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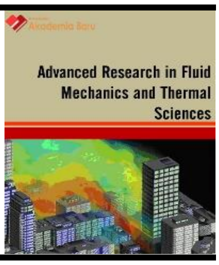

Effect of Isothermal Heat Treatment on Hardness of X7475 Aluminium Alloys

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ABSTRACT

The heterogeneous nature of microstructure in most as-cast alloys has created the unique properties for an ally before heat treatments. The scanty nature of scholarly articles on the effect of heat treatments on the hardness of new alloys produced from recycled aluminium alloys prompts this investigation. In this work, we upgraded from 3 series to 7 series alloys to form a new material. Previous attempts were also silent on the Alcao Green Letter specifications of the 7 series alloys, hence producing a 7475 alloy that may not be within the Alcao specifications. This paper presents the result of preliminary studies on new experimental X7475 Al-alloy that was produced per Alcoa Green Letter specifications, with variations of Zn (4.0 - 5.0 wt.%), Mg (1.00 - 1.50 wt.%) and Mn (0.025 - 0.075 wt.%), then subjected to microhardness probe under a load of 490.3mN (0.05Hv) and 10sec duration after isothermal and isochronous "O", T4 and T6 heat treatments. Heating and cooling rates were controlled during heat treatment processes to investigate the effects on the formation of the $MgZn_2$ and other precipitates on the mechanical properties of the alloy. Peak obtainable hardness value of 140.45 Hv and 134.32 Hv are attained within the T6 and T4 conditions in an alloy of 4% and 5% Zn respectively. The increase in wt.% of Zn favours mechanical properties while reverse is the case for Manganese. This result is an indication of the load bearing capacity of our alloy. It has also demonstrated the success in upgrading the 3 series to 7 series alloys from secondary materials. However, the result could be improved on after composition, heat treatments and production route optimization.

Keywords:
X7475 Al-alloy, Green Letter, civilian transformation, hardness

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1. Introduction

Heat treatment is a process of transfer of heat through solids by conduction, the thermal history of which are central to the formation and retention of precipitates responsible for modification of the behaviour and areas of application of aluminium alloys. The versatility and high strength to weight ratio of aluminium has earned it a place in light weight applications [1]. Microstructure of heat treatable aluminium alloys [2] (mechanical, characterization and peak hardness) are altered by heat

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treatments [5,10-12]. Peng *et al.,* [3] reported a linear relationship between hardness and ageing period in 7xxx alloys, with a possibility of declination at over aged condition. X7475 is a new age-hardenable experimental aluminium alloy of the Al-Zn-Mg-Mn-Cu family, herein probed.

The high chances of acceptability of Electric Vehicles (EVs) may increase the use of 7xxx series as structural materials. The 7 series also find wide applications in airframe structures and light armored carriers [2-8]. The effect of heat treatments on hardness of X7475 experimental alloy, produced by casting from secondary aluminium is of engineering relevance to prepare the alloy for structural applications.

Fine precipitates are formed through coherency strains [4] of $MgZn_2$, regrouping and grain refinement in the α-Al matrix during heat treatments. Not only does $MgZn_2$ regroups, other hardness inhibitors like $CuMgAl_2$, Al_4C_3, Mg_5Al_3 or Mg_5Al_8 [5] competes for solid state transformation, taking the advantage of kinetics in heat energy. Formation of aforementioned precipitates creates secondary dendrite arm spacing that directly influences the mechanical properties or the subsequent homogenization treatment of as-cast products. The dendrites grow equiaxially in multicomponent alloys [6] like X7475.

The movement of interstitial atoms within the lattice is restricted and require heat energy to facilitate the rearrangement, that strengthen heat treatable Al-alloys. In 7xxx alloys, the formation and transformation of hexagonal $MgZn_2$ and body-centered cubic (bcc) $Al_2Mg_3Zn_3$ are responsible for peak hardening in Al-Zn-Mg alloys [3]. Parameters (dwell time, heating and cooling rates and quenching medium) adopted during heat treatments are relevant in distorting the mechanical [7] and microstructure properties of various series of aluminium alloys [8].

