



Magnesium inclusion effect on Al-Zn-Cu alloys: A study on microstructure and mechanical properties

J.K. Odusote^a, A.A. Adeleke^{b,*}, S.A. Muraina^c, P.P. Ikubanni^b, I.M.B. Omiogbemi^{d,e,*}

^a Department of Materials and Metallurgical Engineering, University of Ilorin, P.M.B. 1515, Ilorin, Nigeria

^b Department of Mechanical Engineering, Landmark University, P.M.B. 1001, Omu-Aran, Nigeria

^c Department of Mechanical Engineering, University of Ilorin, P.M.B. 1515, Ilorin, Nigeria

^d Mechanical Engineering Department, Air Force Institute of Technology, Kaduna, Nigeria

^e Ahmadu Bello University, Zaria 800282, Nigeria

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ABSTRACT

The microstructure and mechanical properties of Al-Zn-Cu alloy with magnesium inclusion varying between 0.5 and 1.5 wt% were explored in this investigation. Al-Zn-Cu-Mg alloy was prepared by sand casting. Heat-treatment was done on the cast alloy samples at 460 °C for 2 h, which was then water-quenched. The samples at 160 °C were age-hardened for 5 h. Mechanical tests were done on both the heat-treated and as-cast alloy samples. Optical and scanning electron microscopy were used for the surface morphology of the samples. The maximum tensile strength (178.04 N/mm²) and hardness value (42.49 HB) were obtained from the Al-Zn-Cu-Mg alloy samples with 0.33 wt% Mg and 0.001 wt% Mg, respectively. In the as-cast samples, the reinforcing intermetallic phases present was coarse in nature while the precipitation hardened samples showed well-distributed reinforcing intermetallic phases which are fine grain size. Hence, the tensile strength of the cast Al-Zn-Cu-Mg alloy was positively influenced with the addition of magnesium while precipitation hardening eliminates micro segregations, thus, Al-Zn-Cu-Mg alloy mechanical properties were improved. Thus, the Al-Zn-Cu-Mg alloy can be useful in automobile industry.

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1. Introduction

Aluminum is used in automobile and aerospace industries majorly because of its lightweight, corrosion resistance and attractive appearance. Its good electrical and thermal conductivity, high reflectivity and non-sparking characteristics are also important for other applications. After iron and its alloys, aluminium and its alloys have been considered as the second most widely utilized materials in the world due to the unique combination of its properties [1]. Generally, aluminum can be alloyed with other elements including magnesium (Mg), silicon (Si), copper (Cu), zinc (Zn), and so on. The inclusion of these elemental components produces aluminum alloy with incredible casting properties which include such

as high level of fluidity in liquid stage, low softening (melting) point, and low shrinkage on solidifying [2]. Aluminum alloys possess good mechanical properties and low specific weight which have made them to be progressively utilized in the synthesis of moulded parts, particularly in place of iron and its alloys. Alloys of magnesium offer outstanding and incredible blends of mechanical and physical properties, for instance, high explicit quality of strength, light weight, low thickness and density, and astounding electromagnetic protecting properties. Magnesium has the lightest weight of the structural metals. Its weight is approximately 60% lighter than steel and 60% lighter than aluminium [3]. Magnesium is usually used to alloy Al for weight reduction since it has lower density than Al. Pratiwi and Utami [4] investigated the influence of ageing process on the microstructure and hardness of as-cast and heat-treated Al-9Zn-5Cu-4 Mg alloys using SEM analysis. The outcome of the SEM analysis indicated the precipitates spread presence over the dendrite with the second phases (Mg₃Zn₃Al₂,

* Corresponding authors at: Mechanical Engineering Department, Air Force Institute of Technology, Kaduna, Nigeria (I.M.B. Omiogbemi).

E-mail addresses: adeleke.kunle@ymail.com (A.A. Adeleke), omiogbemi1@gmail.com (I.M.B. Omiogbemi).

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Cu_2FeAl_7 , CuAl_2 , and CuMgAl_2) in as-cast Al-9Zn-5Cu-4 Mg alloy presence. The aluminium alloy toughness was reported to be affected as a result of the presence of all these second phases while the hardness value of the Al-9Zn-5Cu-5 Mg heat treated cast alloy was said to be impaired due to the presence of MgZn_2 . Girisha and Sharma [5] investigated the magnesium addition influence on the microstructure and mechanical properties of Al4Cu alloys. The Al4CuMg (Mg added in the range of 0.5 to 2% at 0.5% interval) alloy samples were prepared through gravity die casting and then subjected to T6 type heat treatment for 5 h at 175 °C. Using optical microscopy coupled with the utilization of image analysis software for grain size measurement and dendrite arm spacing, the effects of Mg addition and ageing treatment were studied.

