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EFFECT OF QUENCHING TEMPERATURE ON THE MECHANICAL PROPERTIES AND MICROSTRUCTURE OF RECYCLED ALUMINIUM-COPPER-ZINC ALLOY SUBJECTED TO T_4 AND T_6 THERMAL TREATMENTS

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Abstract: The effect of natural and artificial age hardening and varied quenching temperatures on the mechanical properties and microstructure of secondary Al-Zn-Cu alloy were studied. Stir casting was used to produce rods from aluminium scraps; some of the rods were homogenized in a heat treatment furnace at 550°C for 5 hours before cooling under the atmospheric air at room temperature. Some of the machined specimens from the homogenized allov were subjected to natural ageing after quenching in water at different temperatures. The specimens for artificial ageing were reheated in the furnace to 177°C after quenching, held for 5 hours and brought out to cool under air at room temperature. XRD reveal the precipitation of Si after age hardening process while the micrographs show the precipitated phases. The optimal combinations of mechanical properties were achieved by quenching in water at room temperature (32°C) and 60°C respectively and natural ageing.

Keywords: Age hardening, scrap, aluminium, mechanical properties, microstructure

1. INTRODUCTION

Aluminium alloys have found applications in areas such as automotive panel materials, aircraft structures, foils, electrical conductors, roofing sheets, oil tanks, etc., due to their lighter weight, corrosion resistance, specific strength and formability (Mroczka et al., 2012; Miao and Laughlin, 2000; Tabereaux and Peterson, 2014). The production and use of aluminium also results to the generation of scraps in form of off-cuts, machining swarfs, chips, droses, and end-of-life aluminium products (Wallace, 2011). The scrap aluminium will constitute environmental problems when they are not properly managed. The importance of recycling cannot be over emphasized. Recycled aluminium alloys are also referred to as secondary aluminium. The production of new metals through the mining, extraction and refining processes requires energy. The energy and cost requirements for aluminium recycling are less than that those for production of new metals (Wallace, 2011; Mouritz, 2012). Recycling also reduces the waste that goes to the landfill (Mouritz, 2012). Another reason that favors recycling of aluminium is that it has high scrap value as it can be recycled repeatedly without impairing its properties (Wallace, 2011; Mouritz, 2012). Aluminium scraps are however contaminated by iron and silicon; these elements have limited solubility in aluminium resulting in the formation coarse Fe-Si precipitates usually located at the grain boundaries and are weakly attached to the matrix resulting to low mechanical properties of recycled aluminium alloys (Neikov, 2019). The need to strengthen secondary aluminium alloys becomes necessary. Aluminium alloys can be strengthened by solid solution hardening, age hardening or grain refinement. Among the methods used to strengthen aluminium alloys, age hardening is the most effective and applies to the aluminium alloys containing copper, magnesium, chromium, silicon and zinc (Mroczka et al., 2012). Age hardening of aluminium alloys constitutes heating the alloy to nearly the solvus temperature, quenching in water to obtain super saturated solid solution, followed by natural or artificial aging treatment (Cochard et al., 2017). Natural aging is done by leaving the solution treated and guenched alloy at room temperature for several days while artificial aging is done by heating the alloy to temperature range $150 - 200^{\circ}$ C and held for several hours up to tens of hours to attain maximum strength and hardness by precipitation and dispersion of the second phase in the microstructure (Starke, 1989). Heat treatable aluminium alloys are those that contain any of copper, magnesium, lithium, silicon and zinc because they respond to precipitation hardening and the solid solubility of the alloying elements decrease with decrease in temperature (Rambabu and Kutumbarao, 2017). The combination of mechanical properties obtained in heat treated aluminium alloys depends on several process parameters such as the solution treatment temperature (Reis et al., 2012), guenching media (Abubakre et al., 2009), guenching temperature and cooling rate (Slámová et al., 2008; Kavalco et al., 2009; Kavalco et al., 2009), as well as aging temperature and time (Reis et al., 2012; Hossain and Kurny,

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2013). In this work, the effect of quenching temperatures on the properties of age hardened secondary aluminium-copper–zinc alloy was investigated.