Researches on upgrading of experimental alloys from 3xxx to 7xxx are scanty. Wen *et al.,* [9] studied the effect of variation of zinc on the Aging behavior and fatigue crack propagation of high Zn-containing alloys, but the experiment was outside the specifications of the Alcao Green Letter. Lin *et al.,* [10] like [3,5,11] used as received alloys to conduct a study of the effects of pre-treatments on aging formation of precipitates and corrosion resistance in a 7xxx alloy. This gap calls for a study of effects of heat treatments on the mechanical properties of a new aluminium alloy. The intent of this paper is to study the impact of isothermal and isochronous heat treatments on the hardness of X7475 alloys within micro hardness limit.

2. Methodology

Samples of Al-Zn-Mg-Mn-Cu alloy is melt in a 2 kg induction Electric Melting Furnace, model JT0332 using an improvised stainless steel cup as crucible. The alloy was cast in a customized permanent steel mould made from a pipe of Ø18mm X 160mm dissected and clipped together to form a piece. During melting process, pieces of AA 3004 aluminium ingots, made by melting recycled can was introduced first, followed by zinc, copper, manganese and magnesium in that order. Mixing was achieved using Pentec hand mixer, model TAC 1803 set at high speed stirring gear.

Samples in from of rods were allowed to homogenize for 48 hours before cutting. Test sections were taken from the centre of as-cast rods for heat treatments and micro hardness investigations. A non-destructive, Epsilon 1-Malvern Panalytical - Benchtop X-Ray Fluorescence (XRF) Spectrometer was used to determine a typical elemental composition of the new material presented in Table 1. The wt.% of Zn, Mg and Mn varies between (5.0-4.0), (1.00-1.50) and (0.025-0.075) respectively.

Table 1
Elemental composition of X7475 alloys investigated

Compound	Al	Zn	Mg	Si	P	Cr	Mn	Fe	Cu
Conc. (wt.%)	Balance	4.813	2.067	0.441	0.230	0.144	0.648	1.271	0.557

Commercially pure aluminium alloys have less engineering applications due to poor mechanical properties. Aluminium heat treatable alloy are subjected to series of heat treatments to improve on properties. In all, the solution treatment is a primary and key step [10,11] in treatment process. A Carbolite HTF 1800 furnace was used for the heat treatment per modified AMS 2771. Alumina crucible was used as secondary plate to avoid contamination and protect the wall of the furnace from abrasion. Samples of Ø18 mm × 6 ± 0.5 mm height were solution heat treated, annealed and aged per heat treatment profiles shown in Figure 1. Solution heat treatment was done isothermally. Aging was performed per AMS 2750 furnace specifications isochronously. Cooling rates were controlled to allow rearrangement and dissolution of solute atoms into the aluminium matrix during the annealing and artificial aging processes. Due precautions were observed to prevent the furnace from absorbing hydrogen during eutectic melting which may result in hydrogen blistering.

Hardness sample test preparation was per modified ASTM E384-17 and test was performed per ASTM E18. Microhardness investigation was done on a Shimadzu HMV-2, C227-E013, Vickers Microhardness Tester and S/N 163034501261. Load, duration for test and times of test were set at 490.3mN (0.05Hv), 10sec and 9 respectively. Another macrohardness test was done. The result will be presented in later publication.

