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A Study on the Effect of Mg and Ag Additions and Age Hardening Treatment on the Properties of As-Cast Al-Cu-Mg-Ag Alloys

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***Abstract*—**This study focuses on the effect of the addition of magnesium (Mg) and silver (Ag) on the mechanical properties of aluminum based alloys. The alloying elements will be added at different levels using the factorial design of experiments of 2^2; the two factors are Mg and Ag at two levels of concentration. The superior mechanical properties of the produced Al-Cu-Mg-Ag alloys after aging will be resulted from a unique type of precipitation named as Ω-phase. The formed precipitate enhanced the tensile strength and thermal stability. This paper further investigated the microstructure and mechanical properties of as cast Al–Cu–Mg–Ag alloys after being complete homogenized treatment at 520 °C for 8 hours followed by isothermally age hardening process at 190 °C for different periods of time. The homogenization at 520 °C for 8 hours was selected based on homogenization study at various temperatures and times. The alloys' microstructures were studied by using optical microscopy (OM). In addition to that, the fracture surface investigation was performed using a scanning electronic microscope (SEM). Studying the microstructure of aged Al-Cu-Mg-Ag alloys reveal that the grains are equiaxed with an average grain size of about 50 µm. A detailed fractography study for fractured surface of the aged alloys exhibited a mixed fracture whereby the random fracture suggested crack propagation along the grain boundaries while the dimples indicated that the fracture was ductile. The present result has shown that alloy 5 has the highest hardness values and the best mechanical behaviors.

***Keywords*—**Precipitation hardening, aluminum alloys, aging, design of experiments, analysis of variance, heat treatments.

I. INTRODUCTION

FOR many years, in lightweight materials industry, the process of getting good quality and effective related characteristics of materials is in the developing stage and keeps on progressing over the period of time. For instance, aluminum alloys have been considered useful for applications in transportation, automobiles industry, supersonic aviation, defense, and aerospace applications because of its improved mechanical properties and thermal stability. The alloys have a desirable weight to strength ratio. As such, it has attracted more industrial application. The mechanical properties and performance of these materials are highly improved through alloying and heat treatments. The aluminum alloys are the most popular among the available foundry alloys. The ability to cast aluminum alloys is promoted by its fluidity which is comparable to its low melting point values. However, the sustainability of precipitates of these alloys at elevated temperatures continues to raise a considerable concern. Controlling the coarsening rate of precipitates plays an important role to maintain strength at high temperature. Thus, the main concern of many researches, currently, is to synthesize and develop new aluminum alloys with a stable precipitate at elevated temperatures [1]-[3].

Understanding the relationship between the precipitation behavior and microstructure upon Mg/Ag addition, to increase the strength of such alloys has developed great interest recently. Ringer et al. [4] carried out a comprehensive study to evaluate the precipitation behavior in the ternary alloy (Al-1.7 at %Cu-0.3 at %Mg) and the quaternary alloy (Al-1.7 at %Cu-0.3 at %Mg) with the addition of Ag at two different levels (0.1 at % -0.2 at %). An analysis was done through Atom Probe Field Ion Microscopy (APFIM) and Transmission Electron Microscopy (TEM) techniques. The sequence of precipitation in the ternary alloy was seen to start by formation of Guinier-Preston (GP) zones, θ'' and finally the θ' appeared side-by-side with Ω-phase precipitates. The aging was allowed for 2.5 h. The researchers noted that on the quaternary alloy, rapid zones of GP were formed after 30 s aging. The GP zones were then followed by formation of θ'' at {001}. Further aging of this composition leads to the formation of θ' and an even dispersion of Ω- phase on {111}. Ag plays a role of trapping Mg atoms when added at different levels to the quaternary alloy. When added, there will be formation of Mg-Ag co-clusters providing better sites for a desirable Ω-phase nucleation. Thus, precipitation process of Al-Cu-Mg-Ag alloy was noted to follow a sequence: Supersaturated solid solution (SSSS) → Ag-Mg clusters→ Ω-phase +θ′-phase→ θ-phase [5], [6].

Bai et al. [7] investigated the effect of the addition of Ag at a low Cu/Mg ratio. The study utilized an aging temperature of 170 °C for 0.5 hours of aging time. Addition of 0.54 wt % Ag into cast Al-4.1%Cu-1.45%Mg indicated that Ag has an effect of accelerating the precipitation hardening of Ω-phase. Additionally, the thermal stability of the alloy has been enhanced. Contrary, at longer time, aged casting produced needles with a higher density of S' phase in pre-stretched alloy and the Ω phase as was noted. In addition to that Qiong and Wawner [8] have studied the relation between Cu/Mg ratio with two different levels of Cu addition (3.2 - 4) wt % in (Al-0.45 wt %Mg-0.4 wt %Ag) alloy and precipitations phase, it was clearly noted that the Ag addition increases Ω-phase

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formation in the high Cu/Mg ratio alloy. In contrast low, Cu/Mg ratio alloy could lead to σ-phase formation. One of the unique properties of the σ-phase is that it is an equilibrium phase with a high melting temperature and that leads to further thermodynamic stability at high temperatures.