2. MATERIALS AND METHODS

— Production of the recycled cast aluminium alloy, homogenization and chemical analysis

6 kg of aluminium scrap from roofing sheets earlier cleaned and dried was melted using oil fired pit melting furnace inside a crucible and the melt was superheated to 770°C to remove slag. The chemical composition of the scrap was unknown, but about 2-4 wt% of copper was targeted in the composition of the recycled cast scrap to enhance its mechanical properties and make it more prone to ageing (Adeosun et al., 2011). After removing the slag, 250 g of copper wire melted with another crucible was added to the aluminium melt and stirred. The melt was allowed to cool to about 730°C and then cast in sand mould into rods of 12 mm diameter and 20 cm length. The as-cast specimens were kept and the others were subjected to homogenization heat treatment in order remove internal stress, eliminate compositional segregation and dissolve the coarse interdendritic phases. The homogenization process was carried out in an electric heat treatment furnace by raising the temperature of the cast specimens at the rate of 10°C/minute to 550°C and held for 5 hours before they were brought out to cool under air at room temperature (Birol, 2004; Priya, 2016). Optical emission spectrometer (QUANTO DESK AA83673 Model) was used to determine the elemental composition of the homogenized alloy.

- Machining and precipitation hardening

Tensile specimens of gauge length 30 mm and gauge diameter 4 mm were machined from the specimens in line with ASTM standard E8/E8M -21. Specimens for hardness test and metallography of dimensions 20 mm height by 12 mm diameter were also machined; while those for impact test were machined to 75 mm length and 8 mm diameter following ASTM E23-18 standard. The specimens were grouped into as-cast, homogenized, T_4 specimens and T_6 specimens. Some of the homogenized specimens were subjected T_4 age hardening treatment by raising the temperature in an electric heat treatment furnace to 470°C at the rate of 10°C/minute, holding for 2 hours (solutionization), before quenching in water (Totten and Webster, 2003). Different specimens were quenched in water at temperatures; 0°C, room temperature (32°C), 60°C and 100°C respectively. The specimens were allowed to cool in the water before they were brought out and kept at room temperature for 30 days to complete the natural ageing process (ASM International, 1991; Isadare et al., 2013). Those for T_6 age hardening treatment followed the same heating and quenching scheme as the T_4 specimens, but were subjected to artificial ageing by reheating in the furnace up to 177°C, held for 5 hours and were brought out to cool under air at room temperature (Zainon et al., 2009).

— Mechanical testing of the specimens

Uniaxial tensile tests were carried out on the tensile specimens at room temperature using Instron[®] Universal Testing Machine (Model 3369) at 10 mm/s strain rate following ASTM standard E8/E8M -21. The specimens were tested repeatedly and the average values were reported to ensure reliability.

Rockwell hardness tests were carried out on the specimens using Mosanto[®] Hardness Testing Machine with a diamond indenter under major load of 100 kg and minor load of 10 kg at dwell time of 2.8 seconds. Three indentions were made on each specimen and the average hardness were computed and reported for each specimen.

Izod impact tests were carried out on the specimens using Hounsfield® Balanced Impact Machine in line with ASTM E23-18 to obtain the amount of energy absorbed by each specimen before yielding. Repeated tests were also carried out and the average results also computed and reported. All the mechanical tests were carried out at room temperature.

— Optical metallography on the specimens

The standard method of sample preparation for metallography including mounting, grinding, polishing and etching with 2% NITAL according to ASTM E3-11 was followed. The grit sizes of the emery papers used for the grinding ranged from 500 μ m to 1500 μ m. Suspension of polycrystalline diamond particles of sizes ranging from 0.5 μ m to 10 μ m was used for fine polishing. Accu-Scope[®] optical metallurgical microscope was used to obtain the micrographs (× 400) of the microstructure of the specimens were obtained using Accu-Scope[®] optical metallurgical microscope.

— XRD characterization of the specimens

The phases present in the microstructure were identified and quantified using Rigaku miniflex 600 XRD machine at varied diffraction angle (2 θ) from 2° to 70° at a scanning speed of 2°/min on the as-cast specimen and the naturally aged specimen quenched in water at 60°C. The X-ray source of Cu K α radiation of wavelength ($\lambda = 1.5406$ Å) was used.



