

EFFECT OF HOMOGENIZATION ANNEALING AND HYDROXYAPATITE COATING USING EDP TECHNIQUE ON MAGNESIUM ALLOY FOR ORTHOPEDIC APPLICATIONS

Sadiq Taoheed Olohunde, Idris Jamaliah, Mohd Zamani Ahmad and Mohamad Imran

Abstract—This study was aimed at improving the degradation rate and bioactivity of magnesium alloy by homogenization annealing and hydroxyapatite coating via electrophoretic approach. Hydroxyapatite was synthesized using co-precipitation method and deposited on mechanical treated and homogenized magnesium alloy. The characterization was done using optical microscope (OP), scanning electron microscope (SEM), transmission electron microscope (TEM), energy dispersive spectroscopy (EDS), Fourier transform infrared spectroscopy (FTIR). Homogeneous solution of hydroxy suspension was obtained by performing zeta potential measurement, and in-vitro bioactivity evaluation will be carried out in stimulated body fluid for 28 days. It is our expectation that a significant improvement would be recorded in controlling the degradation rate and in vitro evaluation of this alloy.

Keywords— magnesium alloy, homogenization annealing, hydroxyapatite; electrophoretic deposition.

Introduction

Magnesium alloys are not only very useful in computer, automobile and aeroplane industries owing to their good mechanical features such as high strength, low density, high strength to weight ratio [1, 2], but also as biodegradable implant materials for medical applications due to their good biocompatibility and biodegradability in the physiological body surroundings [3]. Also, they owned mechanical properties like cortical bone and good castability [4]. They are new generation biodegradable implants manufactured with tendency to regenerate critical wounded parts and remodel the skeletal bone [5]. These implant materials are essentially chosen to reinstate a part or recuperate a function of the human body in a way that is physiologically reliable, safe and acceptable [5]. Magnesium as the based metal plays a vital role in human body metabolism and serves as cofactor for many enzymes [6]. They have suitable mechanical properties, efficacious osteoconductivity and good biocompatibility for implantation. Nonetheless, magnesium alloys are limited in their applications due to the challenge to regulate their rapid degradation rate.

Therefore, it is crucially important to fulfil an approach that moderates an appropriate balance between the healing rate of the injurious part and implant corrosion rate [7]. This drawback in magnesium alloys had been dealt with via many surface modification applications like electrophoretic deposition [5,8], sol-gel [4, 9, 10], polymer coatings [11, 12], dip coating [9,13], electrodeposition [14,15], physical vapour deposition [6], micro arc-oxidation [16,17,18&19], fluoride treatment [9, 20], anodizing [21] etc. In this research, homogenization annealing is adopted to address this demerit in magnesium alloy fundamentally. By performing this, we aim at achieving a microstructure with no difference in mechanical properties and a moderate decomposition rate.

Hydroxyapatite is a calcium phosphate bioceramic coating material which protects the metallic substrate against corrosion attack in the biological environment thereby, recuperates the surface bioactivity and helps viability of the cell [10, 22]. It has chemical formula of $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$ with Ca/P ratio of 1.67 [13,23]. The natural bone is a nanostructured composite material made up of about seventy percent by weight of nano-hydroxyapatite crystals of almost 50 nm long and 50% nano-hydroxyapatite crystals by volume [24]. Hydroxyapatite is the most essential calcium compound found in natural hard tissues and teeth as mineral phase, of which the bioactivity and biocompatibility are well demonstrated [25,26,27]. Electrophoretic deposition is a forthcoming technique in depositing calcium phosphate (Ca-P) on surfaces of magnesium and its alloys due to its set-up simplicity, low processing temperature, economic efficiency, ability to form uniform film, easy control of coating thickness by regulating voltage and time of deposition, and capability of coating complex geometries [28, 29]. Also, it is applied for coating porous or microporous calcium phosphate on specimens with subsequent sintering to form a uniform and denser layer associated with good adhesion. The calcium phosphate coatings obtained had been described porous or microporous, while the high temperature for annealing or sintering had been discussed and depicted to produce a denser and uniform calcium phosphate coating associated with greater properties of adhesion [28, 30].