Recently the Concorde gained popularity in supersonic commercial aviation. It was first implemented by airways in France and Britain. This alloy designated as 2618A has a composition of (Al-2.2%Cu-1.5%Mg-1%Fe-1%Ni-0.2%Si). This alloy is considered for several applications since the precipitate in 2618A maintains stability even at very high temperature. More also, the alloy has excellent mechanical properties when compared to other aluminum alloys such as 7075-T6 and 2024 used in the aerospace field [12]. Regardless of the excellent properties of the Concorde, economically, this alloy failed since only 100 passengers could be accommodated in the plane and the distance traveled was also a limiting factor.

Considering the limiting factors, an aluminum alloy designated as Al-Cu-Mg-Ag was proposed in replacing 2618A and other Al commercial alloys used in the aerospace including 7075-T6 and 2024-T6. The proposed system has excellent features including good mechanical properties and excellent thermal stability [5], [6]. In recent days, there have been publications pertaining to the microstructure and the alloy's mechanical properties [17]-[19].

The Ω-phase of the precipitate plays a vital role in achieving the excellent mechanical properties and thermal stability [5], [6]. According to a study by Ringer et al. [4], the precipitates sequence was explored using a TEM. The results of this study showed that the habit planes of the Ω- precipitate has matrix designated as {111} α. Lumely and Polmear [13] carried out a study regarding the precipitate's morphology. The results showed that the Ω phase comprises of an orthorhombic structure with a shape similar to a plate. Even though researchers say that the Ω phase composition is still in question, this research revealed that the precipitate has a composition similar to the one for Al_2Cu while interfaces of α/Ω for Ag and Mg were also detected.

Liu et al. [14] carried out a comprehensive study which focused on the creep behavior an aluminum alloy comprising of Al-5.33Cu-0.79Mg-0.48Ag-0.30Mn-0.14Zr. Based on this work, the researchers carried out an experimental test on three alloys at a stress and temperature of 150-300 MPa and 150 °C respectively. The results of this experiment revealed that the alloys maintained a steady creep at stresses of 300, 250 and 200 MPa at time of 0.03, 0.06, and 0.12 hours respectively. The study also compared the experimental results for aluminum alloy designated as Al 2024 with a conventional aluminum alloy with composition designated as Al-5.6Cu-0.45Mg-0.45Ag-0.3Mn-0.18Zr under the various condition of age hardening fully hardened and underaged (T6) conditions. It was also found that the active creep test parameters considered were 150 MPa - stresses and 300 ^{o}C with respect to the test temperature, the creep rate was low for the alloys in underaged condition. Nevertheless, the alloy Al 2024 exhibited secondary creep at 200 and 400 hrs in the underaged test condition. On the other hand, the alloy tested experimentally had a lower amount of Ag hence did not exhibit any secondary creep when tested under the two aging conditions. Considering the results of creep behavior under different aging conditions in this research, the alloy tested experimentally had a lower amount of Ag at various instances of creep. At a creep condition of about 200 MPa, 130 ^{o}C and time of 20000 hrs, the creep rate was found to be around 0.4 and no secondary creep was observed throughout the test [10].

Al-Obaisi et al. [15] study eight distinct compositions of Al-Cu-Mg-Ag alloy and indicate that the hardness values were affected significantly as the alloy weight percentages varied. Additionally, the study revealed that at a temperature of 190 ^{o}C, the hardness values improved with a realistic duration of aging and could be considered attractive to various industrial applications. Furthermore the study involved the construction of a statistical model amongst different values of hardness and other process inputs including duration, temperatures of aging and variation in weight percentages of the elements used in the alloy.

The studies have unanimously reported that there are effects on mechanical properties of Al-Cu alloy when Mg/Ag added in the composition. An optimum value of Mg and Ag composition in the alloy and age hardening was studied [1], [17]. From these studies, it is evident that the addition of traces of Mg and Ag in the Al-Cu alloy has effects on its mechanical properties. The importance of the addition of Mg and Ag is to alter the process of hardening during heat treatment, hence the strength of the alloy [15], [17]-[19]. These studies have demonstrated the best practice of age hardening of the Al-Cu alloys. Therefore, they can be used as a basis for this research to greatly investigate the alloy's behavior under different hardening, aging, and application conditions.

II. EXPERIMENTAL WORK

Table I presents the nominal chemical composition of the present alloys with different concentrations additions. The alloying elements were added at different levels using a factorial design of experiments of 2^2, the two factors are Mg and Ag at two lev els of concentration; 0.2and 0.5 wt% Mg, 0.3 and 0.6 Ag wt%. The analysis was carried out using arc and spark excitation metal analyzer Spectromax from Spectra Company.