3. RESULTS AND DISCUSSION

Chemical composition cast secondary aluminium alloy

The chemical composition of the cast recycled aluminium alloy is shown in Table 1. It is characterized by high content of iron and silicon impurities, which depicts typical composition of aluminium scrap (Neikov, 2019; Kuchariková, 2016). The iron impurities could have come from sources such as cutting tools, "Fe pickup" while the silicon could have come from possible slag inclusions.

Element	AI	Si	Fe	Cu	Mn	Mg	Zn	Cr	V	Ca	Sr	Other
%	85.79	6.15	1.87	2.39	0.31	0.08	2.56	0.07	0.12	0.004	0.001	0.67

Table 1. Chemical composition of the recycled aluminium alloy

Mechanical properties of the specimens

Figures 1 – 5 show respectively the tensile strength, elongation at break, impact energy, elastic modulus and Rockwell hardness of the cast specimens. The best combinations of mechanical properties were achieved in specimens guenched in water at room temperature and at 60° C respectively and subjected to natural ageing when compared with the as-cast specimen. The specimen quenched in water at room temperature and subjected to natural ageing exhibited 15.3%, 26.9% and 7.9% improvement in tensile strength, ductility and impact toughness respectively; while that guenched in water at $60^{\circ}C$ and naturally aged showed 19.3%, 14.9% and 2.8% increase in tensile strength, ductility and impact toughness respectively. Figure 5 shows that there is no significant change in hardness at those conditions where strength and toughness were improved when compared to the as-cast specimen. The effects of quenching temperature are noticeable on the properties of the age-hardened alloy because the guenching temperature affects the cooling rate which subsequently affects the rate of precipitation of the second phase and their distribution in the alloy (Kavalco et al., 2009; Totten and Webster, 2003). The higher cooling rate resulting to higher heat extraction experienced by the specimen quenched in water at 32°C and 60°C resulted to finer microstructure and more uniform distribution of the precipitated phase in the microstructure (Abubakre et al., 2011). The decrease in elastic modulus observed in the observed in Figure 4 for the specimens guenched at 32° C and 60° C and subjected to natural ageing is as a result of improved toughness and ductility. The specimens subjected to artificial ageing could not replicate the improved mechanical properties obtained in the naturally aged specimen because the longer ageing time resulted to the applomeration and spherudization of the precipitated phase in the microstructure. The effect of variation in the ageing temperature and holding time for the specimens subjected to T₆ heat treatment will further be studied and elucidated.

— Microstructure of the specimens

The micrographs of the microstructure of all the specimens are shown in Plates A – J (\times 400). The micrograph of the as-cast specimen as shown in Plate A shows coarse grains and the presence of impurities around the grain boundaries. High content of silicon in aluminium gives rise to coarse grains (Jaradeh, 2006). Metals characterized by coarse grains usually exhibit inferior mechanical properties to similar metals with finer grains (Abubakre et al., 2009; Umunakwe et al., 2017). Plate B shows the microstructure of the homogenized specimen with improved homogeneity in the structure as a result of redistribution of elements and dissolution of some the impurities around the grain boundaries. Precipitated phases were seen in all the age-hardened specimens (Plates C - J). However, different quenching conditions and type of ageing affected their distribution in the microstructure. Those with poor distribution of the precipitated phase (Plates C, D, F, H, I and J) exhibited poor mechanical properties while those with good distribution of the second phase exhibited improved mechanical properties in Figure 1-3 and 5 respectively.



Figure 1. Effect of quenching temperature, T4 and T6 temper treatment on the ultimate tensile strength of the recycled Al-Cu-Zn alloy



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Figure 2. Effect of quenching temperature, T4 and T6 temper treatments on the percentage elongation at break of the recycled Al-Cu-Zn alloy



Figure 3. Effect of quenching temperature, T4 and T6 temper treatment on the impact energy of the recycled Al-Cu-Zn alloy



Figure 4. Effect of quenching temperature, T4 and T6 temper treatment on the elastic modulus of the recycled Al-Cu-Zn alloy



Figure 5. Effect of quenching temperature, T4 and T6 temper treatment on the Rockwell hardness of the recycled Al-Cu-Zn alloy



Plate A: As-Cast specimen Plate B: Homogenized specimen Plate C: Quenched at 0°C and naturally aged





Plate D: Quenched at 0°C and artificially aged Plate E: Quenched at 32°C and naturally Plate F: Quenched at 32°C and artificially aged



