

Mechanical Properties Enhancement of 66/34Mg-Alloy for Medical Application

S. O. Adeosun, O. I. Sekunowo, O. P. Gbenedor, W. A. Ayoola, A. O. Odunade, T. A. Idowu

Abstract—Sand cast samples of the as-received 66/34Mg-Al alloy were first homogenized at 4900C and then divided into three groups on which annealing, normalising and artificial ageing were respectively carried out. Thermal ageing of the samples involved treatment at 5000C, soaked for 4 hours and quenched in water at ambient temperature followed by tempering at 2000C for 2 hours. Test specimens were subjected to microstructure and mechanical analyses and the results compared. Precipitation of significant volume of stable Mg₁₇Al₁₂ crystals in the aged specimen's matrix conferred superior mechanical characteristics compared with the annealed, normalized and as-cast specimens. The ultimate tensile strength was 93.4MPa with micro-hardness of 64.9HRC and impact energy (toughness) of 4.05J. In particular, its Young modulus was 10.4GPa which compared well with that of cortical (trabecule) bone's modulus that varies from 12-17GPa.

Keywords—Mg-Al alloy, artificial ageing, medical implant, cortical bone, mechanical properties.

I. INTRODUCTION

CONSIDERATION for requisite mechanical properties is essential in materials selection for various applications. The development of a cutting-edge processing method capable of inducing appropriate microstructure transformation for enhanced mechanical characteristics is also imperative. Hence, there are growing interests in the potentials of magnesium alloys use as an effective and cost competitive substitute for existing surgical implant materials [1], [2]. The possibility of using magnesium (Mg) implants to act as a scaffolding frame on which new bone can grow as well as fixtures to hold together bones long enough to allow for natural healing is also gaining wide interest [3]. Generally, metals are more suitable for load-bearing applications than ceramics or polymeric materials due to their characteristic combined high strength and fracture toughness. According to Staiger et al., [4] the elastic modulus of modern metallic biomaterials is higher than that of natural bone tissues. Comparable high elastic modulus often results in reduced stimulation of new bone growth and remodelling which impairs implant stability. Magnesium based alloy is particularly desirable in these applications because of its comparable modulus of elasticity with human bone which is in the range of 3-20GPa while that of Magnesium based alloy is about 45GPa [5]. These values are in contrast with most implant materials currently in use namely; chrome-cobalt (230GPa), stainless steel (200GPa) and

titanium (115GPa). The use of these materials often leads to the implant carrying a greater portion of the imposed load with the risk of causing stress shielding of the bone [6].

Further, medical implant materials are usually required to be nontoxic or carcinogenic and able to be tolerated by the body without causing further harm. Magnesium possesses good biocompatibility and relatively large amounts of magnesium are tolerated by the body without adverse side effects. This is because excess magnesium ions are readily excreted through the kidneys [7]. Given this scenario, the need for extra surgery to remove an implant is unnecessary. Hence, there is an upsurge in research on magnesium alloys' use as biodegradable metal implant in orthopaedic surgery. However, the focus currently is mainly on commercial alloys such as Mg-Al and Mg-RE alloys, due to their relatively high strength and good corrosion resistance [8], [9].

Although Li et al [10] alluded to the inherent low strength characteristics of pure Mg as a major challenge however; an appropriate alloy composition could be introduced to improve its mechanical properties. In particular, the strength of cast magnesium alloys can be enhanced through various hardening techniques such as solid solution hardening, grain size hardening, age-hardening and dispersion hardening. Amongst these techniques, grain refinement is a proven favorable method of improving mechanical properties of Mg and its alloys [11].

Hence, significant efforts have been devoted to developing wrought Mg alloys with ultra-fine grains [12], [13]. In this context, the most common hardening elements are aluminum, zinc, silicon, rare earth elements, silver and yttrium. Some of these alloying elements have abilities to stabilize the structure under load at elevated temperature by forming high density and stable dispersions of precipitates [14]. This is further enhanced because magnesium and aluminum are fully soluble in the liquid state, and the eutectic reaction takes place at 437°C, forming a mixture of α -Mg phase and brittle binary β -Mg₁₇Al₁₂ phase. During cooling of the alloy with more than 8 percent Al (depending on cooling rate) precipitation starts at grain boundaries producing lamellar precipitates which gives rise to improved mechanical characteristics. The present study attempts a comparative heat treatment procedure of a typical magnesium-aluminum alloy with a view to enhancing the mechanical properties suitable for medical implant application.