Brinell hardness tester and Universal testing machine were employed in the determination of the hardness value and tensile strength of the samples. The microstructural analysis result showed that the Mg addition at 2% reduced the grain size and dendrite structure by 20 and 21.52%, respectively. The tensile strength and hardness increased with increase in the amount of Mg. The 2% Mg addition increased the hardness and tensile strength by 25 and 57.9%, respectively. Heat-treated specimens revealed that addition of 1% Mg had more influence on grain refinement and mechanical properties due to smaller grain size. Nwaeju et al. [6] studied the effects of copper addition on the microstructure and mechanical properties of Al-4%Zn alloy. The results obtained revealed that hardness, impact strength and yield strength of the alloys increased with increase in copper content up to 3.0 wt%.

In this study, effort is being made to determine the effects of addition of varying amount of magnesium on the mechanical properties and microstructure of an aluminum-zinc-copper alloy. The alloy samples were prepared using sand casting due to its wide acceptability for the manufacturing and production of complex shapes. More so, among other processes of manufacturing, sand casting is relatively cheap.

2. Materials and methods

2.1. Materials

The materials used include aluminum, copper wire and zinc scraps which were bought from a local metal scrap shop in Ilorin, Nigeria. Magnesium was obtained from Engineering Materials Development Institute (EMDI), Akure, Nigeria. The weight of the scraps was obtained via the electronic weighing balance before they were charged into the furnace

2.2. Preparation of test samples

In order to cast Al-Cu-Zn-Mg alloys, the metal Zn with the lowest melting temperature was charged for melting followed by Mg, Al, and finally Cu as posited by Odusote et al. [7]. The Mg content of the new alloy was proposed to range between 0.5 and 1.5 wt% (at 0.5% interval) based the report of Girisha and Sharma [5]. Based on

the metals melting temperature, the furnace was heated to about 1110 °C for about 90 min melting time. Four different test samples were prepared based on the magnesium variation. The casting procedure (as shown in Figs. 1–3) involved the wooden pattern fabrication, preparation of green sand mould, scrap melting through a diesel-fired bale out furnace, and molten metal pouring into the prepared mould. Subsequently, the molten metal was allowed to solidify and then cooled at room temperature to produce 150 mm long and 15 mm diameter cylindrical shaped samples. The samples were then machined into different standard shapes for compositional and microstructure analyses, hardness and impact tests. Tensile test was also carried out on the samples and the test specimen (Fig. 4) was prepared based on BSEN 10002-1 [8].

Using a muffle furnace, the machined samples from different composition were heat-treated for 2 h at a temperature of 400 °C. The samples were then quenched in water for total cooling. For artificial ageing, the samples were reheated in a muffle furnace at temperature 160 °C for 5 h. After this, fast cooling operation was performed by quenching the samples in water. Mechanical tests which include impact, hardness and tensile strength tests in addition to microstructure analyses were then investigated. The precipitation hardened samples were similar to the final form of the as-cast samples.

2.3. Experimental procedure

2.3.1. Chemical composition analyses

X-ray fluorescence (Model-Olympus delta professional S/N 540723) was used to determine the elemental constituents of the samples.

2.3.2. Tensile test

Tensile test was carried out on the samples by employing a universal tensile testing machine (50 kN/model-FS0AT). Sample with the dimensions as shown in Fig. 4 was loaded until it fractured. The result of the test was obtained on digital controlled computer system connected to the testing machine. The procedure was repeated for all the samples.

2.3.3. Hardness

The hardness test was carried out using a standard Brinell hardness testing machine in accordance with ASTM E18 standard [9]. The mean value of five repeated readings on each sample was used. This is to avoid the possible effects of any alloying element segregation.

2.3.4. Impact test

This was done using Izod impact testing machine (Model-Charpy dimension) based on ASTM E23 standard [10].

2.3.5. Optical examination

Microstructure analysis was carried out using a digital metallogical microscope (Nikon Eclipse ME600). A sequential grinding was carried out using emery paper grade 80, 220, 320 and 600

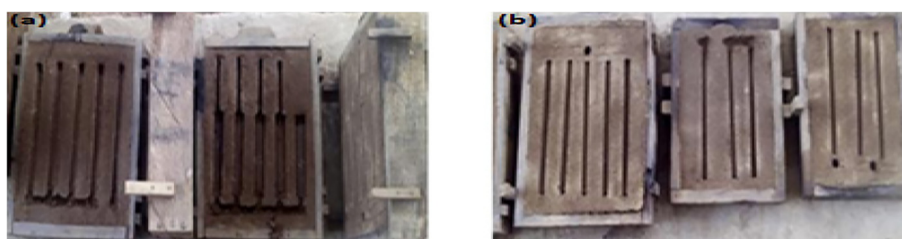


Fig. 1. Mould box for samples: (a) impact and hardness tests; (b) tensile test.