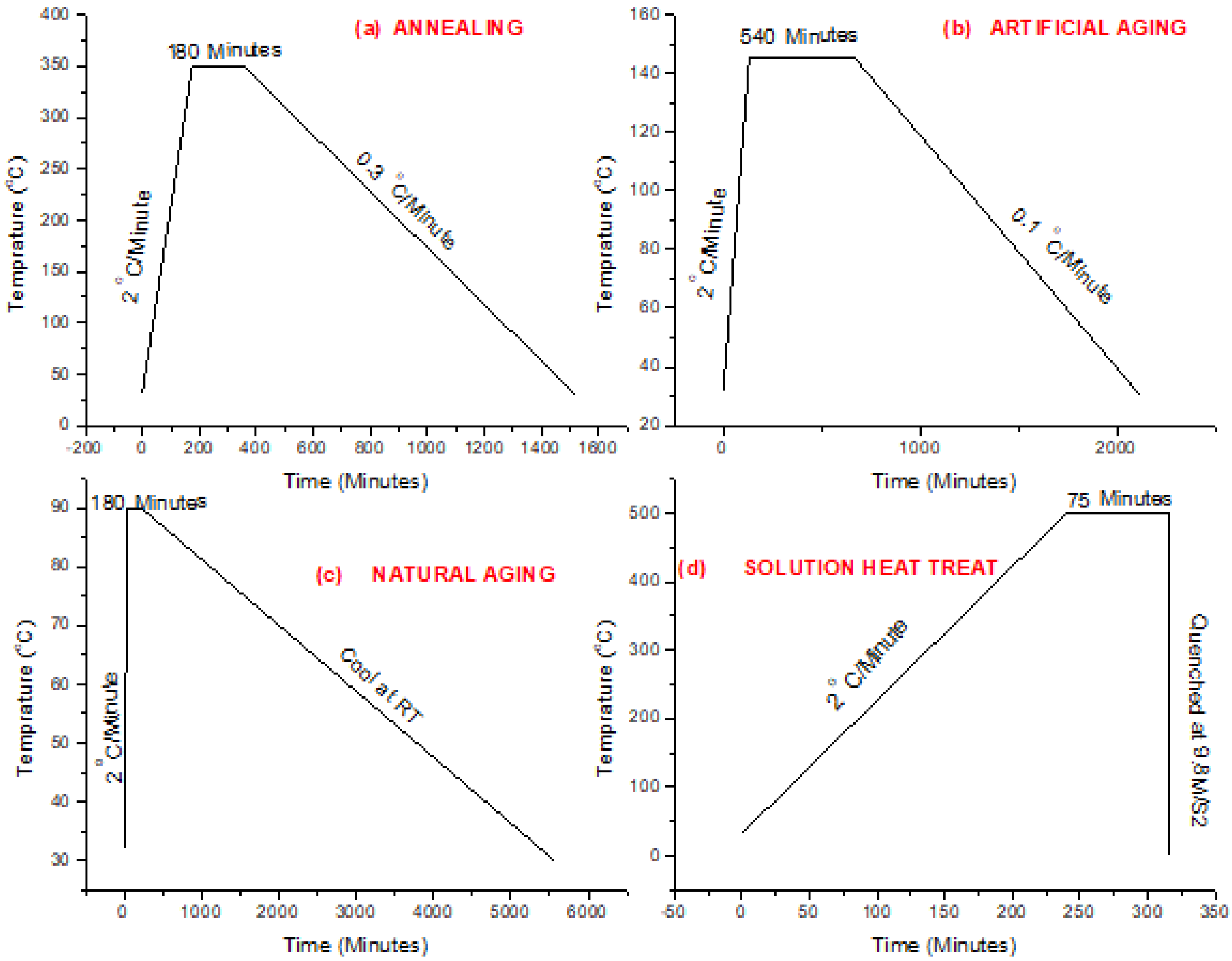


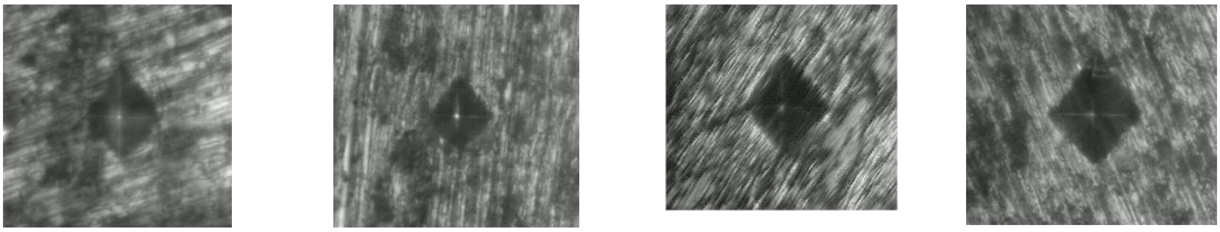
Fig. 1. Heat treatment profiles (a) annealing (b) artificial aging (c) natural aging (d) solution heat treatment quenched in clean water

3. Results

3.1 Effect of Heat Aging Time on Hardness

3.1.1 Annealing (O)

During the isothermal annealing at 2 °C/minute, soaked for 180 minutes, the formation of metastable η' phase segregations, (held in solution) are normalised by controlled cooling in furnace at 0.3 °C/minute for 1520 minutes (see Figure 1). These semi-coherent η' phases within the Al matrix constitute chief hardening and accountable for maximum hardening in Al-Zn-Mg(-Cu) alloys [12]. However, the peak hardness of 89.11 Hv detailed to 5.0 wt.% Zn is an indication that the η' phases were not transformed, but formed, into dendrites. The penetration of indenter (see Figure 2(a)) is correlated by the least mean hardness of 52.18 Hv (1.75 wt.% Mg) in Figure 3. The morphology revealed the presence of non-coherent precipitates in the α-Al matrix. This is an indication that higher temperature and/or longer time were needed for homoginization [13] of these dendrites between eutectic [14]. This stage of heat treatment is usually combined with solution heat treatments and expectantly has less impact on the hardness due to the militarized phase [4] transformation.