TABLE I
CHEMICAL COMPOSITION OF THE INVESTIGATED AS-CAST AL –CU –MG –AG ALLOY WT%*

Alloy No.	Cu	Mg	Ag	Cu / Mg	Mg / Ag	Al
1	2.71	0.24	0.33	11.30	0.72	Balance
2	2.75	0.52	0.33	5.30	1.57	Balance
3	2.89	0.23	0.65	12.60	0.35	Balance
4	2.96	0.47	0.65	6.30	0.72	Balance
5	2.84	1.11	0.64	2.6	1.73	Balance

The experimental alloys were prepared at different stages by using pure Al (99.97%), Cu (99.9%), Ag (99.9%) and Al-

20%Mg master alloy, melted and refined in a graphite crucible at 720 °C and then cast in steel permanent mold to produce rods of 85 mm in long and 15 mm in diameter, then the casted alloys were homogenized. The casted rods were solid solution treated (SST) at 520 °C for 8 h, water quenched then aged at 190 °C for different thermal exposure time (0.16, 0.5, 1, 2, 5, 10, 24, 48 and 72 h) followed by water quenching after each aging condition. The multi-stages SST modifies the mechanical properties of cast structure of Al - Cu alloys [9], [11], especially in the high temperature ranges. Priya et al. [11] observed that the sequence and rates of kinetic transformations, of a three-stage (420 °C for 10 h, 470 °C for 4 h and 480 °C for 15 h) homogenization for 7xxx aluminum alloys series namely Al-Zn-Cu-Mg-Zr, is highly suggested to enhance the mechanical behavior of the certain alloys. Hence, two-step solution treatment was performed at 520 °C for 8 h, The treatments were carried out in a constant temperature drying oven (CARBOLITE furnace), The five alloys were aged in a mixture of salt bath consisting of 50% potassium nitrate, KNO$_3$ (Lobachemic) and 50% sodium nitrite, NaNO$_2$ (Pharma) grade. The mixture was placed into a graphite crucible and heated in the furnace for 5 h before the aging process starts at 190 °C regular temperature drying oven. The tensile properties at room and elevated temperatures were investigated by using Instron universal electronic testing machine model 3385H, with data acquisition system, at a constant speed of 1 mm/min corresponding to initial strain rate of 10^{-4} s^{-1}. Engineering stress-strain diagrams were plotted and corrected for the machine compliance. Specimens for tensile test samples were machined from the solution treated rods. The tensile samples were designed according to the ASTM E8 as shown in Fig. 1, with standard cylindrical tensile specimens gage length (Lo)/gage region (Do) = 4. SEM was used to reveal the fracture morphology.

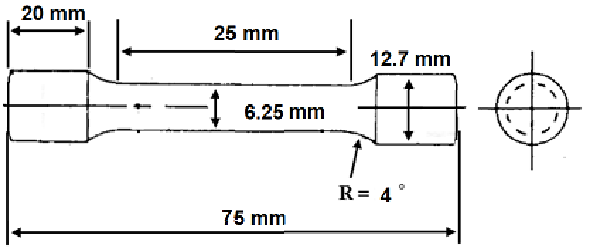


Fig. 1 Tensile test samples diminutions

III. RESULTS AND DISCUSSION

A. Microstructure

In this study five different compositions of Al-Cu-Mg-Ag alloys, are cast, homogenized, and age hardened at different aging time durations. Microstructures and mechanical properties of an Al-Cu-Mg-Ag alloy aged for different period at 190 °C were characterized in the present work by means of Vickers hardness tests (HV5), where the results are presented in Table II.

TABLE II
HARDNESS VALUES (HV_5) - THREE AGING CONDITIONS OF ALL ALLOYS WITH ITS TIME AT ISOTHERMAL AGING TREATMENT – 190 °C

Alloy #	Under Aged (HV5)	Time (Hrs)	Peak Aged (HV5)	Time (Hrs)	Over Aged (HV5)	Time (Hrs)
1	92	5	93	10	90	24
2	87	1	108	2	102	5
3	96	2	109	2	103	2
4	103	5	115	10	105	24
5	116	2	130	5	116	10

