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Optimization of Mechanical Properties of Age-Hardened 6063 Aluminium Alloy Via Diffusion Annealing

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Authors' contributions

This work was carried out in collaboration between all authors. Author DAI designed the study, carried out the laboratory work, wrote the first draft of the manuscript and managed literature searches. Author KJA performed the statistical analysis and wrote the protocol. Author KMO performed the scanning electron microscopy and Authors MOA and ARA supervised and interpreted the results. All authors read and approved the final manuscript.

Original Research Article

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ABSTRACT

This paper investigates the influence of diffusion annealing on some mechanical properties of 6063 aluminium alloy. Standard tensile and hardness test samples were subjected to diffusion annealing at 570°C for 2, 2.5, 3 and 3.5 hours respectively prior to T6 tempering, another set of these samples were directly T6 tempered without prior diffusion annealing treatment. The two sets of samples were subjected to tensile and hardness tests and the evolving microstructure were characterized using Scanning Electron Microscope (SEM). Results indicated an increase in the mechanical properties for samples subjected to diffusion annealing prior to aging as compared to conventionally aged samples and there was also a remarkably good combination of mechanical properties especially percentage elongation (ductility) and yield strength. Samples treated by this technique find useful applications in the development of sustainable infrastructure

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in marine environments where other materials like 7075 aluminium or steel perform poorly as a result of ingress of chloride ion.

Keywords: Diffusion annealing; T6 temper; ductility; marine environments; 6063 and 7075 aluminium alloys.

1. INTRODUCTION

High strength aluminium alloys are usually chosen because of their high strength and stiffness, which are derived from precipitation hardening treatment. However, high strength aluminium alloys have poor resistance to stress corrosion cracking (SCC), particularly when they are at peak strength condition [1]. Precipitation hardening is directly responsible for the stress corrosion cracking susceptibility of high strength aluminium alloys. This high susceptibility of 7075 aluminium to corrosion especially in marine environment has shifted the attention of researchers to 6063 aluminium.

The 6xxx alloys are heat treatable and have moderately high strength coupled with excellent corrosion resistance. They are readily welded unlike 7xxx. The unique feature which makes 6063 AI a preferred choice for architectural and structural members where strength or stiffness is critically important includes its extrudability, excellent corrosion resistance, weldability and moderate strength. It is termed "marine grade alloy" due to its excellent corrosion resistance in marine environments [2].

Conversely, aluminium alloy 7075, exhibits superior strength, over 1.5 times that of the marine grade alloys, but is much more susceptible to corrosion. This alloy finds heavy use in the aircraft industry where the environmental degradation, because of corrosion, is not likely to occur. While a high performance material in the aircraft industry, it would perform poorly in marine environments.

Previous research works on homogenisation of aluminium alloys have been concentrated on direct chill cast ingots or billets homogenisation prior to extrusion; Samaras and Haidemenopoulos [3] modeled the microstructure evolution during homogenization of 6xxx aluminium alloys and concluded that the model is capable for the prediction of the homogenization completion times in industrial scale and Rivas et al. [4] investigated the effect of microstructure on the mechanical properties and surface finish of an extruded 6063 aluminium alloy and realized that Fragmentation and spheroidization of the α -Al-Fe-Si occurred during homogenization which is a temperature-time dependent process.

Zajac et al. [5], Nowotnik and Sieniawski [6] studied the influence of the cooling rate on the final mechanical properties for the 6063, 6082, 6005 alloys. Reiso et al. [7] looked at the influence of the cooling rate on the extrusion speed for various chemical compositions of Al-Mg-Si alloys. Birol [8] reported his findings on the microstructure evolution of the 6063 alloy during homogenisation for various thermal cycles. Cai et al. [9] studied the Mg₂Si dissolution during homogenisation through electrical resistivity measurements and the distribution of the alloying elements with electron microprobe measurements for the 6061, 6069 alloys. A research work on dissolution/coarsening kinetics of the Mg₂Si particles during reheating of the homogenised material was explored by Usta et al. [10].

The present study is aimed at investigating the influence of diffusion annealing on the mechanical properties of age-hardened 6063 aluminium alloy.

2. MATERIALS AND METHODS

The chemical composition of 6063 AI alloy used for this study is shown in Table 1. Standard samples for tensile and hardness tests as well as microstructural observation were prepared from 6063 AI alloy by using lathe machine. These samples were divided into groups I and II; group I samples, which were sub-divided into four: B, C, D and E, were first subjected to diffusion annealing at 570°C for 2, 2.5, 3 and 3.5 hours respectively before air-cooling. After air-cooling, the samples were solution-treated at 530°C for 4 hours, quenched in water and artificially aged at 180°C for 5 hours.