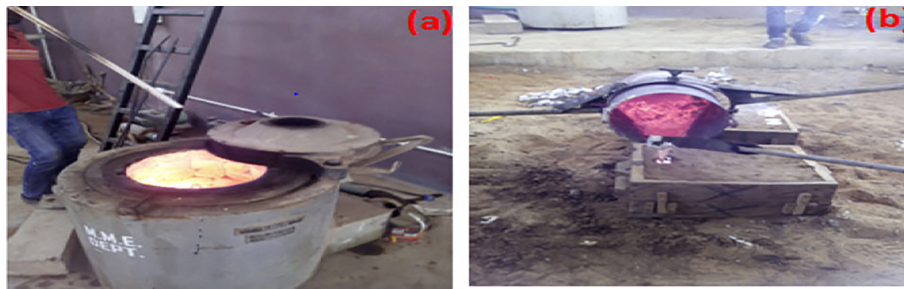


Fig. 2. (a) Stirring of hot metals for homogenization (b) Tong used to separate slag from molten alloy.



Fig. 3. (a) Pouring of molten alloy into mould (b) Solidify cast alloys.

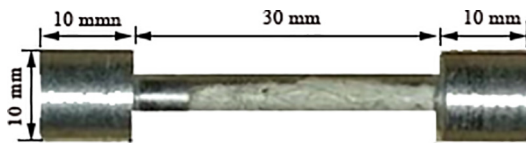


Fig. 4. Tensile test specimen.

µm in succession, while lubricating the sample with water to attain a mirror-like surface. Samples were etched using Keller’s reagent. The microstructure of the samples was examined and photographed at a magnification of 20.

2.3.6. Scanning electron microscope (SEM) and energy dispersive spectrometry (EDS)

Scanning electron microscope (Phenomprox, Netherlands) was also used to carry out the morphology analysis for a comprehensive understanding. The energy dispersive spectrometry (EDS) was used for point and overall elemental analysis across the surface of the samples.

3. Results and discussion

3.1. Compositional analysis

The different weight percentages of the elements present in samples A, B, C, and D are shown in Table 1. The actual percentage of aluminium existing in sample A is 80.33% which is against the expected amount of 80.00%. This implies that the Al scraps contain impurities that might be from other elements. Other samples are observed to also exhibit similar trend where the actual wt.% are a bit more or less than the aimed compositions. The scrap sources as well as the types of scraps used might be responsible for this observed trend in the elemental compositions.

The actual wt.% of magnesium in sample B is 0.001%; sample C is 0.33%, while in sample D is 0.62%. Mg is less than the target wt.% of 0.5, 1 and 1.5% in samples B, C and D, respectively. The Zn ele-

mental composition in samples A, B, C and D (in wt.%) were 17.03, 17.14, 9.70 and 13.99%, respectively. These are less than the targeted value of 18%. The percentage composition of Cu in samples A, B, C and D are 0.77, 1.56, 1.26 and 2.17%, respectively. The value in sample D is above the 2 wt% target, while the values in samples A, B and C are below the expected value of 2%.

According to Odusote et al. [7] and based on analysis, the alloying elements volatilization, especially zinc and magnesium, might have been due to the procedures of melting. This is an indication that there might be the possibilities of obtaining very low Zn amount based on the different sources of the charged Zn scrap. More so, Table 1 displays the % constituents of the elements such as silicon, manganese, iron, nickel and various other elements as they vary across the cast samples. It has been reported that Si presence in Al alloys is beneficial because there is usually an improvement to the Al-Si alloys castability through fluidity increment and solidification shrinkage reduction [11]. Furthermore, Fe is always present in little amount due to the processing and handling of alloy [12]. However, the mechanical properties of the alloys are usually adversely affected when iron content above 0.7 wt% is found in the alloy. This adverse effect is said to be probably eliminated if manganese is present in the alloy. Samples A, B and D has iron content which is greater than 0.7 wt%. Cu content in sample D is greater than 2 wt%, while Cu content for samples A, B and C is less than 2 wt%. Based on this, it would be unsuitable to utilize samples A, B, and C for most structural applications. This is as a result of the poor mechanical properties ensuing from the low cu content as well as the possibility of detrimental effect of higher Fe content in the alloy [12].

3.2. Tensile testing

The ultimate tensile strength (UTS) of both the as-cast samples (A1, B2, C2 and D2) as well as the precipitation hardened samples A2, B2, C2 and D2 can be seen in Fig. 5. The UTS of sample A1 is 145.49 N/mm² in which magnesium is not added. It was observed that addition of 0.001 wt% magnesium (sample B1) in the aluminum alloy increased the UTS of the resultant alloy to

Table 1
Elemental compositions of the Al-Zn-Cu-Mg specimens (wt.%).