The changes in the properties of the present alloys containing Al, Cu, Mg, and Ag are dependent on the addition of suitable materials and the quantity of the same. An analysis of hardness values as illustrated in Fig. 3 reveals that the addition of Mg concentration in the alloy decreases at the time when the alloy attains the peak hardness. This is due to the fact that Mg has the tendency of accelerating the precipitation process [1] which thus leads to low precipitates coarsening rate as shown in Alloy 5. This alloy has the highest value of hardness as a function of aging. The reason behind this is because the elements making up the alloy has high ratios as compared to Alloy 1. These high amounts delay the achievement of the peak hardness as well as greater precipitation, which increases the strength of the alloy. Higher amounts of Cu require longer homogenization time in the alloy as compared to other elements. This is evident in Alloy 4 whereby the peak hardness is achieved within a short time and a considerable amount of precipitation is possible. Addition of Ag weight presents a suitable condition of treatment of the alloy whereby there is a fine dispersion of the precipitate causing a higher coarsening resistance rate in the material alloy. However, there is a significant loss of strength in the material due to the fact that the material thickens and loses the coherence of the precipitate after some time of aging, which is clear in Alloy 2. Addition of Ag/Mg has a significant effect on the achievement of the peak hardness as shown in Alloy 1. As pointed out Ag increases precipitation, however the level of material strength is reduced significantly. As for the Mg portion, the alloy precipitates were formed and during heat treatment, the precipitate formed is smaller and widely scattered. However, as seen in alloy 5 that increase in Ag/Mg ratio in the alloy achieves better stability in hardness with the initial delay in peak hardness achievement. Moreover, the effect of the Mg/Ag ratio on the hardness values is evident as shown in alloys 2 and 5. As observed from Figs. 3 & 5, Alloy 5 achieves the highest hardness value as aging proceeds. This is due to the fact that there is more precipitation. A high stable balance is also achieved. As for the alloy 2 and 3, the peak hardness value is achieved at a shorter aging duration as compared to alloy 5. This is due to the increase in the Mg/Ag co-clusters accompanied by faster nucleation rate at an early aging stage giving a quick peak hardness value [4]. However, a much stable hardness value is achieved in alloy 3 as compared to alloy 2 as age hardening proceeds.

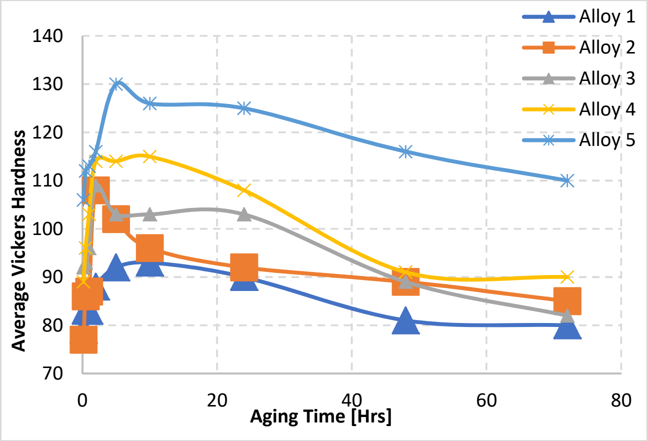


Fig. 3 Vickers hardness values HV5 of alloys 1- 5 after different aging periods of times

OM was conducted, on alloys 1-5, by an Olympus BX60 light microscope. The samples were ground and polished by standard procedures and then chemically etched based on ASTM Standard E 407 by using fresh Keller's reagent (10 ml HNO_3, 6 ml HCl, 4 ml HF, and 190 ml dist. water). The line intercept method was employed to estimate the average grain size. The microstructure of aged Al-Cu-Mg-Ag alloys revealed that the grains are equiaxed with an average grain size about 50 µm, Fig. 4. The cast microstructure has cored dendritic formation with Cu-Mg-Ag content growing progressively from center to edge with an interdendritic distribution of second-phase particles or eutectic. Furthermore, it can be understood from the data in Fig. 4 that large equiaxied grains' distribution with dendritic segregation exists in Al–Cu–Mg–Ag alloy. There are many undissolved intermetallic precipitates formed during solidification expected at the grain boundary as shown in (Fig. 4 (d) - arrow 2) and the distribution of the main elements along interdendritic region varies regularly, the remaining phases are dissolved into the matrix sequentially, grain boundaries become dispersed and all elements become more homogenized compared to the microstructure before the heat treatment, dendrite segregation was observed during increasing the Mg concentration on alloy 2 (Fig. 4 (b) - arrow 1).

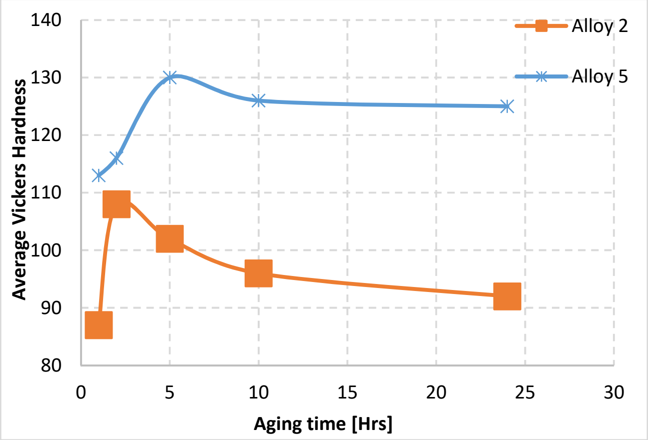


Fig. 4 Vickers hardness values HV5 of alloys 2 and 5 with high ratio between Mg/Ag after different aging periods of times