II. EXPERIMENTAL

A. Materials and Heat Treatment

The 66/34Magnesium-Aluminum alloy used in this study

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was obtained from Nigalex PLC Ikeja, Nigeria and its chemical composition is presented in Table I. The alloy was put in a crucible pot and then placed in a furnace where it was heated until molten. The melt was cast in sand moulds of rectangular dimension, 150x30x30mm, allowed to cool, stripped and fettled by sand blasting. The cast samples were divided into four groups and identified as A, B, C, and D respectively. The A group of samples were left untreated in the as-cast condition while the B and C samples were solutionised at 4900C, soaked for 4 hours, normalized in still air and annealed in the furnace respectively. However, the D group of samples was artificially aged. The ageing process involved heating the samples to 5000C, soaked for 4 hours, quenched in water to assume a super saturated solution state. This was followed by tempering at 2000C for 2 hours to ensure redistribution of phases within the matrix.

TABLE I
ALLOY COMPOSITION

Elements	%Composition
Mg	65.914
Al	33.54
Cu	0.353
Fe	0.101
Others	0.092

B. Mechanical Test

Standard tensile, impact energy (Notched Charpy V) and micro-hardness test specimens were prepared according to ASTM E8 from A, B, C, and D group of samples. The specimens' tensile behavior was determined using Instron electromechanical testing machine while a Topla digital hardness testing machine and an Avery impact tester was used respectively to obtain the specimens' micro-hardness and impact energy absorbed to fracture. Results of these tests are illustrated in Figs. 2-5.

C. Microstructure Analysis

Test pieces of about 20mm in length were cut from each group of samples for microstructure analysis. The surfaces of the specimens were ground in succession using emery paper of grades 40, 32, 10 and 8. Each specimen surface was thereafter polished using aluminum-diamond paste until a mirror-like surface was obtained. The surfaces were then etched for 20 seconds in a solution of sodium hydroxide (Pellets) dissolved in 100ml of water. The evolved microstructures of the specimens were viewed under an optical metallurgical microscope at $\times 800$ magnifications and the photo micrographs are shown in Fig. 1.

III. RESULTS AND DISCUSSION

A. Microstructure

The micrographs in Fig. 1 show the microstructures of the test specimens as viewed under the microscope. Directional solidification is a noticeable feature of the as-cast specimen while the crystals appear coarse due to grain growth. The morphology of the grains appears plate-like surrounded by

inclusions that are dispersed sparsely within the matrix. Dark-colored crystals of β -Mg₁₇Al₁₂ phase are also seen at the grain boundaries and their size relatively coarse. The coarseness of the second phase precipitates appear significantly reduced in the normalized structure while the α -Mg crystals are fine and well dispersed. Sundry intermetallics are apparent within the matrix which may have evolved sequel to the relatively low cooling rate of the alloy according to normalization condition which favors the growth of the intermetallics. The annealed microstructure shows substantial increase in the volume fraction of β -Mg₁₇Al₁₂ grains but as well a significant reduction in the degree of coarseness compared with the normalized specimens. The thermally aged structure after tempering developed fine precipitates of Mg₁₇Al₁₂ with a near perfect blend with the α -Mg matrix.

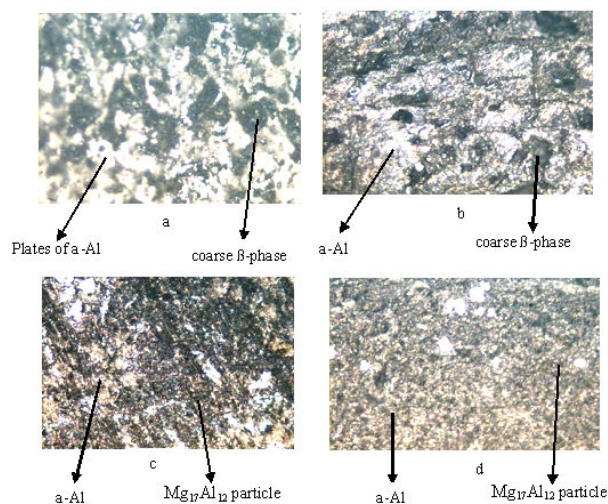


Fig. 1 Micrographs of 66/34Mg-Al alloy specimens in (a) as-cast (b) normalized (c) annealed and (d) aged conditions

B. Tensile Strength

The tensile behavior of test specimens is illustrated in Fig. 2. In comparison, the thermally aged specimens exhibited the highest ultimate tensile strength, 93.7MPa closely followed by the normalized, 81.6MPa while the annealed and as-cast show minimal difference having 75.2 and 70.3MPa respectively.