Table 1. Chemical composition of the 6063 alloy

Element	Si	Mg	Fe	Cr	Ti	Mn	Zn	Са	Al
wt. %	0.45	0.5	0.2	0.02	0.02	0.02	0.02	0.02	98.87

Group II samples were solution treated at 530°C for 4 hours, quenched in water and artificially aged at 180°C for 5 hours without prior diffusion annealing. They were then subjected to tensile and micro hardness tests. Tensile test was carried out in accordance with British Standard BSEN 10002-1 [11] at room temperature with a cross head speed of 5 mm/min using a computerized Instron 3369 electromechanical testing machine. Proof stress, ultimate tensile strength and percentage elongation values were obtained accordingly. Hardness testing was done using the LECO ASTM E384 micro hardness tester. The tests were performed on the six etched samples observed on the scanning electron microscope. The micro hardness test was carried out at 3 different points on each sample using a test load of 490.3 mN with a dwell time of 10 s. The average hardness value was calculated and recorded.

The samples for scanning electron microscopy (SEM) in each of the six condition were grinded with graded emery grit papers and polished with 0.5 micron diamond paste followed by etching with Keller's solution (1.0 ml HF, 1.5 ml HCl, 2.5 ml HNO₃ and 95.0 ml H₂O) [12].

3. RESULTS AND DISCUSSION

The heat treated samples were code named as: solution treated, quenched and artificially aged samples without prior diffusion annealing (A), diffusion annealed at 570°C for 2, 2.5, 3 and 3.5 hours prior to artificial aging at 180°C for 5 hours (B), (C), (D) and (E) respectively. The samples were air-cooled after annealing to prevent grain coarsening that is associated with furnace cooling and its deleterious effect [5,13]. The mechanical properties measured in all cases are presented in Table 2.

Mechanical Properties	Α	В	С	D	F
Proof Stress (MPa)	75.56	200.35	286.67	271.83	260.03
UTS (MPa)	161.23	225.44	468.73	314.95	302.05
Hardness (VHN)	61.3	72.4	113.1.	73.9	69.2
Elongation (%)	21.88	11.37	21.37	18	17.36

Table 2. Results of mechanical testing for heat treated 6063 al samples

Proof stress (yield strength) values for the samples were found to increase significantly after diffusion annealing (homogenisation). This is seen in Table 1 where there was increase from

75.56 MPa for no homogenisation to 161.23 MPa after homogenisation for 2 hours. However, maximum value was obtained after homogenisation for 2.5 hours beyond which it decreased slightly. The same trend, as for the yield strength was observed for the ultimate tensile strength. In these cases, homogenisation prior to solution treatment has been found to be necessary. The significant increase strength has been found to be due to removal of deleterious intermetallic phases and structures which are difficult to remove by solution treatment only; enrichment of the solid solution matrix with solute atoms for solution strengthening; as well as the release of solute atoms for subsequent formation of favorable and coherent precipitates.

Figs. 1 and 2 show micrographs of as-received samples which were solution-treated and aged without prior homogenisation. Here the presence of incoherent and elongated intermetallic phases is adjudged responsible for low yield and ultimate tensile strengths.



Fig. 1. SEM micrograph of the as-received 6063 aluminium alloy used for this study. Showing elongated phases

The optimum values of yield and ultimate tensile strengths (286.67 and 468.73 MPa) were obtained at 2.5 hours homogenisation prior to aging. This implies that optimum homogenisation treatment was achieved with removal of segregations, spheroidisation of dispersoids and transformation of β -AI-Fe-Si to α -AI-Fe-Si resulting in formation of coherent precipitates during aging. This is in agreement with the findings of [4,14] whose results show that no more than 2.5 hours is required for complete homogenisation of 6063 aluminium alloy. This structure is provided in Fig. 3 where spheroidisation occurred and coherent precipitates were not revealed.

Although, it has been reported that grain coarsening may cause reduction in strength of metals [5]. However, this reduction is likely to be due to formation of non-coherent precipitates caused by excessive dissolution of solute atoms during extensive homogenisation which resulted in a condition of over aging. This is evident in the amounts of precipitates in Figs. 4 and 5 as compared with Fig. 3.

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Fig. 2. SEM micrograph of 6063 aluminium alloy, solution treated at 530°C for 4 hours, quenched and artificially aged at 180°C for 5 hours. Showing the presence of few elongated phases



Fig. 3. SEM micrograph of 6063 aluminium alloy homogenised at 570°C for 2.5 hours, solution treated at 530°C for 4 hours, quenched in water and artificially aged at 180°C for 5 hours. Showing the prominence of spherical precipitates

Similar trend as in the yield and ultimate tensile strengths in Table 2 was observed for hardness values. Hardness values increased from 61.30 VHN (for no homogenisation) to 113.1 VHN (for 2.5 hours homogenisation). Thereafter it significantly dropped to 73.9 VHN and 69.20 VHN for 3 and 3.5 hours homogenisation respectively. This reduction is likely to be due to dissolution of high alloy compounds which precipitated out at 180°C to form greater amounts of incoherent precipitates. The observed drastic drop in the hardness

values may be attributed to incoherency of the precipitates that formed after 2.5 hours homogenisation and which might also encourage overaging [5,15]. The high hardness value of samples homogenised for 2.5 hours prior to aging at 180°C for 5 hours affirmed the theory that strength and hardness are constant multiples of each other for some materials [13,16].