In this experiment, a hydroxyapatite coating was deposited on homogenized magnesium AZ91 samples. Afterward, microhardness tests, AFM analysis, corrosion behavior and in vitro bioactivity were investigated.

Sadiq Taoheed Olohunde

Address 1: Faculty of Mechanical Engrg, Universiti Teknologi Malaysia
line 2: Malaysia

Address 2: Engineering Materials Development Institute
KM4, Ondo Road, P.M.B 611, Akure, Ondo State, Nigeria.

Idris Jamaliah

line 1: Faculty of Mechanical Engineering, Universiti Teknologi Malaysia
line 2: Malaysia

A. Anticipated procedure

This research aims at providing a surface treatment for AZ91 alloy samples that would optimize their degradation rate for the applications of biodegradable implant through homogenization heat treatment with a hydroxyapatite coating. The first step is saddled with altering the microstructure of AZ91 samples to achieve enhanced corrosion behavior by subjecting them into homogenization annealing heat treatment to dissolve beta particles. The second stage deals with synthesizing hydroxyapatite with intention to produce a corrosion protective film that would guarantee a high degree of bioactivity. The next stage aims at depositing hydroxyapatite on heat treated samples via electrophoretic deposition technique using a graphite plate as anode and AZ91 as cathode at a constant voltage of 20V for 180sec. Finally, the effect of these surface modifications on AZ91 will be investigated via bioactivity evaluation and electrochemical corrosion behavior. The work is going, it is our expectation that the stated objectives will be achieved.

B. Sample preparation.

The experiment flowchart is presented in Figure 1, a magnesium-based alloy of grade AZ91 was cut into samples with dimension 20 mm x 20mm x 4 mm, following by mechanical polishing up to 800grit SiC paper, ultrasonic washing was performed on the samples using acetone and deionized water for 1800s to remove possible dirt [31]. Thereafter, homogenization annealing was carried out at a temperature of 410 ± 1 °C for 10h in the rotary furnace. The microstructure of the samples was characterized after polishing with 0.5µm diamond, rinsing in water and etching in 2% natal solution.

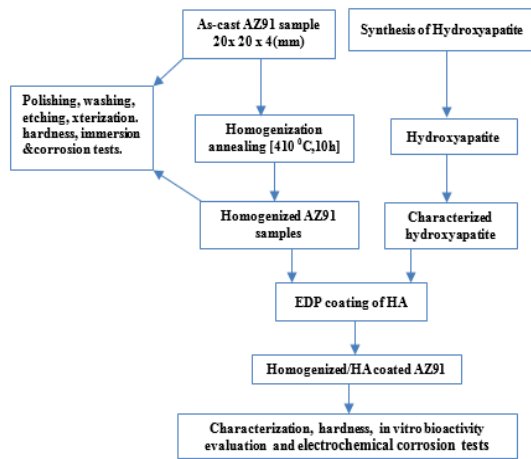


Figure 1: Experimental flowchart

A graphite plate was used as the anode and AZ91 sample as the cathode [5, 32]. The space between the cathode and opposite electrode in the electrophoretic deposition cell was kept constant at 10mm [33]. Electrophoretic deposition process was performed at constant voltage of 20V for 180s [34]. Finally, the coated samples were dried at a room temperature. The characterization was done using optical microscope (OP), scanning electron microscope (SEM), transmission electron microscope (TEM), energy dispersive spectroscopy (EDS), Fourier transform infrared spectroscopy (FTIR). Homogeneous solution of hydroxy suspension was obtained by performing zeta potential measurement, and in-

vitro bioactivity was evaluated in stimulated body fluid for 28 days.