(a) Annealed 1.75 wt.% Mg (b) T4, 5.0 wt.% Zn (c) T6, 0.025 wt.% Mn (d) T4 0.025 wt.% Mn

Fig. 2. Micro hardness images 25µm

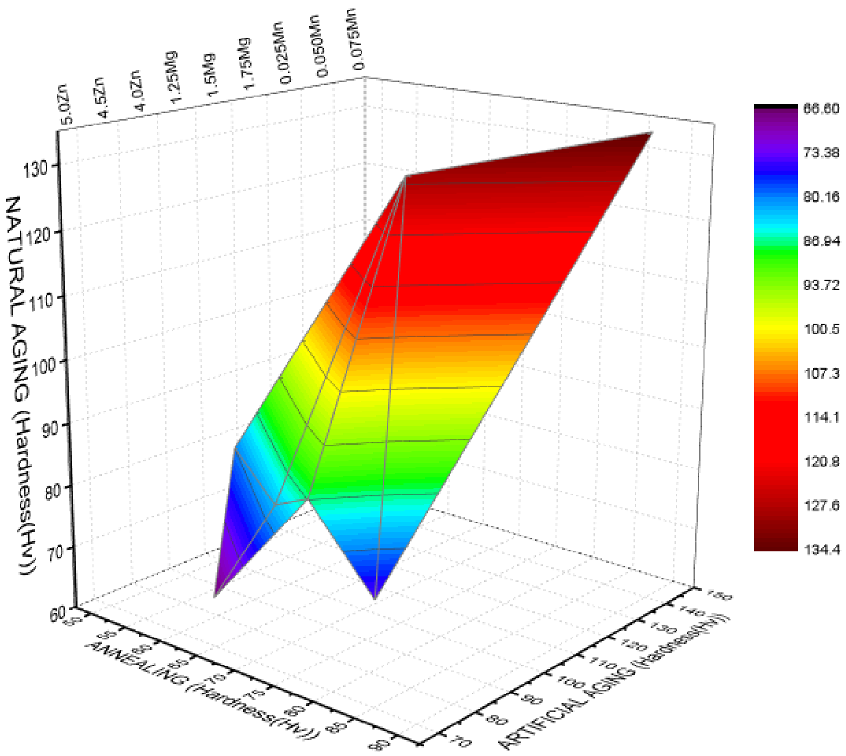


Fig. 3. Micro hardness of alloy compositions against T4, T6 and O heat treatments

3.1.2 Natural aging (T4)

Further here is mentioned the evident of phase transformation alongside continuous growth of precipitates, $MgZn_2$, with aging time as favourable to hardness [12]. Hardness was at peak, average of 134 Hv in an alloy of 5 wt.% Zn and T4 treatment condition. Figure 1(c) publicised isothermal treatment aged at room temperature for 5565 minutes. Figure 3 shows the same alloy recording 89.11 Hv in annealed condition. Morphologically, the indenter penetration is shallow with precipitates of fine grain sized η ($MgZn_2$) T (AlZnMgCu) or S $(AlCuMg)_2$ that may be formed during T4 heat treatment [13]. These civilian phase transformation [4] occurred within 180 minutes of soaking, isothermally cooling and within the GP I zone with a similar value as reported in [15]. The formation and partial dissolution [4] of this zone may be responsible for a single site peak indentation of 170.51 Hv and a mean of 134.32 Hv in the same alloy at T4. While increase in the wt.% Zn may be favourable to hardness, on the other hand, the presence of Mg_5Al_3 or Mg_5Al_8 makes the alloy susceptible to stress corrosion [15].