Elements	Sample A	Sample B	Sample C	Sample D
Mg	ND	0.001	0.33	0.62
Al	80.33	80.16	87.60	80.05
Si	ND	ND	ND	1.16
P	ND	ND	ND	0.04
S	0.04	0.10	ND	0.60
V	ND	ND	ND	ND
Cr	0.02	0.02	0.01	0.02
Mn	0.08	0.08	0.06	0.10
Fe	0.92	0.91	0.68	0.94
Ni	0.02	0.02	0.01	0.02
Cu	0.78	1.56	1.26	2.17
Zn	17.03	17.14	9.70	13.99
Sn	0.01	0.01	ND	0.01
W	0.75	0.02	0.34	0.23
Pb	0.01	0.04	0.01	0.06

*ND – Not detected.

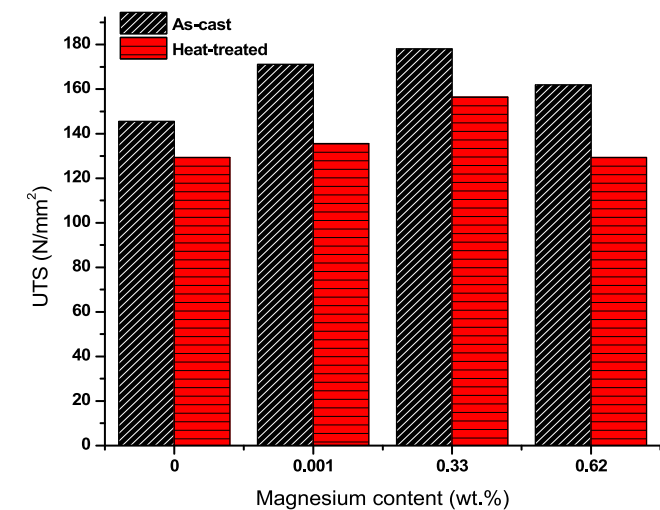


Fig. 5. The influence of variation in wt.% Mg on UTS.

171.06 N/mm² as shown in Fig. 5. With 0.33 wt% of magnesium added in sample C1, UTS increased to 178 N/mm² as against 171.06 N/mm² in sample B1. When 0.62 wt% magnesium was added as observed in sample D1, the UTS decreased to 162 N/mm². Therefore, when manganese content becomes higher in conjunction with the existence of zinc, magnesium and other alloying elements, tensile strength of resultant alloys A1 and D1 is said to be affected negatively. This finding is in agreement with Girisha and Sharma [5] findings, which stated that for 1% of magnesium, the alloy exhibited maximum strength in terms of UTS. In terms of the precipitation hardened samples, sample B2 has UTS of 136 N/mm² compared with the UTS of samples C2 (156 N/mm²) and D2 (199 N/mm²). Higher UTS was displayed in precipitation hardened sample D2 when compared to the as-cast sample D1. This may be as a result of coherent precipitate presence which caused the mechanical properties to be improved [9]. More so, the obtained results of Sample D2 is in line with the studies of Isadore et al. [13], Du et al. [14] and Kaya et al. [15] where the resultant effect of high ultimate tensile strength was caused by high number of grain boundaries for the age-hardened samples.

Fig. 6 shows the percentage elongation of as-cast samples A1, B1, C1, D1 and precipitation hardened samples A2, B2, C2 and D2. The addition of 0.001 wt% magnesium increases the elongation from 2.48% to 5.25% in sample B1. Peak value of 5.25% was achieved with the addition of 0.001 wt% magnesium in sample

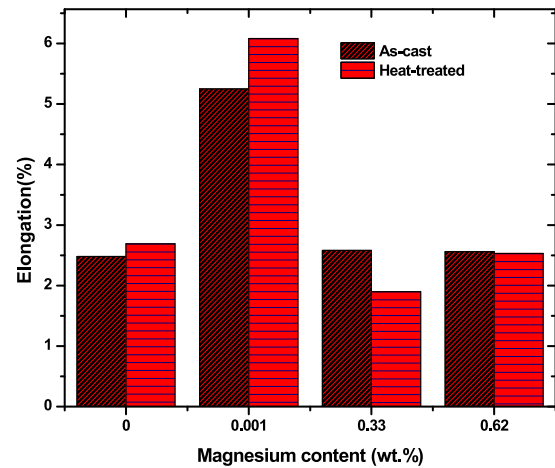


Fig. 6. The influence of variation in wt.% Mg on percentage elongation.

