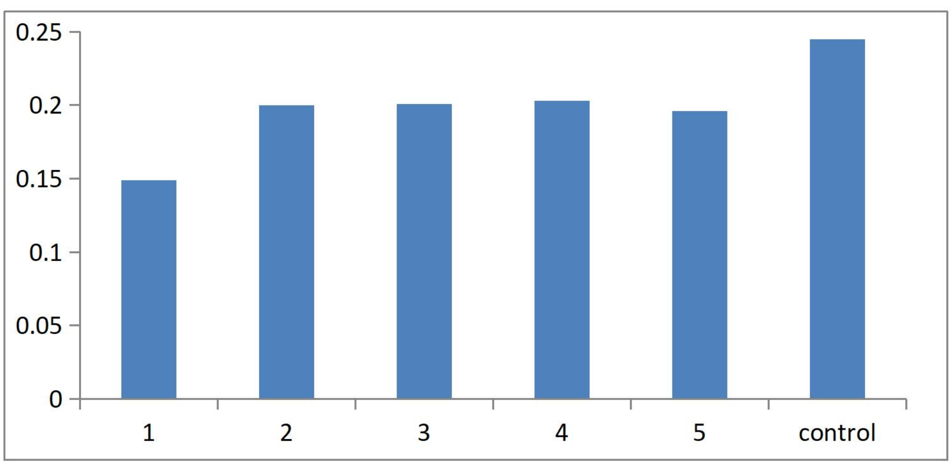

Figure 2: The wear rate of Al-Cu-Mg/RHA alloy as function of time.

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Scanning Electron Microscopic Study of the Heat-Treated Composite

The surface characteristics of Al-Cu-Mg/RHA alloy samples were analyzed using a scanning electron microscope (SEM) after undergoing a wear test and presented in Plate 2. The SEM images reveal the surface morphology of the samples. The wear sample shown in Plate 2 exhibits a combination of abrasive and adhesive wear, suggesting a softer or more ductile material that can transfer and adhere to other surfaces. In contrast, the control sample depicted in Plate 1 primarily shows abrasive wear, indicating a harder and more brittle material.

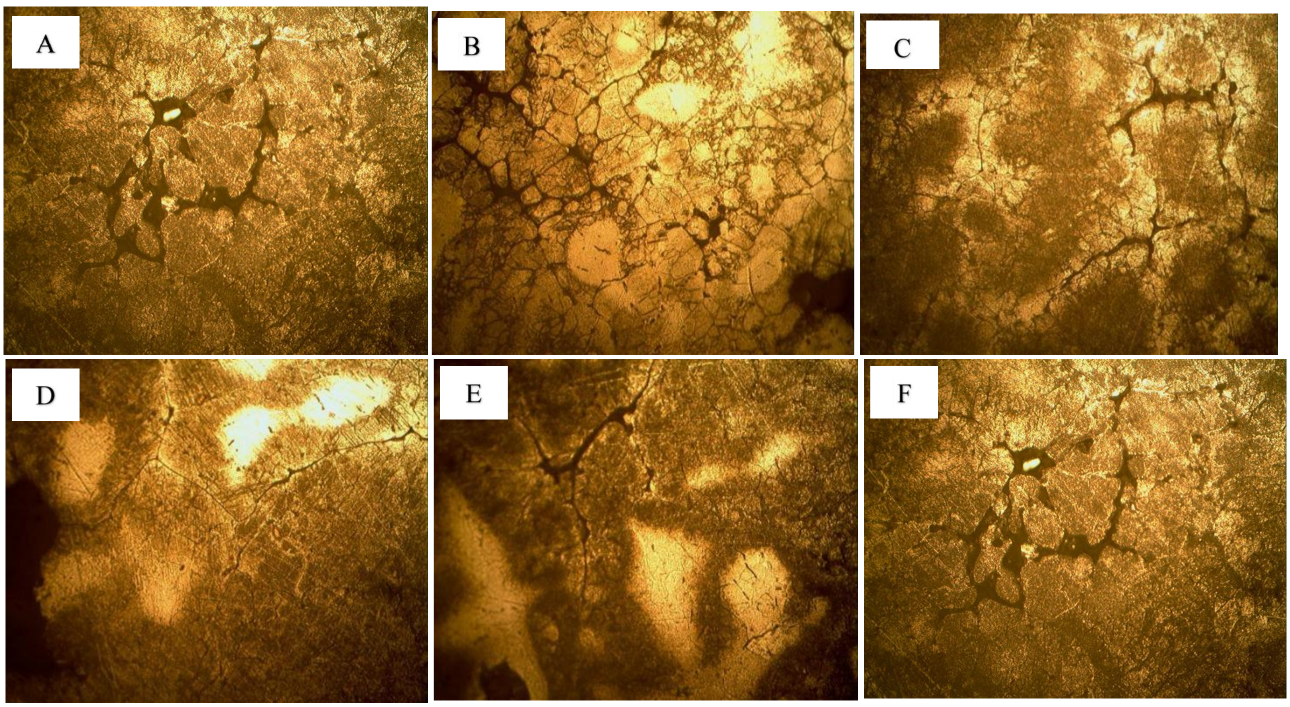

Plate 2: SEM analysis of A-the control sample, B- Sample pre-aged at 90^{0}C & aged at 180^{0}C/ 1hr, C- Sample pre-aged at 90^{0}C & aged at 180^{0}C/ 2hr, D- Sample pre-aged at 90^{0}C & aged at 180^{0}C/ 3hr, E- Sample pre-aged at 90^{0}C & aged at 180^{0}C/ 4hr and F- Sample pre-aged at 90^{0}C & aged at 180^{0}C/ 5hr

The Microstructural analysis showed that more precipitates (possibly Al, Cu, AlCuMg, Mg_2Cu or $MgCu_2$) may have been formed and that they were more evenly distributed in the pre-aged sample than control sample (Umaru *et al*., 2016B; Umaru *et al*., 2013). The microstructure also disclosed that the control samples exhibited abrasive wear for the most part, while the wear sample displayed a combination of abrasive and adhesive wear, suggesting a softer and more ductile material.

CONCLUSION

The investigation of the effect of step quenching ageing on wear and corrosion resistance on Al-Cu-Mg 3% RHA composite was conducted by solutionizing at temperatures of 560^{o}C, pre-ageing at 1 hour at a temperature of 90^o C and ageing temperature of 180^o C for 1-5 hours respectively and compared with the control samples. From the discussions made, the above conclusion can be drawn; i) The Al-Cu-Mg 3% RHA composite was cast then solutionizing at temperature of

560^{0}C, pre-ageing at 1 hour with a temperature of 90^0 C and followed by ageing at a temperature of 180^0 C with an interval of 1 hour respectively. ii) The heat-treated Al-Cu-Mg/RHA composite exhibited significantly lower corrosion rates than the control sample. This improvement can be attributed to a reduction in grain boundaries, which are known to be susceptible to corrosion. iii) The wear mechanism of the Al-Cu-Mg/RHA composite was primarily abrasive in the control sample, indicating a harder and more brittle material. In contrast, the wear sample exhibited a combination of abrasive and adhesive wear, suggesting a softer and more ductile material. iv) The Microstructural analysis suggests that more precipitates (likely Al, Cu, AlCuMg, Mg_2Cu or $MgCu_2$) were formed. It also indicated that there were more evenly distributions in the pre-aged sample than control sample.

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