Fig. 4. SEM micrographs of 6063 aluminium alloy homogenised at 570°C for 3 hours, solution treated at 530°C for 4 hours, quenched in water and artificially aged at 180°C for 5 hours



Fig. 5. SEM micrographs of 6063 aluminium alloy homogenised at 570°C for 3.5 hours, solution treated at 530°C for 4 hours, quenched in water and artificially aged at 180°C for 5 hours

The percentage elongation (% elongation) which is the change in length of a tensile test material at fracture is a measure of material ductility. From Table 2, the elongation increased with homogenisation time. Generally, the % elongation does not significantly increase with homogenisation period and seems to reach the lowest level after 2.5 hours homogenisation.

The comparatively high % elongation obtained when no prior homogenisation was carried out is not expected. It appears that solution treatment alone resulted in stress relief annealing of the as-received structure (Fig. 1).

The % elongations values for specimens homogenised for 3 and 3.5 hours were still considerably higher than when no homogenisation was carried out. This is also confirmed by the microstructure in Fig. 6, where there is substantial quantity of unmodified second phase particles.



Fig. 6. SEM micrographs of 6063 aluminium alloy homogenised at 570°C for 2 hours, solution treated at 530°C for 4 hours, quenched in water and artificially aged at 180°C for 5 hours. Showing the presence of some unmodified second phase particles

The general improvement in the mechanical properties of those samples homogenised prior to aging can be explain by using the physical nature of alloy strengthening, which appears as result of diffusion annealing. Diffusion annealing ensures complete dissolution of sharpedge phases that are associated with the direct chill casting of this alloy. All these alloying elements (except for copper) increase the quench sensitivity of the 6000 alloys, with magnesium having the greatest impact. Manganese and chromium also increase guench sensitivity as they combine with aluminium, silicon and iron at homogenising temperatures to form fine dispersoids. These AI-Fe-Mn/Cr-Si dispersoids act as heterogeneous nucleants for magnesium-silicide precipitates in subsequent thermal treatments thereby increasing the quench sensitivity of 6000 alloys. This leads to an increased in the density of precipitate formed. The finer these grains are the more the boundaries. During plastic deformation, slip or dislocation movement must take place across these grain boundaries. Since polycrystalline grains are of different crystallographic orientations at the grain boundaries, a dislocation passing from one grain to another will have to change its direction of motion. Such changes of direction causes impediment to dislocation movement, and increases both the yield strength and ultimate tensile strength. Because age hardening samples have the highest number of grain boundaries, dislocation movement becomes more and more difficult during plastic deformation. This is responsible for optimum combination of mechanical properties for those samples homogenised prior to aging as compared to those that were just T6 tempered.

The optimum mechanical properties obtained at 2.5 hours homogenisation can be attributed to complete spheroidisation (Fig. 3) of rod-like phases present in the as-received samples (Fig. 1) and formation of new dispersoids which acted as heterogeneous nucleants for subsequent formation of magnesium-silicide during aging. It is important to note that this complete spheroidisation may or may not take place during solution treatment (Fig. 2).

The subsequent reduction in mechanical properties can also be attributed to overaging that may be brought about by excessive homogenisation time. When the precipitates are coherent they posed severe impediment to dislocation motion and thereby strengthening the alloy as high stress will be required to cause dislocation movement, the refers is the case for incoherent precipitates (Isadare et al. [13]).

In commercial alloys containing Al–Mg–Si–Fe–Mn–Cu–Cr–Zn several phases may be present. Besides the prominent Mg₂Si phase, which contributes to the final mechanical properties, Fe in combination with Si may form the ternary phases α -AlFeSi, β -AlFeSi or else with Cr, Mn the quartenary phase α -Al(MnCrFe)Si (Samaras and Haidemenopoulos 2007).

4. CONCLUSION

Aluminium alloy 6063 has been found to respond well to age hardening process, which is influenced by homogenisation temperature and time, solution treatment temperature and time and aging temperature and time.

Homogenisation treatment causes formation of uniform structure with respect to uniform distribution of solute atoms, dissolution of solid state phases in the matrix and formation of uniform grain structure. For these reasons, matrix composition and structure are affected.

Solution treatment on the other hand prepares the matrix condition for subsequent precipitation of solid phases for strengthening and hardening.

Homogenisation treatment prior to aging has been found suitable for ensuring optimum combination of strength and ductility in age-hardened alloys. It causes the formation of fine dispersoids that eventually acted as heterogeneous nucleants for subsequent formation of magnesium-silicide precipitate during aging; this increased the density of precipitates formed and thereby strengthening the alloy by increasing the grain boundaries. The formation of these rounded or spherical dispersoids also prevents the weakening of the grain boundaries; since most of the precipitates nucleated on these dispersoids.

The result of this work can also be extended to other heat treatable aluminium alloys to prevent the concomitant decrease in ductility and toughness of age-hardenable aluminium alloys after aging. It also depicts that complete homogenisation of 6063 aluminium alloy can be achieved at 570°C for 2.5 hours.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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