C. Microstructure of AZ91 before and after homogenization annealing

The microstructural spectra of as-cast and homogenized AZ91 magnesium alloy samples are presented in Figure 2 and Figure 3 respectively. In Figure 2, dark spots are identified due to natal effect as etching material. These areas are regarded as eutectic α -phase composed aluminium composition more than the primary alpha phase. The corresponding EDS spectrum with all the main elements as composed in the AZ91 cast alloy is presented in Figure 3. Also, beta-phase particles and $\alpha+\beta$ phase are also recognized. However, its microstructure was changed after homogenization annealing, $\alpha+\beta$ phase was dissolved and larger percentage of beta phase was dissolved as presented in Figure 4. The SEM spectrum after homogenization annealing and hydroxyapatite coating on magnesium AZ91 alloy sample is presented in Figure 5. The SEM image showed hydroxyapatite coated surface revealing particle gather together with porous surface, this porous layer improves good bonding and proliferation of cells and bone attachment affinity [35].

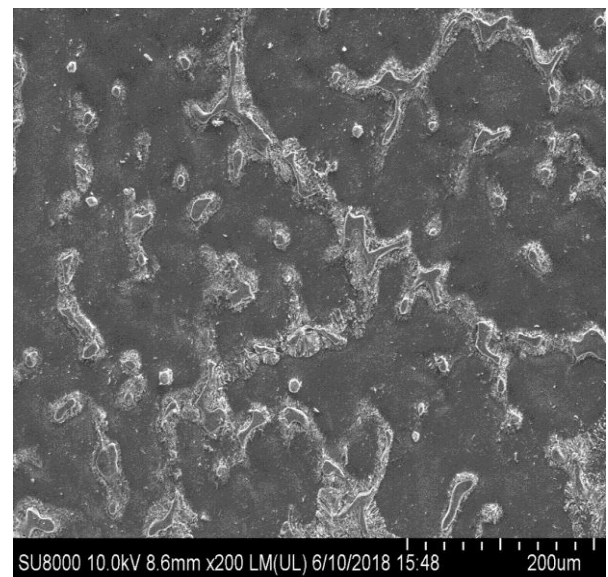


Figure 2: SEM microstrural image of as-cast AZ91

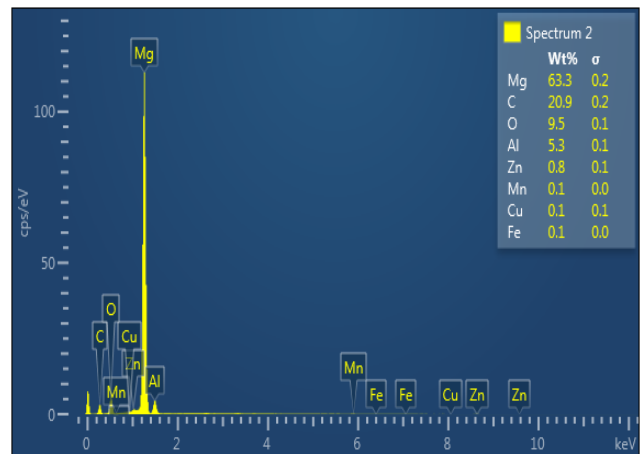


Figure 3: EDS spectrum of AZ91 alloy

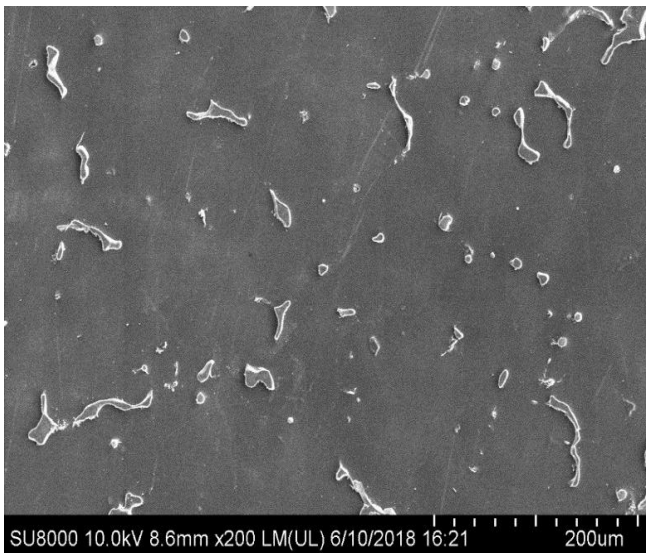
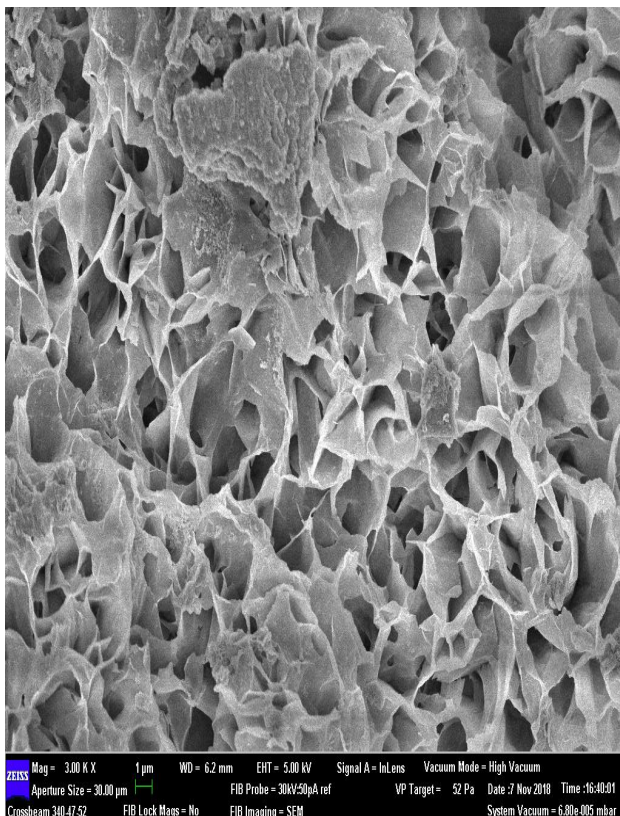


Figure 4: SEM spectrum of AZ91 alloy after homogenization annealing.

D. *In vitro* bioactivity assessment

Figure 6 presents the SEM spectrum of homogenized/hydroxyapatite coated specimen after immersed in SBF for 672h. The sample is observed to have been covered with occupied white layers of circular hydroxyapatite in both polished and unpolished sides as reflected in the past reports [13, 36]. Therefore, this generated layer possessed the required bioreactivity that could reduce the degradation rate of magnesium alloy.