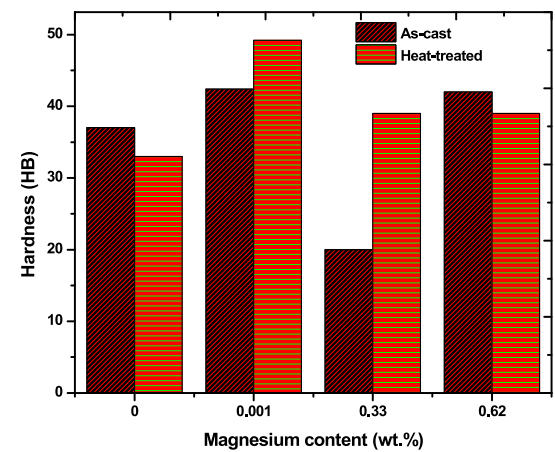


Fig. 7. Variation of Brinell hardness value with increase in Mg content.

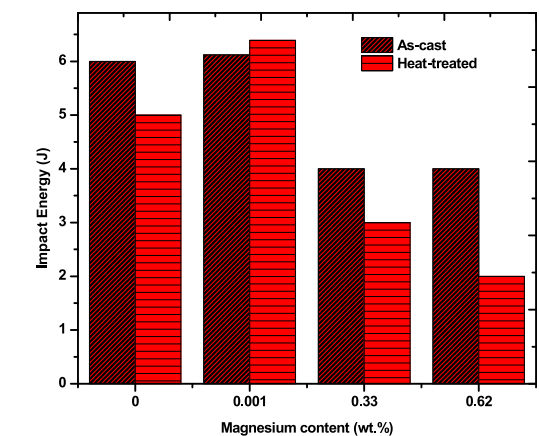


Fig. 8. Influence of variation in wt.% Mg on impact energy.

B1, while elongation falls to 2.56% with the addition of 0.62 wt% magnesium in sample D1. The precipitation hardened samples C2 and D2 have a lower elongation of 1.90% and 2.53%, respectively, compared to the as-cast samples C1 and D1. Samples A2 and B2 have a better elongation than A1 and B1, which indicate better ductility of the heat-treated sample. As evident from the results

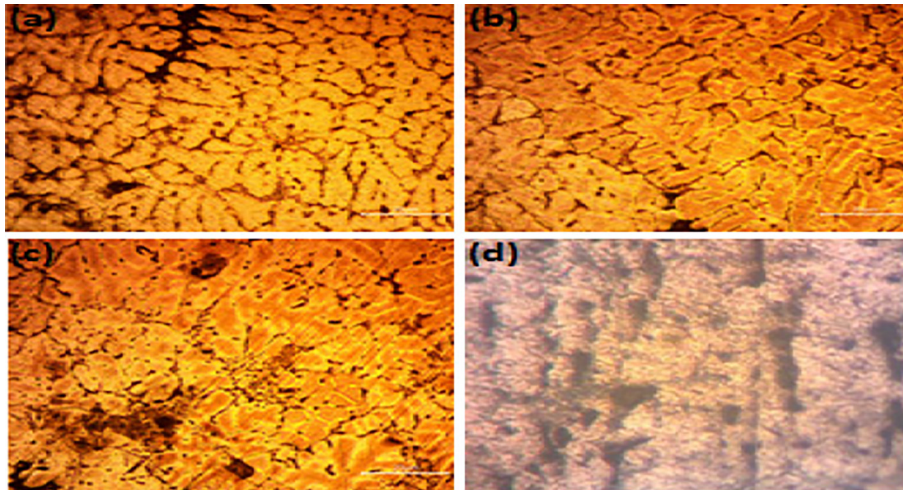


Fig. 9. Optical micrographs of As-cast Al18Zn2Cu alloy: (a) 0.001% magnesium, (b) 0.33% magnesium, (c) 0.62% magnesium, (d) no magnesium content.

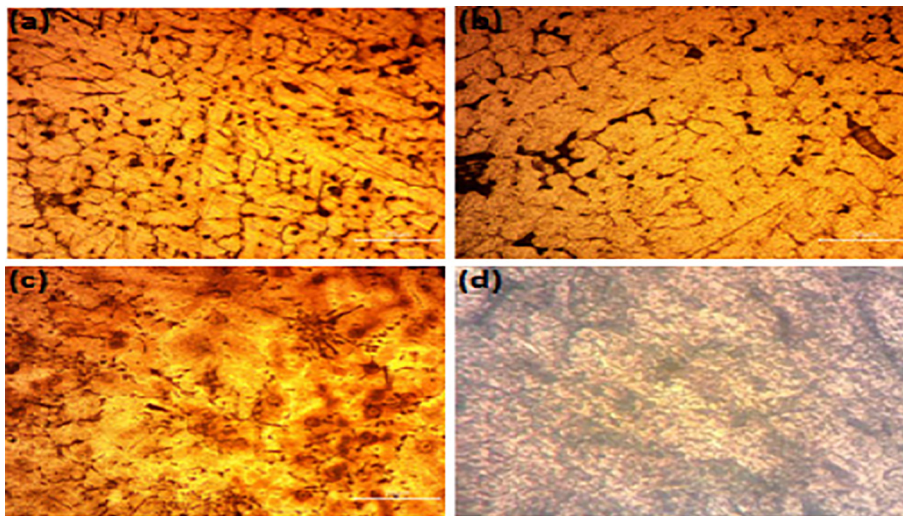


Fig. 10. Optical micrographs of heat-treated Al-18Zn-2Cu alloy (a) 0.001% magnesium, (b) 0.33% magnesium, (c) 0.62% magnesium (d) no magnesium content.