The result has demonstrated the transformation of phases at 90^{o}C and a soaking time of 180 minutes. Similarly, these phases could only be formed when the wt.% of Zn, Mg and Cu is higher than 3, 1 and 1 respectively [13]. The implication is that the choice of X7475 alloy composition and heat treatment supported the time-temperature-transformation (TTT) for the formation and transformation of precipitates within the α-matrix. Worthy mention is the presence of Si in the alloy. It was reported to support the formation of Mg_2Si compound. These formations hinder dislocation glide that imp3roves hardness if the precipitates are small or numerous [8].

3.1.3 Artificial aging (T6)

The presence of Fe, prior to optimization of the cystalization, may be favourable to hardness but makes the alloy suceptible to corrosion. However, for the purpose of this paper, apparently, wt.% of Zn may be responsible for the higher maximum attainable hardness values in shorter ageing incubation. Ghiaasiaan *et al.,* [15] associated this to its strong effect on the supersaturation degree of solid solution (SSSS) forming within the Al-matrix.

From Figure 3, a peak obtainable hardness value of 140.45 Hv is attained within the T6 heat treatment (see Figure 1) parameter in an alloy of 4 wt.% Zn. This value was immediately followed by 139.24 Hv in an alloy of 5 wt.% Zn. The implications of this obtainable hardness is that a higher ageing temperature may lead to a lower or higher maximum attainable hardness value [15], subject to optimzation. Yang *et al.,* [16] had reported metastable η′ phases formation as responsible for controlling this behaviour when they recorded 193 Hv peak hardness in a 7xxx alloy aged for 6 h. The transmission electron microscopy (TEM) characterization of morphology claimed η′ phases are the main strengthening precipitates.

In earlier studies, Kaneko *et al.,* [18] justified the trend in hardness with improvement in grain size. This is in agreement with the findings of Kenji *et al.,* [17] where it was reported that solid-solution and grain refinement contribute to the hardening of Al-Mg alloys. The numerous grain boundaries in fine grained materials makes such materials harder and stronger than that coarse grained materials with less grain boundaries [19]. The availability of sufficient grain boundaries promoted by artificial age hardening placed more impediments to movement of dislocations during load deformation and makes the alloy harder and stronger than the T4 and O conditions.

In our alloy, (see Figure 2c for the morphological presentation and penetration of indenter) a site peak hardness of 235.47 Hv is achieved in 5 wt.% Zn-0.025 wt.% Mn. On the other hand, the aging time may be responsible for the differences observed in this result, even as the alloy composition and homogenization mechanism play equal roles [3-7].

The morphology of T6, alloys of Al-(1.00-1.50) wt.% Mg revealed that the penetration depth of the indenter correlates the least average obtainable hardness in these alloys. A peak of 75.26 and minimum hardness of 71.85 Hv is recorded in these alloys. This may be linked to the stoichiometric ratio of solute elements for $MgZn_2$ (η) and $Mg_3Zn_3Al_2$ (T) phases. Liu *et al.,* [19] had reported higher chances of achievable hardness with η-phase with the correct Mg/Zn ratio of 1:5 (for η-phase). On the other hand, there are needs, in the EV industry, for innovative approach to hardening, leading to the selection of T61 temper for structural parts with lower formability requirements (rear rail reinforcements). The delivery of such aluminium alloys in a partial age hardened condition has the potentials to increase the final strength of component [20], this alloy may be a candidate for such applications.

4. Conclusions

New X7475 experimental aluminium alloys, produced by upgrading 3xxx secondary alloy to 7xxx series, within the Alcao Green Letter limits, has shown an average elastic recovery property under test load within the micro hardness limit. Precipitates of $MgZn_2$, $Mg_3Zn_3Al_2$ and other dendritic structures were formed and partially dissolved into the α-Al matrix during heat treatments. The transformation of which is responsible for the increase in hardness of the alloys. The T6 treatment delivered maximum obtainable hardness followed by T4 and O. The formation and transformation of military and civilian phases, credited to heat treatments, were apparently, responsible for the increase in hardness. The heat treatment process is non-exhaustive. Here, the effect of the variations of Zn (4.0 -5.0 wt.%), Mg (1.00-1.50 wt.%) and Mn (0.025 - 0.075 wt.%) remains uncovered. The correlation between heat treatment and electrical conductivity of this alloy will be considered in subsequent papers. It is pertinent to adopt response surface methodology (RSM) for optimization of the effect of heat treatments, homogenization and characterization of the precipitates in these alloys for optimal mechanical properties.

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