Plate G: Quenched at 60°C and naturally Plate H: Quenched at 60°C and artificially aged Plate I: Quenched at 100°C and naturally aged



Plate J: Quenched at 100°C and artificially aged — X-ray diffraction pattern of the as-cast and the aged specimens

The XRD pattern of the as-cast and agehardened specimens are shown in Figure 6 and 7. The major phases identified were aluminium, graphite and the intermetalics; aluminin chromite, garnet, and andalusite formed as a result of interaction of aluminium and the impurities. However, the amount of andalusite (Al_2SiO_5) increased from 12% in the as-cast specimen to 22% in the age hardened specimen. It implies that more silicon was precipitated after the age-hardening treatment and interacted with aluminium resulting to the distribution of andalusite in the age-hardened specimen.





4. CONCLUSIONS

The study on the effects of various quenching temperatures on the mechanical properties

Figure 7. XRD pattern of the age-hardened specimen

and microstructure of the secondary Al-Cu-Zn alloy subjected to T_4 and T_6 thermal treatments reveals that a combination of quenching temperature and method of ageing affects the distribution of the precipitated silicon in the microstructure. High and extremely low quenching temperatures and artificial ageing for 5 hours at 177°C resulted to poor distribution and agglomeration of precipitated phase in the microstructure and that translated to poor mechanical properties. The optimal water quenching temperature were 32°C and 60°C and natural ageing of the quenched specimen resulted to good distribution of the precipitated phase and improved



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mechanical properties. The recycled aluminium alloy with improved mechanical properties can find applications in areas such as window frames, dish washers, household furniture, etc.