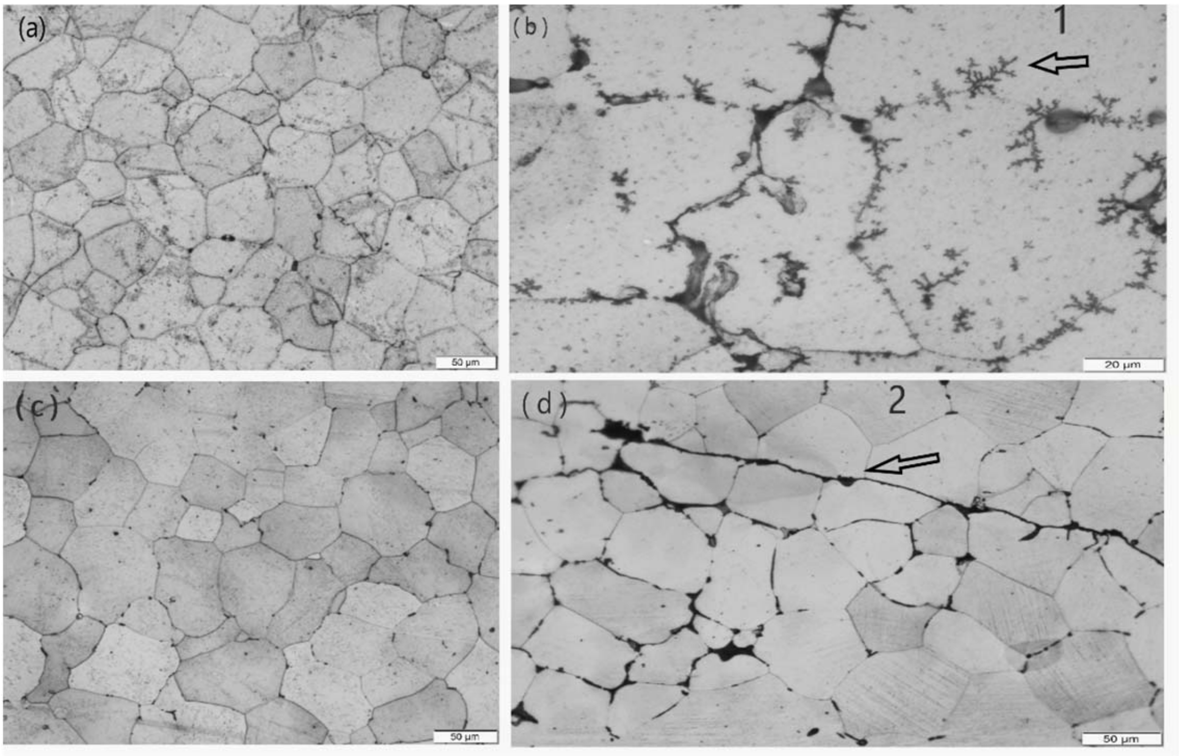


Fig. 4 Metallographic pictures of the present Al-Cu-Mg-Ag alloys at different heat treatments conditions: (a) Alloy 1, (b) & (c) Alloy 2, (d) Alloy 5