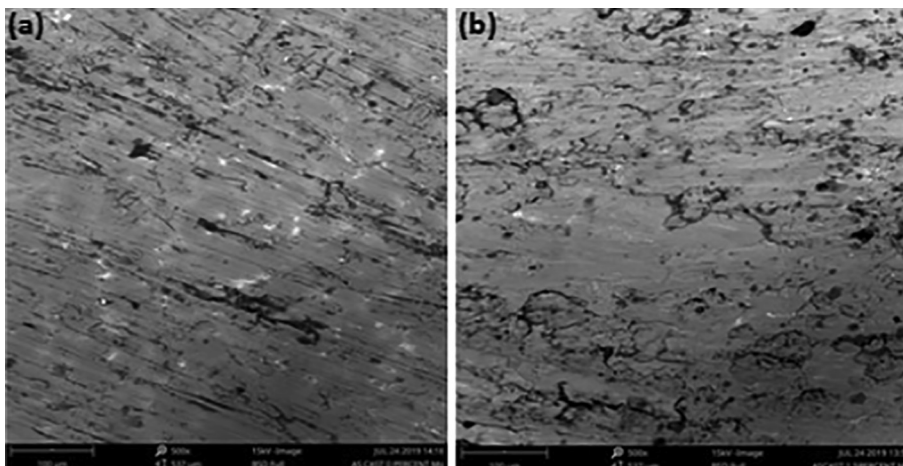


Fig. 11a. SEM micrograph of As-cast sample (a) A1 (b) D1 alloy.

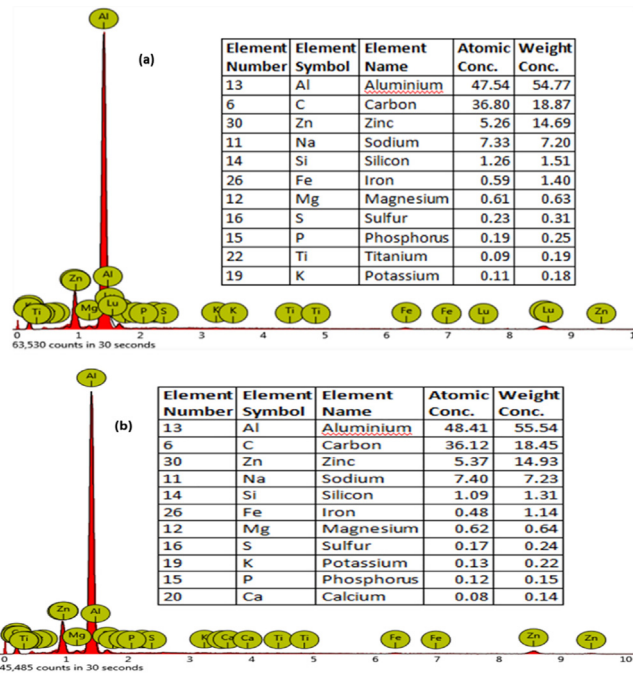


Fig. 11b. Microanalysis report by Energy-Dispersive Spectroscopy (EDS) of samples: (a) A1 (b) D1 alloy.

shown in Fig. 6, magnesium does have less impact on the elongation of aluminum alloys [16].

3.3. Hardness test

The inclusion of 0.001 wt% magnesium to the Al-Zn-Cu alloy (Sample A) led to an increase in hardness from 37 to 42.40 HB as shown in Fig. 7. However, with the addition of 0.33 wt% magnesium (Sample C), which gives 20 HB, this difference was significant. The addition of 0.62 wt% magnesium (Sample D) yielded a rise in hardness values to 42 HB. The high yield in hardness value may be as a result of the increment in manganese content amount present in the alloy. Also, increase in magnesium may form some metallic that in turn gives a higher HB number. It was also observed that the more the magnesium addition, the lower the hardness value. This could be attributed to the fact that the density of magnesium is lower than aluminium. The result agrees with the observation of Agarwal et al. [16] where a slight reduction in

hardness value was observed when the magnesium content is increased. The higher hardness of as-cast alloy samples A1 and D1 could be attributed to micro segregations present in the Al-Zn-Cu-Mg structure that embrittled the alloy. It has been established that the smaller the grain boundaries, the harder and stronger the metal [17].