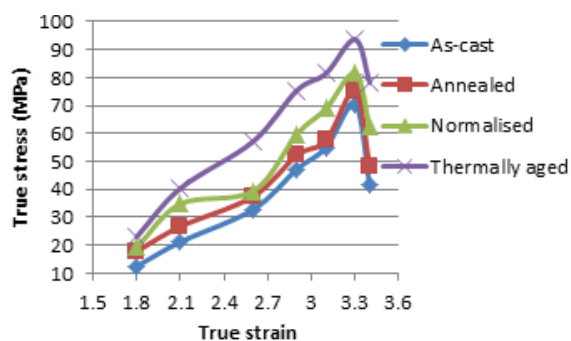


Fig. 2 True stress-strain graph of test specimens

The development of fine, round and a relatively large volume of Mg17Al12 precipitates in the thermally aged specimens coupled with the grains coherence offered increased resistance to dislocation motion. This phenomenon appears to be responsible for the relatively high ultimate tensile strength demonstrated by the aged specimen. Given the dearth of Mg17Al12 precipitates in the matrices of the annealed, normalized and as-cast specimens, their tensile strength values reduced progressively in relation to the volume, size, morphology and dispersion of the reinforcing precipitates in those specimens. In particular, the near absence of the magnesium-aluminum precipitates in the as-cast matrix coupled with inhomogeneous dispersion of the few precipitates present resulted in low strength (70.3MPa) while the relatively coarse crystals of the annealed impaired its strength (75.2MPa) in comparison with that of normalized specimens (81.6MPa).

C. Modulus of Elasticity

As shown in Fig. 3, the test specimens' constituents' tenacity behavior is illustrated by their modulus of elasticity. The specimens modulus vary according to the extent and nature of microstructure modification that occurred in response to the different heat treatment carried out. Clustering of the magnesium-aluminum precipitates in the as-cast and normalized specimens may have impaired cohesion of grains in particular at grain boundaries hence their relatively low elastic modulus, 5.6 and 6.4GPa, respectively. However, both the annealed and thermally aged specimens show significant improvement in their elastic modulus being 9.0 and 10.4GPa respectively.

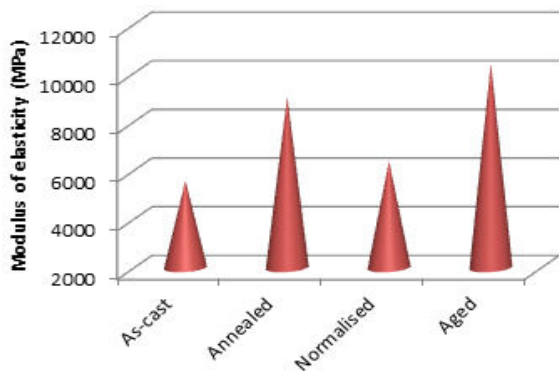


Fig. 3 Variations in test specimens' modulus of elasticity

These values compared well with the elastic modulus of human cortical bone which value falls in the range of 12-17GPa. The cortical (trabecule) represents nearly 80 percent of the skeletal mass which forms a protective outer shell around every bone in the body [15]. Cortical bone has a slow turnover rate and a high resistance to bending and torsion. Hence, it provides strength where bending would be undesirable. With these results, both the annealed and thermally aged Mg-Al alloy specimens have demonstrated the potentials for application as orthopaedic fixture without fear of stressshielding and warpage in service.

D. Impact Energy

The chart in Fig. 4 depicts variations in the amount of energy absorbed to fracture the test specimens. The values obtained invariably indicate the specimens' toughness characteristics which are 2.73J, 3.28J, 3.68J and 4.05J respectively for the as-cast, annealed, normalized and thermally aged. Adequate impact energy is usually required for enhanced plastic deformation of a coupling fixture such as an implant which may be daily subjected to stresses above the materials yield strength.

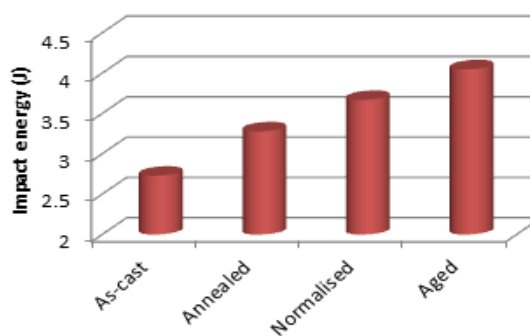


Fig. 4 Impact energy variations of test specimens

On close observation, the specimens' impact response shows similarity in trend with the tensile strength characteristics. The closeness between the ultimate tensile strength and strain as shown in Fig. 1 correlates the narrow average range, 1.3 of impact energy response of test specimens. This behavior stems from the specimens' microstructure in tandem with the general coarseness of the Mg17Al12 precipitates developed in the heat treated specimens except the thermally aged that exhibits fine precipitates dispersed homogeneously within the matrix integrity in terms of the amount and distribution of precipitates within the matrices (Fig. 1 (d)).

E. Micro Hardness

With the exception of the thermally aged specimens, the micro-hardness developed by the other test specimens is relatively low (Fig. 5).