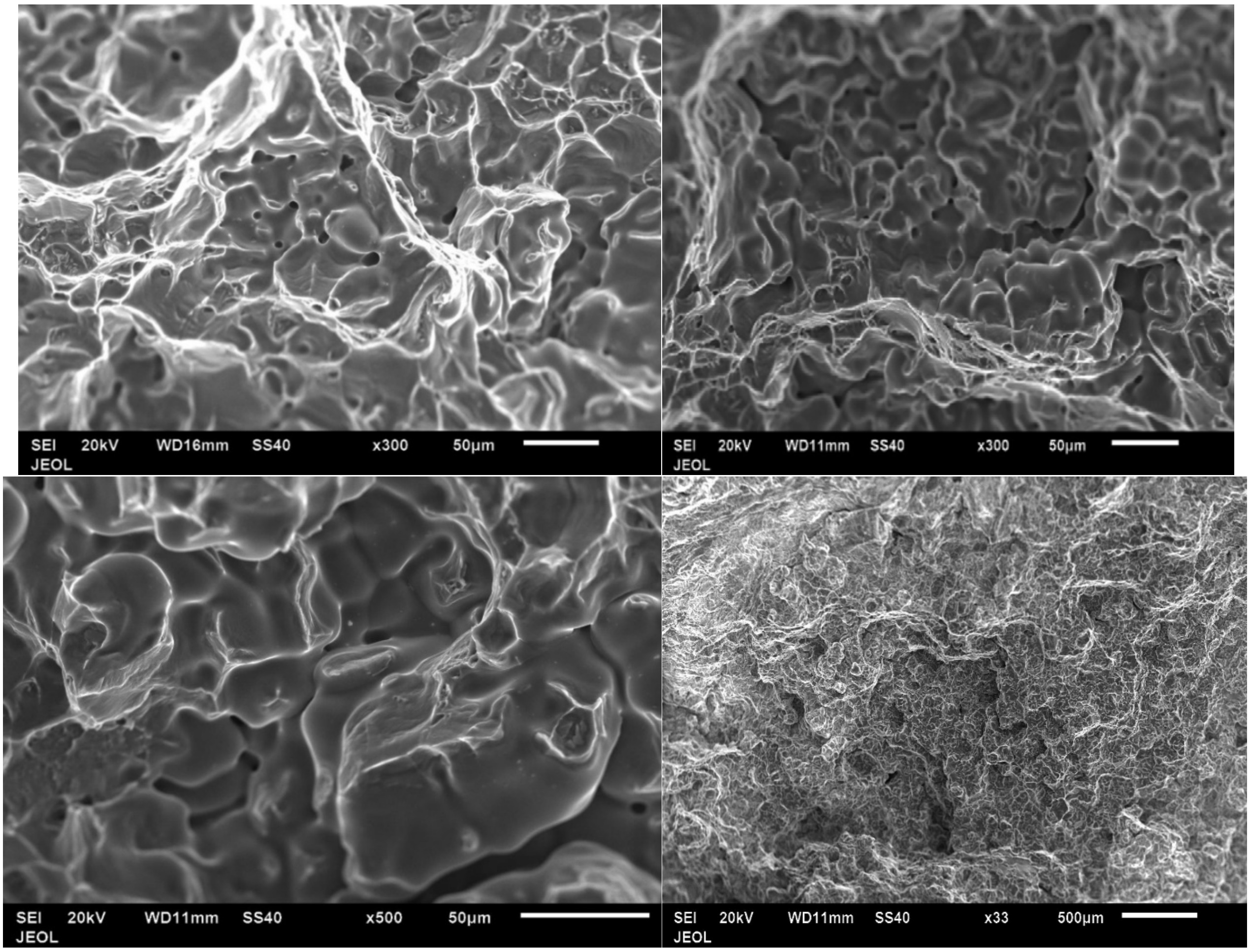


Fig. 5 Secondary electron SEM image of the fractured surface formed by tensile testing of alloy 1 in peak-aged condition at different magnifications

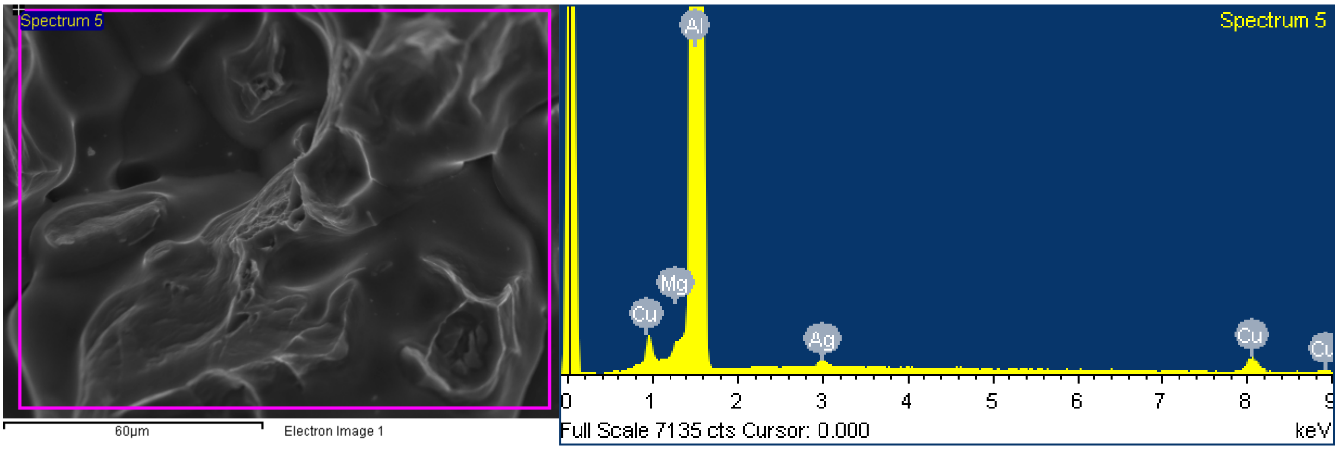
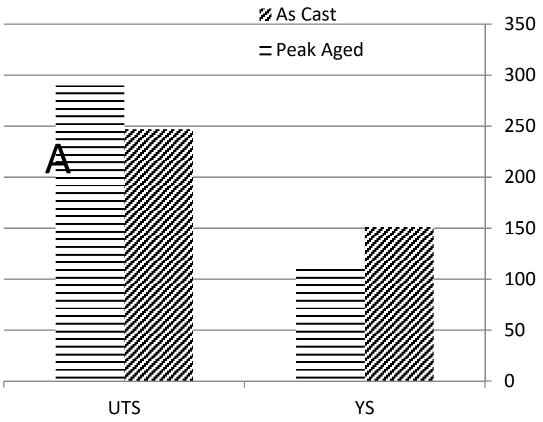