3.4. Impact test

Fig. 8 shows the as-cast sample A1 (0.77Cu 17.03Zn 80.33Al), Sample B1 (1.56Cu 17.14Zn 80.16Al 0.001 Mg), Sample C1 (1.26Cu 9.7Zn 87.6Al 0.33 Mg) and Sample D1 (2.17Cu 13.99Zn 80Al 0.6 Mg), and precipitation hardened samples A2, B2, C2, and D2. However, the highest impact energy was observed with Sample B based on the Zn and Cu contents in the sample. Sample B has Zn content that is above than that of sample C and D while sample B has lower Cu than sample D and higher amount of Cu than sample C. This implied that impact energy could be said to be influenced by high amount of Zn and low amount of Cu. As magnesium content is increased in sample C and D, there is a decrease in Zn content and Cu content increases, making the impact energy to reduce. Hence, these samples will be unsuitable for shock load application. Sample B indicates that as-cast aluminium sample impact property can be improved through heat treatment as a result of dislocation movement during the precipitation hardening process of heat treatment. Isadare et al. [13] reported that during the age-hardening of the alloy, impact energy is a function ductility which will increase as the ductility increases.

3.5. Microstructural examination

3.5.1. Optical microscope

Fig. 9 displays the optical micrographs of the as-cast alloy samples. The microstructure of the samples mainly consists of the primary crystallized alpha-Al phase (yellow region) and secondary crystallized eutectic region (dark region) [5]. Fig. 9(a) displays the microstructure of as-cast sample with addition of 0.001 wt% Mg which indicates micro-segregation of MgZn₂ in aluminum matrix while Fig. 9(b) displays the as-cast sample microstructure with addition of 0.33 wt% Mg. Uniformly distributed fine grains of MgZn₂ phase was revealed in the microstructure of the aluminum matrix. When 0.62 wt% of Mg was added to the alloy, a non-uniformly distributed MgZn₂ phase was revealed in the microstructure of the alloy as depicted in Fig. 9(c).

Fig. 10 (a-d) shows the microstructures of precipitation-hardened samples. The samples show finely spread precipitates

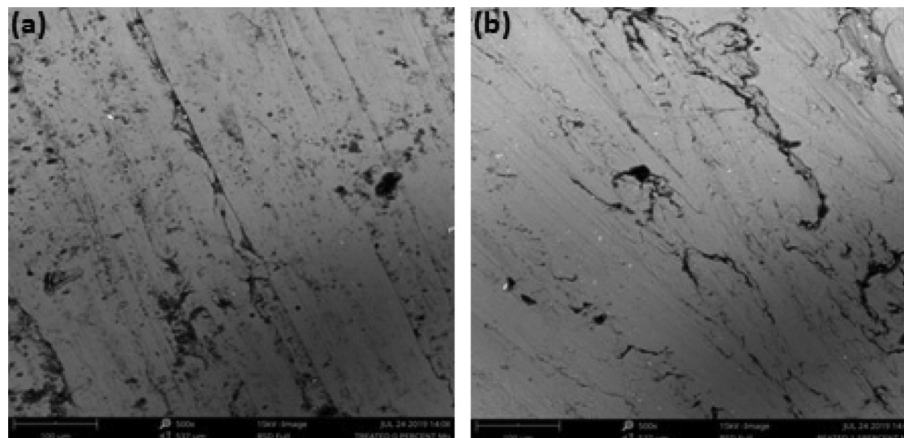


Fig. 12. SEM micrograph of heat-treated samples (a) A2 (b) D2 alloy.