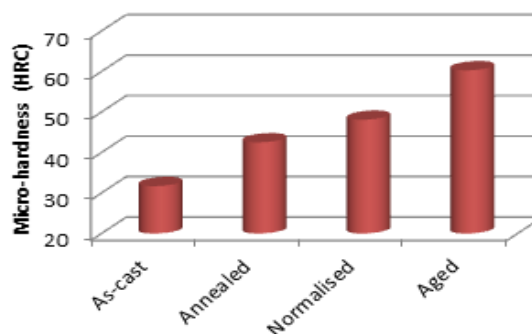


Fig. 5 Variations of micro-hardness developed by test specimens

IV. CONCLUSION

The prospect of 66/34Mg-Al alloy for orthopedic implant application has been investigated. From the results and their analyses, modest improvement in mechanical properties was achieved over the conventional implants. Specifically, the thermally aged specimen demonstrated superior ultimate tensile strength, 93.7MPa compared with 81.6MPa, 75.2MPa and 70.3MPa respectively for normalized, annealed, and as-cast specimens. The heat treated specimens also exhibited increase in impact energy, 3.28-4.05J compared to 2.73J of the as-cast ditto micro-hardness, 42.5-64.9HRC in contrast to that of as-cast which is 31.7HRC. The elastic modulus developed by the heat treated specimens in the range of 6.4-10.4GPa is comparable with that of conventional implants which is 3.0-20.0GPa.

It is concluded that through a simple and cost effective precipitation hardening procedure of thermal ageing, the mechanical properties of 66/34Mg-Al alloy can be improved and render suitable for medical implant application.

REFERENCES

- [1] Y. O. Kojima, "Platform Science and Technology for Advance Magnesium Alloys," Science Forum, pp. 350-35, 2000.
- [2] C. L. Liu, Y. C. Xin, and P. R. Chu, "Influence of Heat Treatment on Degradation Behavior of Bio-Degradable Die-Cast AZ63 Magnesium Alloy in Simulated Body Fluid," Materials Science Engineering A, vol. 456, pp. 350-357, 2007.
- [3] E. Zhang, and L. Yang, "Microstructure, Mechanical Properties and Bio-Corrosion Properties of Mg-Zn-Mn-Ca Alloy for Biomedical Application," Materials Science Engineering A, vol. 497, pp. 111-118, 2008.
- [4] M. P. Staiger, A. M. Pietaka, J. Huadamaia and G. Dias, "Magnesium and Its Alloys as Orthopedic Biomaterials, Journal of Biomaterials, vol.27, pp.1728-1734, 2006.
- [5] X. Guand and Z. Zheng, "A Review on Magnesium Alloys as Biodegradable Materials," Materials Science, vol.4, no.2, pp. 111-115, 2010.
- [6] M. Niinom and M. Nakai, "Titanium Based Biomaterials for Preventing Stress Shielding between Implant Devices and Bone," International Journal of Biomaterials, 2011.
- [7] Y. Hideki, F. Mikio and C. Akihiko, "Determination of the Mechanical Properties of Extruded Pure Magnesium during Tension-Tension low-Cycle Fatigue Using Ultrasonic Testing," Materials Transactions, vol.51, no.11, pp. 2025-2032, 2010.
- [8] C.K. Seal, K. Vince and M.A. Hodgson, "Biodegradable Surgical Implants Based On Magnesium Alloys – A Review of Current Research, IOP Conf. Series: Materials Science and Engineering, vol.4, pp. 1-4, 2009.
- [9] F. Wittea, V. Kaese, H. Switzer, L. A. Meyer, C. J. Wirth and H. Winuag, "In vivo Corrosion of Four Magnesium Alloys and the Associated Response, Biomaterials, vol.26, no.17, pp. 3557-3563, 2005.
- [10] L. Li, J. Gao and Y. Wang, "Evaluation of Cyto-Toxicity and Corrosion Behavior of Alkali-Heat-Treated Magnesium in Simulated Body Fluid, Surface Coating Technology, vol.185 no.7, pp.92-98, 2004.
- [11] P. Zartner, R. Cesenjever, H. Singer and M. Weyand, "First Successful Implantation of a Biodegradable Meta Stent into the Left Pulmonary Artery of a Preterm Baby, Catheter CardiovascInterv, vol. 66, no. 4, pp. 590-595, 2005.
- [12] R. A. Goyer, "Toxicity of Metals," ASM Metals Handbook, vol.2, 1992.
- [13] K. U. Kainer, "Magnesium-Alloy and Technologies," Wiley-VCH Verlag GmbH &Co, Germany, pp.1-184, 2003.
- [14] C. H. Turner and D. B. Burr., "Basic Biomechanical Measurements of Bone," Journal of Biomaterials Engineering, vol. 14, pp. 595-608, 1993.
- [15] F. Wittea, J. Fischer, J. Nellesen, H. Crostack and H.A. Crostack, "In vitro and in vivo Corrosion of Magnesium Alloys, Biomaterials, vol.27,no.7, pp. 1013-1018, 2005.