Fig. 6 EDS spectrum from a point analysis taken at a particle from the fractured surface of alloy 1 (Cu: 2.02 wt.%, Mg: 0.33 wt.%, Ag: 0.26 wt.% and Al: 94.06 wt.%)

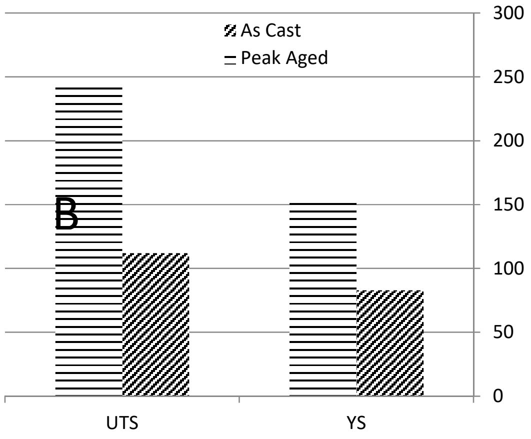
Figs. 5 and 6 indicated that some of the particles distributed on the fractured surfaces were chemically investigated through Energy Dispersion Spectroscopy (EDS) technique. Fig. 6 shows the EDS spectrum of one of the particles distributed on the fractured surface of Alloy 1 tested at room temperature for the peak-aged condition. The EDS spectrum presents clustering of Cu, Mg, Ag and Al atoms suggesting a coarsened phase of the Ω-precipitate [4]. Further, the spectrums show clusters of Al and Cu atoms as well as lower amounts of Mg and Ag atoms. This affirms that the Ω-precipitate phase has actually been coarsened.

B. Tensile Tests

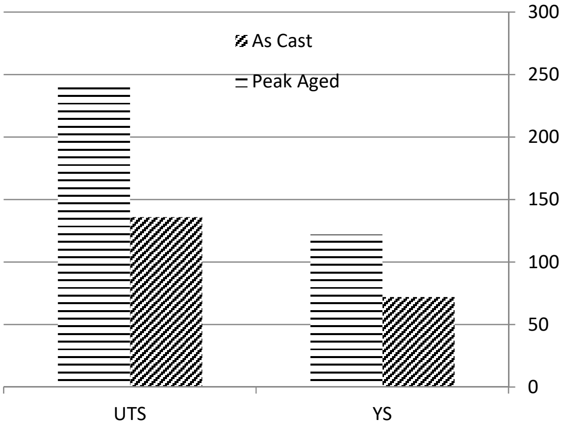
Robinson et al. [16] carried out an experimental tensile test on five alloys at room temperature. The authors reported that this temperature is suitable for many industrial applications coupled with initial strain rate of $5*10^{-4}$ s^{-1}, in solution treated conditions as well as in the peak aged conditions. The fracture surface of five alloys investigation was performed using SEM.



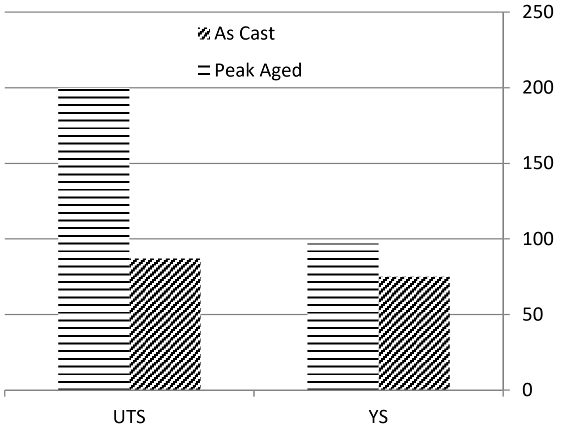
(a) Alloy 1 (2.71% Cu – 0.24% Mg – 0.33% Ag)



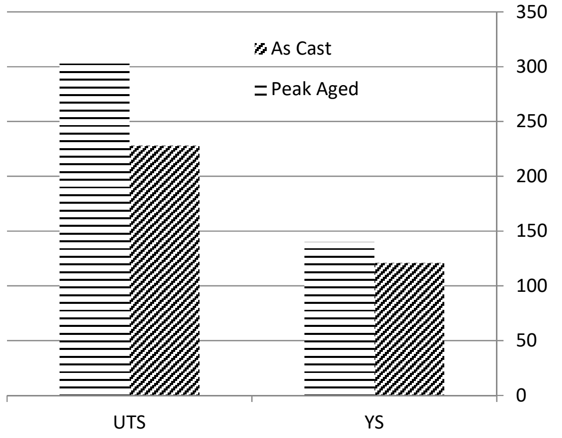
(b) Alloy 2 (2.75% Cu – 0.52% Mg – 0.33% Ag)