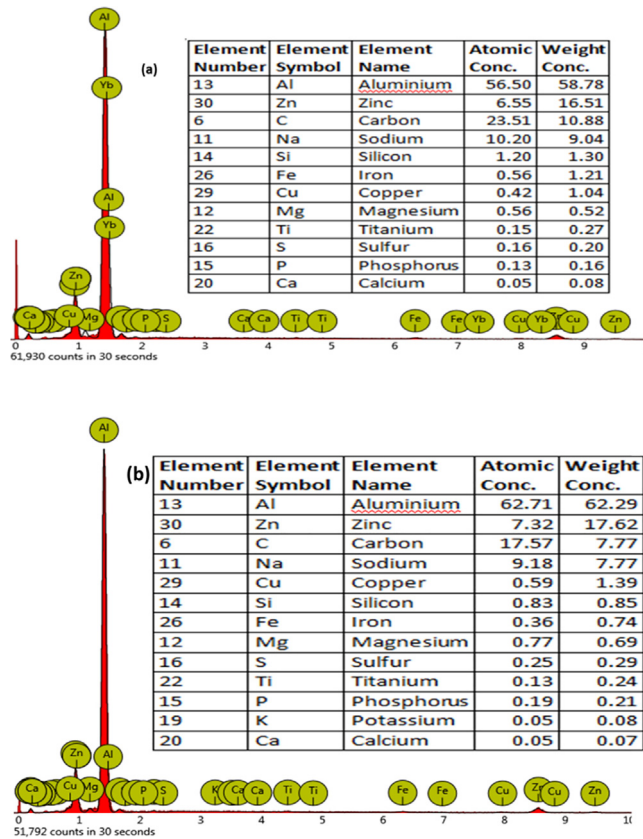


Fig. 13. Microanalysis report by Energy-Dispersive Spectroscopy (EDS) of Heat-treated samples (a) A2 (b) D2 alloy.

of MgZn₂ in aluminum matrix. The presence of distributed MgZn₂ precipitate corresponds with the studies of Salamci [18] and Du et al. [14]. These studies revealed that the formation of MgZn₂ intermetallic phase in the structure could have resulted from Al-Zn-Mg-Cu aging heat treatment. Based on the study on eutectic structures evolution in Al-Zn-Mg-Cu alloys by Fan et al. [19], several coarse intermetallic phases which include MgZn₂, Al₂Mg₃Zn₃, Al-Cu-Mg, Al₂Cu, Al₇Cu₂Fe, Al₁₃Fe₄ and Mg₂Si were revealed. It was reported that they can be formed below the solidus line when as-cast 7000 series aluminum alloys start to solidify consequently to the solute redistribution of metals. It was also revealed that almost all of eutectics have dissolved into the matrix after the heat treatment of the alloy in Fig. 10, but the grain size is obviously increased at the same time [20].

3.5.2. Scanning electron microscopy

Fig. 11(a and b) shows the SEM micrograph of as-cast samples A1 and D1. The micrographs are characterized by the presence of alpha and beta phase. The darker phases (black phase) are the α-phase while the finer phases (lighter phase) are the β-phase [5]. The as-cast sample in Fig. 11a shows the presence of Zn and Cu clusters precipitated along the Al matrix. Fig. 11b shows a coarse precipitation of Cu and Zn clusters in the matrix and micro-segregation of MgZn₂ along the grain boundaries [7]. The holes in the surface demonstrate the second phase precipitations presence that were segregated from the surface through etching and polishing processes [4]. This precipitation forms the second phase that contained dendrites in the grain boundaries. MgZn₂ and Mg₃Zn₃Al₂ are the probable second phases that may be formed. The second phases presence affect the aluminium alloy toughness and the hardness value, strength and impact values are weakened due to

MgZn₂ presence. Fig. 11(a, b) shows microanalysis report by energy-dispersive spectroscopy (EDS) of as-cast samples A1 and D1.

Fig. 12(a, b) shows the SEM micrograph of heat-treated samples A2 and D2. Fig. 12a consists of finely dispersed precipitates of MgZn₂ well distributed at grain boundaries in the matrix which serves as reinforcement within the sample, while Fig. 12b consists of less cluster of Zn and Cu precipitated within the matrix, which is homogeneously dispersed in the aluminum matrix, hence shows the effect of heat treatment on the samples [7]. The presence of MgZn₂ precipitate that is homogeneously distributed agrees with the results of Du et al. [14] and Salamci [18]. It was found that the Al-Zn-Mg-Cu alloys age-hardening resulted into the development of MgZn₂ intermetallic phase in the alloys structure. In this study, the micro-segregation removal after annealing and age-hardening processes agrees with the findings of Guo et al. [21]. It was reported that solution treatment reduces the degree of micro-segregation in 2024 wrought aluminum alloy. Fig. 13(a, b) shows microanalysis of the heat-treated samples 80Al-18Zn-2Cu and 78.5Al-18Zn-2Cu-1.5 Mg.

4. Conclusion

The effects of magnesium inclusion in Al-Zn-Cu alloys on the microstructure and mechanical properties were studied. The utmost UTS and hardness values are 178.038 N/mm² and 42.49 HB, respectively. These were attained when 0.33 wt% Mg and 0.001 wt% was added into Al-Zn-Cu alloy and Al-18Zn-2Cu alloy, respectively. Hardness value declined with an increase in magnesium content. Heat treatment generated the presence of embrittled phase MgZn₂ within the dendrites, and it influenced the strength of the developed alloy significantly. Finally, the developed alloy can find application in the automobile industries.

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CRedit authorship contribution statement

J.K. Odusote: Conceptualization, Supervision, Validation. **A.A. Adeleke:** Writing - original draft, Software, Investigation, Validation. **S.A. Muraina:** Data curation, Writing - original draft. **P.P. Iku-banni:** Writing - reviewing & editing, Validation. **I.M.B. Omiogbemi:** Writing - review & editing, Visualization.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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