References

- Abubakre, O. K.; Mamaki, U. P.; and Muriana, R. A.: Investigation of the quenching properties of selected media on 6061 aluminum alloy. Journal of Minerals and Materials Characterization and Engineering, 4 (4), 303–315, 2009.
- [2] Adeosun, S. O.; Balogun, S. A.; Osoba, L. O.; Ayoola, W. A.; and Oladoye, A. M.: Effect of Cu and Zn addition on the mechanical properties of structural aluminum alloy. Journal of Modern Manufacturing Technology, 3 (1), 103–110, 2011.
- [3] Birol, Y.: The effect of homogenization practice on the microstructure of AA6063 billets. Journal of Materials Processing Technology, 148, 250–258, 2004.
- [4] ASM International: Heat treating of aluminum alloys. In ASM Handbook, Volume 4: Heat Treating (pp. 841-879). Ohio: ASM International, 1991.
- [5] ASTM E8/E8M -21 "Standard test method for tension of metallic materials in Annual Book of ASTM Standards". Philadelphia: American Society of Testing and Materials, 2021.
- [6] ASTM E3-11 "Standard guide for preparation of metallographic specimens in Annual Book of ASTM Standards". Philadelphia: American Society of Testing and Materials, 2017.
- [7] ASTM E23-18 " Standard test methods for notched bar impact testing of metallic materials in Annual Book of ASTM Standards". Philadelphia: American Society of Testing and Materials, 2018.
- [8] Cochard, A.; Zhu, K., Joulié, S.; Douin, J.; Huez, J.; Robbiola, L.; Sciau, P.; and Brunet, M.: Natural aging on Al-Cu-Mg structural hardening alloys- investigation of two historical duralumins for aeronautics. Materials Science and Engineering: A, 690, 259-269, 2017.
- [9] Hossain, A.; and Kurny, A. S.: Effect of ageing temperature on the mechanical properties of AI-6Si-0.5Mg cast alloys with cu additions treated by T6 heat treatment. Universal Journal of Materials Science, 1 (1), 1 -5, 2013.
- [10] Isadare, A. D.; Aremo, B.; Adeoye, M. O.; Olawale, Ö. J.; and Shittu, M. D.: Effect of heat treatment on some mechanical properties of 7075 aluminium alloy. Materials Research, 16 (1), 190–194, 2013.
- [11] Jaradeh, M. M.: The Effect of processing parameters and alloy composition on the microstructure formation and quality of DC cast aluminium alloys. Sundsvall, Sweden: Department of Engineering, Physics and Mathematics, Mid Sweden University, 2006.
- [12] Kavalco, P. M.; Canale, L. C.; and Totten, G. E. (2009). Quenching of aluminium alloys: cooling rate, strength and intergranular corrosion. Heat Treating Progress, 25–30.
- [13] Kavalco, P. M.; Canale, L. C.; and Totten, G. E.: Quenching of aluminum alloys: property prediction by qunch factor analysis. Heat Treating Progress, 23–28, 2009.
- [14] Kuchariková, L.; Tillová, E.; and Bokůvka, O.: Recycling and properties of récycled aluminium alloys used in the transportation industry. Transport Problems, 11 (2), 117–122, 2016.
- [15] Miao, W. F.; and Laughlin, D. E.: Effects of Cu content and preaging on precipitation characteristics in aluminum alloy 6022. Metallurgical and Materials Transactions A, 31A, 361-371, 2000.
- [16] Mouritz, A. P.: Disposal and recycling of aerospace materials. In A. P. Mouritz (Ed.), Introduction to Aerospace Materials (pp. 558-568). Cambridge: Woodhead Publishing Limited, 2012.
- [17] Mroczka, A.; Wójcicka, A.; and Kurtyka, P.: 2017A aluminum alloy in different heat treatment conditions. Acta Metallrgical Slovaca, 18 (2–3), 82–91, 2012.
- [18] Neikov, O. D.: Production of aluminum alloy powders. In O. D. Neikov, S. S. Naboychenko, and N. A. Yefimov, Handbook of Non-Ferrous Metal Powders (Second ed., pp. 481–531). Amsterdam: Elsevier Ltd, 2019.
- [19] Priya, P.: Microstructural evolution during the homogenization heat treatment of 6xxx and 7xxx aluminum alloys. Indiana: Purdue University, 2016.
- [20] Rambabu, P.; and Kutumbarao, V. V.: Aluminium alloys for aerospace applications. In N. E. Prasad, and R. J. Wanhill (Eds.), Aerospace Materials and Material Technologies, Indian Institute of Metals Series (pp. 29-52). Springer, 2017.
- [21] Reis, D. A.; Couto, A. A.; Domingues Jr, N. I.; Hirschmann, A. C.; Zepka, S.; and Neto, C. M.: Effect of artificial aging on the mechanical properties of an aerospace aluminum alloy 2024. Defect and Diffusion Forum, 326–328, 193–198, 2012.
- aluminum alloy 2024. Defect and Diffusion Forum, 326-328, 193-198, 2012. [22] Slámová, M.; Janeček, M.; Cieslar, M.; and Šíma, V.: Effect of quenching temperature on age hardening of AA6016 Sheets. Materials Science Forum, 567-568, 333-336, 2008.
- [23] Starke, J. E.: Heat-treatable aluminum alloys. In A. Vasudevan, and R. Doherty (Eds.), Aluminum Alloys-Contemporary Research and Applications (pp. 35-36). San Diego, CA: Academic Press, Inc, 1989.
- [24] Tabereaux, A. T., and Peterson, R. D.: Aluminum production. In S. Seetharaman (Ed.), Treatise on Process Metallurgy Volume 3: Industrial Processes (pp. 839– 917). Amsterdam : Elsevier Ltd, 2014.
- [25] Totten, G. E.; and Webster, G. M.: Quenching. In G. E. Totten, and D. S. MacKenzie (Eds.), Handbook of Aluminum: Vol. 1: Physical Metallurgy and Processes (1 ed., Vol. 1, pp. 971–1062). Boca Raton, Florida: CRC Press, 2003.
- [26] Umunakwe, R.; Olaleye, D. J.; Oyetunji, A.; Okoye, O. C.; and Umunakwe, I. J.: Assessment of some mechanical properties and microstructure of particulate periwinkle shell-aluminium 6063 metal matrix composite (PPS-ALMMC) produced by two-step casting. Nigerian Journal of Technology, 36 (2), 421-427, 2017.
- [27] Wallace, G.: Production of secondary aluminium. In R. N. Lumley (Ed.), Fundamentals of Aluminium Metallurgy (pp. 70–82). Cambridge: Woodhead Publishing, 2011.
- [28] Zainon, M. F., Ahmad, K. R., Zaki, R. M., and Daud, R., Effect of age hardening on wear properties of aluminum alloys piston. Malaysian Metallurgical, (pp. 1-7). University Malaysia Perlis, Perlis, 2009.







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