(c) Alloy 3 (2.89% Cu – 0.23% Mg – 0.65% Ag)



(d) Alloy 4 (2.96% Cu – 0.47% Mg – 0.65% Ag)



(e) Alloy 5 (2.84% Cu – 1.11% Mg – 0.64% Ag)

Fig. 5 Ultimate tensile strength (UTS) and yield strength (YS) of Alloys 1- 5 at room temperature. after isothermally aging treatment – 190 °C

The data expressed in Fig. 5 indicate that the peak aging condition highly affects yield strength (YS) and ultimate tensile strength (UTS) compared with SST condition for all five alloys at room temperature. As expected, the stress values increase with increasing of Mg weight percentage. For UTS values, Alloy 5 gave the highest values in cast and peak-aged conditions. The reason for this is based on high concentration of Mg and Ag in Alloy 5 as compared to the other alloys. However, Alloy 4 gave the lowest UTS values. It is obvious that the difference between Alloy 4 and Alloy 5 was around 113 MPa at room temperature. For YS values, Alloy 3 gave the highest values while Alloy 4 gave the lowest. The difference between Alloy 3 and Alloy 5 was around 38 MPa at room temperature. Fig. 6 presents various mechanical behaviours of the alloys under this study. As such, Mg addition plays the most significant role to accelerate the hardness progress and improve the UTS values for alloy 3 and 5.

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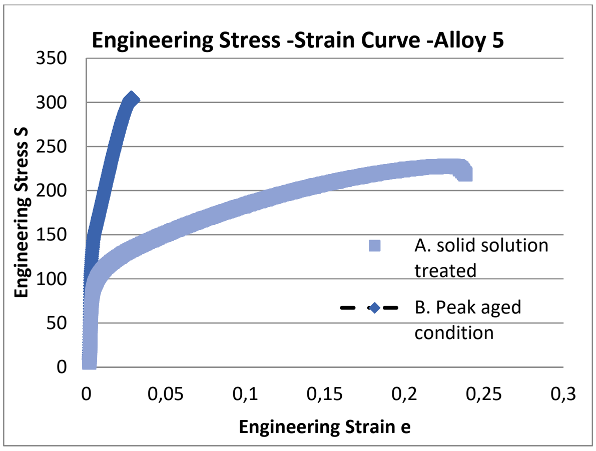

Fig. 6 Tensile properties UTS, YS and percent of elongation (ε %) of Alloy 5 at room temperature for SST and at peak aged conditions and isothermally aged condition at 190 °C

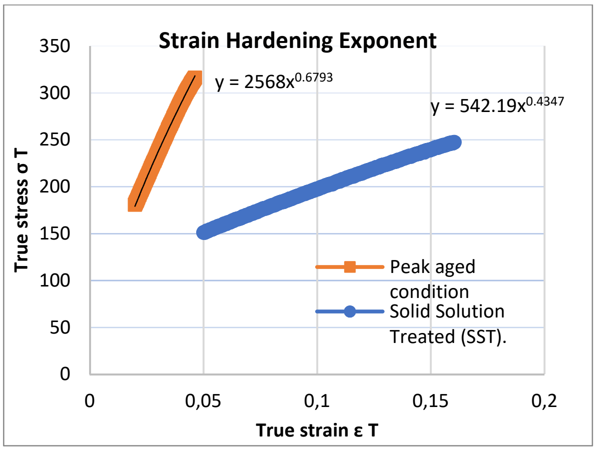

Fig. 7 True stress strain curve - Alloy 5 - for SST and peak aged condition

As illustrated in Fig. 7, an increase in strain hardening coefficient is inversely proportional to the elongation. As a result, the overall strain hardenability increases. This explains the unique mechanical features of alloy 5. Further, the change in strain hardening coefficient between the solid solution treated condition and the peak aged condition depicts that a material exhibits different strain-hardening properties under various treatment conditions.

It is clear that aging at 190 ^{o}C over a given period of time increases the hardness value of an alloy. This is well illustrated in Figs. 6 and 7. On the contrary, ductility of the alloy decreases as the coarsening rate of the Ω precipitates increases.

IV. CONCLUSIONS

The solidification behavior changed by addition of Mg and Ag to Al-Cu-Mg-Ag alloys. Five alloys with different compositions of Al-Cu-Mg-Ag were cast. The alloys were homogenized at different stages. Tensile tests samples were machined from the solution treated rods. These samples were thermally exposed at 190 °C for different thermal exposure periods. After carrying out solution treatment at 520 °C, tensile test was performed at room temperature. Alternating weight percentages of alloying elements had notable effects on the tensile properties. The encouraging mechanical properties of Al-Cu-Mg-Ag make them a potential replacement for 2.xxx series, 7.xxx and other commercial aluminum alloys applied within elevated-temperature applications such as aviation and transportation applications.

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