SEM image of homogenized/hydroxyapatite coated AZ91 sample after immersion in SBF for 672hr.

E. *Conclusion*

The complete dissolution of fine lamellar particles, utmost dissolution and homogeneity of beta particles would definitely delay the corrosion and aid in regulating the corrosion rate of AZ91 alloy. Similarly, the generated layers after immersion would reduce the degradation rate of this alloy and enhance the bioactivity between the implant material and hosting tissue. It is anticipated that the homogenization annealing and hydroxyapatite coating via electrophoretic application would guarantee an improved bioactivity and the degradation rate of magnesium AZ91 alloy.

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About Author (s):



Sadiq T Olohunde received the B.Tech. & MSc. in Mech. Engineering from Ladoke Akintola University of Technology and University of Ibadan, Nigeria in 2009 & 2012 respectively. He is pursuing his PhD in the Faculty of Mech. Engineering, University Technology Malaysia, Malaysia. In 2011, he joined National Agency for Science and Engineering Infrastructure, Nigeria [EMDI, Akure]. He had been a Senior Engineer with Manufacturing and Production Section. He is a registered Engineer with the Council for the Regulation of Engineering in Nigeria. He is a professional member of American Society of Engineer, Nigeria Society of Engineers & International Association of Engineers.



Prof. Idris Jamaliah is one of the most Senior Professors in the Department of Materials, Manufacturing and Industrial Engineering, Faculty of Mechanical Engineering, Universiti Teknologi Malaysia, UTM, Skudai 81310, Johor Bahru, Malaysia. She received her PhD in Metallurgy and Material Engineering in 1989 from University of Wales, College of Cardiff.